

# **AutoChem 2950 HP**

## **Automated Catalyst Characterization System**

### **Operator's Manual**

**Rev A**

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# 1. GENERAL INFORMATION

## Manual Conventions

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This manual uses the symbols shown below to identify notes of importance, cautions, and warnings.



**Notes contain a tip or important information pertinent to the subject matter.**



**Cautions contain information to help you prevent actions which could damage the instrument.**



**Warnings contain information to help you prevent actions which could cause personal injury.**

## Equipment Description

The AutoChem 2950 HP is an automated high-pressure catalyst characterization system. The AutoChem enables you to perform experiments at pressures up to 1000 psia and at temperatures from -100 to 1100°C, based on type of sample tube used. Refer to [page 1-16](#).



**Do not use stainless steel sample tubes with temperatures above 850 °C\*. Doing so could damage the sample tube.**

\* Published material specifications, which include design safety factors, limit these tubes to 650 °C and 1000 psia. However, extensive testing at 850 °C and 1000 psia of numerous tubes of identical design and material have resulted in no failures. Additionally, as a safety measure, the tube in operation is enclosed in a clam-shell furnace. The furnace incorporates a limit switch which cuts the current and releases the pressure if opened to the point of switch actuation.



**Do not use quartz sample tubes with pressures significantly above ambient. Doing so could loosen the O-ring seal and allow gas leakage, and could also cause damage to the sample tube.**

The instrument can perform the following experiments individually or in various sequences:

- Temperature-Programmed Desorption (TPD)
- Temperature-Programmed Reduction (TPR)
- Temperature-Programmed Oxidation (TPO)
- Temperature-Programmed Reaction0
- Pulse Chemisorption
- BET Surface Area



The AutoChem 2950 HP system allows you to prepare and analyze samples at pressures up to 1000 psia. This capability makes it suitable for:

- Fuel cell applications that require reforming hydrocarbon feeds to hydrogen.
- Hydrogen storage applications using mixed metal hydrides that change hydrogen capacity as temperature changes. The AutoChem 2950 HP allows you to vary hydrogen pressure to determine storage capacity as a function of pressure.
- Simple chemical reactions where the product mix changes as a function of pressure. The AutoChem 2950 HP functions as a micro-reactor at typical process temperatures and pressures, allowing you to collect reaction data at commercial conditions. This capability reduces the typical risks and inaccuracies of using ambient pressure reaction data to design high-pressure reaction vessels.

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## Software

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The Autochem 2950 HP software represents a major advance in the convenience and control of temperature-programmed analyses because of the high level of automation offered. Very few steps must be physically performed by an operator; the vast majority are controlled with precision by the software.

This automated process is determined by the sample file you create. In addition to information about the sample itself, the sample file contains a list of specific instructions, which the analyzer follows to perform any series of experiments. The instrument monitors the progress of each step, then initiates the next one while continuously controlling temperatures and gas flows. Delays are eliminated, because most experiment steps do not require an operator to initiate them. The potential for human error is reduced, and less labor is required. You can create standard sets of analysis conditions, making it possible for a laboratory manager to standardize procedures for laboratory technicians to use during repeat sample analyses.

The Windows® interface makes it easier than ever to create sample files, control analyses, and generate the reports you need. You can:

- Monitor detector signal, time, temperature, and pressure data.
- Create and store standard sets of analysis conditions for frequently performed analyses.
- Integrate detector signal peaks both automatically and manually.
- Display and print peak graphs and reports.
- Establish calibration curves for calculation of quantity adsorbed, consumed, and desorbed.
- Reprocess stored analysis data using different parameters.
- Save data to a spreadsheet or in ASCII text format for use in other applications.

## Flexible Peak Editing

The AutoChem's powerful Peak Editor enables you to evaluate results quickly and easily, edit peaks, and produce reports that reflect your specific needs. Adjusting peak boundaries is a matter of simply pointing and clicking. The Peak Editor also enables you to separate composite peaks

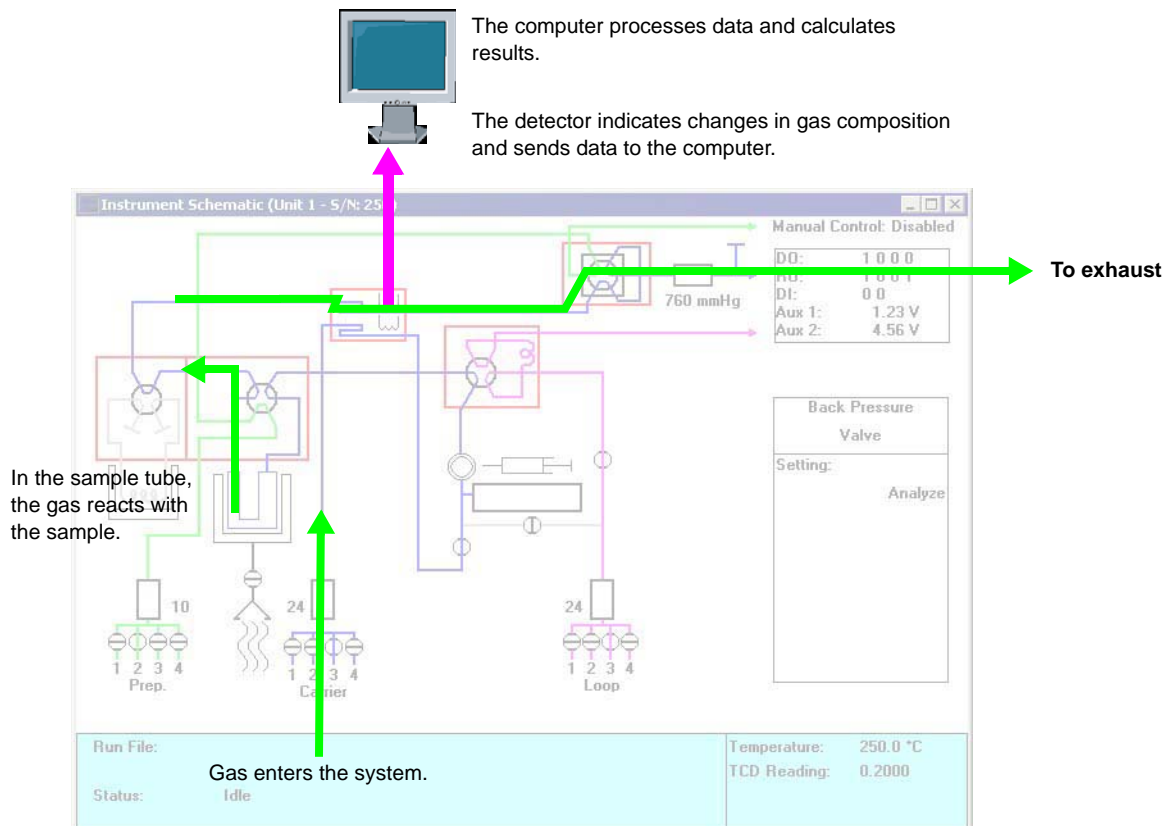
## Temperature-Programmed Analyses



This section provides a basic, simplified description of the technique for performing temperature-programmed analyses.

The AutoChem 2950 HP is a sophisticated instrument that enables you to perform a variety of complex experiments. Most temperature-programmed experiments, however, are based on the following *highly simplified* steps:

1. Gas flows into the analyzer.
2. The gas interacts with the sample as the temperature changes.
3. Gas flows past the detector.
4. The detector signal varies as the gas composition changes. The detector signal is recorded.
5. The software plots and calculates results.



**How does the detector work?**

The detector contains heated filaments; it measures the change in gas thermal conductivity between the gases flowing over the sample and reference filaments.

The gases flowing past the detector cool the filament by extracting heat. The *thermal conductivity*<sup>1</sup> of the gas determines how quickly the gas removes heat from the filament. A gas with a high thermal conductivity cools the filament rapidly, and more power is required to maintain its temperature. A gas with a lower thermal conductivity removes heat from the filament more slowly.

When the sample reacts with the gas, it causes changes in the composition of the gas and, consequently, changes the thermal conductivity of the gas. These changes are sensed by the detector as an increase or decrease in the amount of power required to maintain the filament at a constant temperature.

**What role does sample temperature play?**

Because the sample's temperature determines how rapidly it reacts with the analysis gas (or if it reacts at all), data are typically sent to the computer over a linear range of temperatures which you specify.

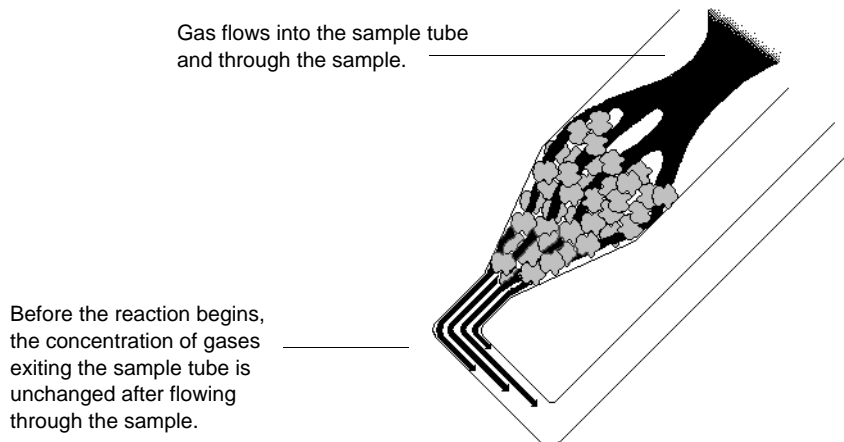
In some experiments, to start collecting data at a very low temperature to establish a baseline where the gas is completely unaffected by the sample. In other cases, to collect data after a reaction has started. In still other experiments, your primary interest may be determining the temperature at which the maximum rate of reaction occurs.

**Here's one example:**

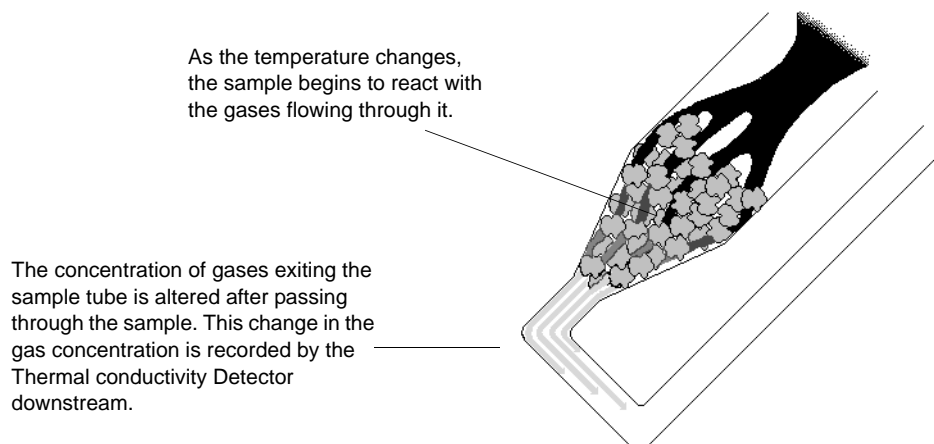
Consider the example of a Temperature-programmed reduction (TPR). During the TPR, a metal oxide reacts with hydrogen to form a pure metal. This reaction is referred to as "reducing" the metal; for example, TPR of a catalyst containing platinum. Argon, which has a very low relative thermal conductivity<sup>2</sup>, is used as a component in the carrier gas. It is blended in a fixed proportion with hydrogen, the reducing gas with a much higher thermal conductivity. Then the gas mixture flows through the analyzer, through the sample, and past the detector.

When the hydrogen and argon gas blend begins flowing over the sample, a baseline reading is established by the detector. This baseline is established at a low enough temperature so that no reduction of the sample occurs. The baseline level indicated by the detector is that of the thermal conductivity of the two gases in their fixed proportion. In other words, the proportion of gases flowing over the detector is the same as the proportion of gases entering the analyzer, because at the low temperature, there is no interaction with the sample.

1. The thermal conductivity of a gas is its ability to conduct heat. Each gas has a distinct thermal conductivity. The thermal conductivity is most commonly referred to as relative thermal conductivity and the values are relative to air.
2. The ratio of relative thermal conductivities is an indication of sensitivity - a large ratio (>5) is desirable.



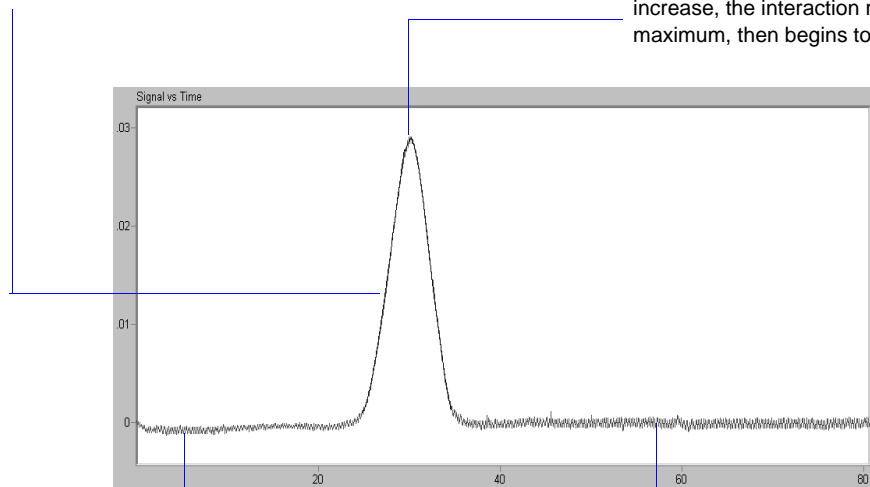
The temperature is then changed, and when a critical temperature is reached, hydrogen atoms in the gas flow react with the sample, forming  $\text{H}_2\text{O}$  molecules. The  $\text{H}_2\text{O}$  molecules are removed from the gas stream using a cold trap. As a result, the amount of hydrogen in the argon/hydrogen gas blend inside the analyzer *decreases*, and the proportion between the two gases shifts in the direction of argon, as does the mixture's thermal conductivity.



Since argon has a lower thermal conductivity than hydrogen, the mixture's thermal conductivity consequently decreases. The flowing gas removes heat from the filament more slowly, requiring less electricity to maintain a constant filament temperature. The instrument records the electrical demand as it changes (this is called the detector signal). The detector signal is recorded continuously over a range of temperatures. When these readings are graphed, the data form one or more peaks. Peaks can be positive or negative.

2. As the temperature changes, the sample begins to react with one of the gases. Therefore the gas mix is then made up of a larger proportion of the other gas. This causes a shift in the mixture's thermal conductivity. The detector measures this change by recording the change in the amount of electricity required to maintain constant filament temperature.

3. As temperature continues to increase, the interaction reaches a maximum, then begins to diminish.



1. Baseline readings. The gas(es) is (are) not reacting with the sample, so there is no change in the signal from reading to reading.

4. As fewer and fewer sample atoms are available to bond with the analysis gas, there is less and less change in the mix of gases flowing into the analyzer and past the detector, so the thermal conductivity shifts back toward the baseline value.

This example illustrates the fundamental concept upon which the analyzer operates. Of course, the various types of analyses the AutoChem 2950 HP can perform result in different types of traces. For example, a pulse chemisorption analysis results in a series of peaks that gradually increases in size as the sample is dosed with separate increments of gas. Initially, the gas uptake by the sample results in smaller peaks. But when all the active sites are saturated, no more gas can be taken up and the peaks become equal.

## Peak Area

The area beneath each peak is calculated to provide information about the volume of gas reacted during the analysis. Calculations are described in Appendix B, [CALCULATIONS](#).



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## Automatic Operation

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The AutoChem 2950 HP software provides a simple format for you to specify all the analysis conditions for the experiment; you create a sample file which contains sample information and a list of specific steps the analyzer will follow to perform the experiment(s). Then, the instrument automatically performs the analysis, from controlling the gas mixture and flow rate to monitoring the temperature and pressure. Data points are recorded automatically. BET analyses require some operator interface. After analysis, you can use the Peak Editor to adjust the peaks to create reports that contain the data you need, without baseline noise or other undesirable effects.

Because you can specify up to 99 experiments and each experiment can contain up to 99 steps, the AutoChem 2950 HP can perform a wide variety of preparation and analysis functions automatically.

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## Cold Trap

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In some cases, it is desirable to trap condensable substances resulting from the reaction. In the previous example, water is produced during the analysis. If the gas flow is diverted through the cold trap, the water can be removed using an appropriate cryogen before the gas flows past the detector. The cold trap can also be used as a delay path for BET surface area experiments.

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## Injection Loop

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Injection loops are provided for injecting carefully measured doses of gases for analyses such as Pulse Chemisorption. The AutoChem 2950 HP is shipped with a 0.5-cm<sup>3</sup> loop installed; 1.0- and 5.0-cm<sup>3</sup> loops are also included. If you set up your sample file so that a loop is used for introducing gas into the analyzer, the instrument will automatically dose the sample as specified in the sample file.

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## Sample Preparation and Calibration

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Depending on the type of experiment(s), sample preparation and/or calibration may be required. More specific instructions are contained in the appropriate sections of this manual.

A sample is prepared for analysis by removing unwanted adsorbates from the surface of the sample. This is usually accomplished by flowing gas over the sample and may include heating the sample. The flowing gas may be inert or chemically active gases may be used to activate the surface.

Calibration routines provide the AutoChem 2950 HP analyzer and software with the appropriate information to convert electrical signals to physically meaningful data such as volume adsorbed, loop volume, and gas concentration.

## Analysis Types

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Regardless of which type of experiment you perform, the basic concept is the same: the filament detects changes in the gas composition. The sample, gas selection, and analysis conditions determine what changes occur. Some of the analyses you can perform with the 2950 are described in the following sections.



**A tutorial for some of the different types of analyses is provided in Appendix J.**

### TPD Analysis

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Temperature-Programmed Desorption (TPD) analyses determine the number, type, and strength of active sites available on the surface of a catalyst from measurement of the amount of gas desorbed at various temperatures.

After the sample has been outgassed, reduced, or otherwise prepared, a steady stream of active gas flows through the sample and adsorbs on the active sites. Programmed desorption begins by raising the temperature linearly with time while a steady stream of inert carrier gas flows through the sample.

At a certain temperature, the energy exceeds the activation energy; therefore, the bond between the adsorbate and adsorbent will break, and the adsorbed species desorb. If different active metals are present, they usually desorb the adsorbed species at different temperatures. These desorbed molecules enter the stream of inert carrier gas and are swept to the detector, which measures the gas concentrations. The volume of desorbed species, combined with the stoichiometry factor and the temperature at which pre-adsorbed species desorb, yields the number and strength of active sites.

### TPR Analysis

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Temperature-Programmed Reduction (TPR) determines the number of reducible species present in the catalyst and reveals the temperature at which the reduction occurs. An important aspect of TPR analyses is that the sample need not have any special characteristics other than containing reducible metals.

The TPR analysis begins by flowing analysis gas (typically hydrogen in an inert carrier gas such as nitrogen or argon) through the sample, usually at ambient temperature. While the gas is flowing, the temperature of the sample is increased linearly with time and the consumption of hydrogen by adsorption/reaction is monitored. Changes in the concentration of the gas mixture are determined. This information yields the hydrogen uptake volume.

## TPO Analysis

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Temperature-Programmed Oxidation (TPO) examines the extent to which a catalyst can be oxidized or was previously reduced.

Usually the sample is pretreated and the metal oxides are reduced to the base metal, typically with a gas mixture of hydrogen with either nitrogen or argon. Then the reactant gas, typically 2 to 5% oxygen with helium, is applied to the sample in pulses or, alternatively, as a steady stream.

The furnace heats the sample tube and sample according to the user-selected temperature program. The oxidation reaction occurs at a specific temperature. The AutoChem 2950 HP measures the uptake of oxygen.



**When using any mixture of gases for TPR or TPO analyses, make sure the thermal conductivities of the two gases in the mixture are quite different for maximum sensitivity (see Appendix C).**

## Pulse Chemisorption Analysis

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A Pulse Chemisorption analysis determines active surface area, percent metal dispersion, and the average active particle size by applying measured doses of reactant gas to the sample.

The gas reacts with each active site until all sites have reacted. Once the active sites have completely reacted, the discretely injected gas volumes elute from the sample tube unchanged. The amount chemisorbed is the difference between the total amount of reactant gas injected and the amount that eluted from the sample. The quantity of each pulse of reactant gas is determined by the loop volume on an automatically controlled valve. Three loops of different volumes are provided with the analyzer.

## BET Surface Area Analysis

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The BET Surface Area analysis lets you evaluate total surface area of the catalyst before and after chemisorption. Using this method, you can study pore-plugging phenomena, which might occur due to the irreversible adsorbed species during chemical reactions, as well as the occurrence of sintering.

After outgassing the sample, a mixture of nitrogen and helium (typically 5 to 30% N<sub>2</sub>) flows over the sample which is immersed in a liquid nitrogen (LN<sub>2</sub>) bath. Both the adsorption and desorption of the nitrogen are recorded. The amount of nitrogen desorbed at LN<sub>2</sub> temperatures and the sample weight are used to calculate total specific surface area.

The entire BET analysis — or even repeat analyses — is performed *in situ*.

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## Langmuir Surface Area Analysis

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The Langmuir surface area analysis allows you to evaluate the total surface area of the catalyst and is especially useful for adsorbate/adsorbent systems that produce only a monolayer. These materials typically exhibit a Type 1 isotherm. The Langmuir surface area analysis may be best applied to chemisorption, zeolites, and microporous carbons – typically the Langmuir surface area will exceed the BET surface area for these materials and provide a more accurate estimation of the total surface area.

The Langmuir surface area report may be applied to a wide range of gas concentrations and is not limited to the typical BET range (5 to 30% N<sub>2</sub>).

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## Total Pore Volume Analysis

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The Total Pore Volume is a single-point estimate of the pore capacity of a material. The total pore volume analysis is usually conducted near the saturation pressure of the adsorbate (0.995 P/P<sub>0</sub>). The total pore volume of a material can be determined on both fresh and used materials, the difference in pore volume may indicate pore plugging and directly relate to changes in the performance of catalysts and adsorbents.

For high surface area (> 100 m<sup>2</sup>/g), a sample quantity less than 50 mg is recommended. The high sensitivity combined with the large quantity of adsorbed gas allows the use of smaller sample quantities while maintaining high precision. The reduced sample quantity will also reduce the likelihood of saturating the high sensitivity detector.

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## Additional Uses of the AutoChem 2950 HP

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You may also use the AutoChem 2950 HP for Temperature-Programmed Reactions, Catalyst Pretreatment, and Isothermal Reactions. These techniques are similar to many of the procedures that have been previously described. The tremendous flexibility of the AutoChem 2950 HP allows you to use custom applications.

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### Catalyst Pretreatment

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Catalyst Pretreatment usually consists of activating a catalyst prior to its use in a chemical reaction. For example, to perform a temperature-programmed reaction, you must reduce the catalyst under a flow of H<sub>2</sub> at a specific temperature. The reduction is necessary to activate the surface, or reduce atoms of active metals, which are responsible for the activity of a catalyst.

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## Temperature-Programmed Reaction

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A Temperature-Programmed Reaction lets you monitor the products from the reaction between gases and a catalyst at a specified temperature. You can program the AutoChem 2950 HP analyzer to raise the temperature of a catalyst bed at a constant ramping rate as the gases flow through the catalyst. At the optimal temperature, the gases react in the presence of the catalyst, creating products. The products of the reaction and the excess reactants can be diverted to a mass spectrometer to be analyzed.

---

## Isothermal Reaction

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An Isothermal Reaction is similar to a temperature-programmed reaction except that the catalyst is kept at a constant temperature (isothermal) to perform the catalytic reaction. Both the product of the reaction and the excess reactants can be diverted to a gas chromatograph or to a mass spectrometer to be analyzed.

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## Applications

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Catalytic processes that benefit from TPD/TPR analyses include:

Polymerization  
Hydrogenation  
Catalyst Cracking  
Hydrocracking  
Isomerization

Oxidation  
Dehydrogenation  
Hydrotreating  
Alkylation Reforming

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## System Requirements

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### Gases

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Compressed gases are required for sample analysis with the AutoChem 2950 HP analyzer. Gas bottles and a rack for storage of these bottles should be located near the analyzer. As an alternative, outlets from a central source may be provided nearby. Gas sources must be able to supply flow rates of up to 200 cm<sup>3</sup> per minute. The gas bottle should be capable of supplying 1050 psig of gas.

Gas purity should be 99.999% and gases should be free of H<sub>2</sub>O.

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### Gas Regulators

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High-purity regulators should be used with the AutoChem 2950 HP. (Appropriate gas regulators are available from Micromeritics; refer to **Ordering Information** on page 10-1.) Refer to **“Changing Gases”** on page 9-31 for instructions on connecting gas bottles and regulators.

## Coolant

An inlet port is provided on the front of the furnace for attaching a coolant. One cooling device, the KwikCool, is included as a standard accessory. The optional CryoCooler may be purchased from Micromeritics. Refer to **Ordering Information** on page [10-1](#).

- The KwikCool enables you to cool the furnace rapidly to ambient temperature. This speeds the analysis and allows for more rapid throughput.
- The CryoCooler makes it convenient to perform subambient analyses. In some cases, such as the reduction of Platinum, the reaction begins at approximately -28 °C, so analyses begun at ambient temperature are undesirable. The CryoCooler, in conjunction with the furnace, lets you continuously ramp temperatures from -120 °C to 1100 °C. The CryoCooler can also be used to speed cooling to ambient following an analysis.



### Safe Operation Guidelines for Stainless Steel Tubes

- External cooling of the tube above 200 °C may be assisted only with air not to exceed 3 cfm @ 30 psi.
- External cooling of the tube below 200 °C may be assisted with the CryoCooler at a rate less than 90 °C/min.
- Do not use other cooling agents below 200 °C because they may embrittle the sample tube.
- Do not submerge the tube in water or oil, or spray the tube with water or oil.

Failure to follow these practices may cause bodily harm and/or damage to the instrument and sample tube. The AutoChem HP has been optimized to use these guidelines for safe operation.

## Specifications

The following table contains the specifications of the AutoChem 2950 HP.

### *AutoChem 2950 HP Specifications*

Characteristic	Specification
<div>TEMPERATURE SYSTEM</div>	
Range:	<div>Standard Pressure (up to slightly above atmospheric pressure) in quartz sample tubes: -100 to 1100 °C (with optional CryoCooler) Ambient to 1100 °C (without CryoCooler)</div> <div>High Pressure (up to 1000 psia) in stainless steel sample tubes: -100 to 850 °C (with optional CryoCooler) Ambient to 850 °C (without CryoCooler)</div>
Selection:	Digitally set, 1 °C increments
Ramp rates:	Up to 50 °C per minute within -70 to 500 °C range; Up to 30 °C per minute within 500 °C to 750 °C range; Up to 10 °C per minute within 750 °C to 1100 °C range
<div>GASES</div>	
Loop (Analysis):	H <sub>2</sub> , CO, O <sub>2</sub> , N <sub>2</sub> O, He, Ar, NH <sub>3</sub> (maximum 15% NH <sub>3</sub> in helium), and others compatible with 316 SS
Carrier:	
Preparation:	
<div>GAS FLOW RATE</div>	
All Mass Flow Controllers (MFC)	
Manual control:	0 - 100 STD cm <sup>3</sup> /min. (Rate for Nitrogen; other gases have a different range)
Automatic analysis:	10 - 100 STD cm <sup>3</sup> /min.
Pressure Controller	
Pressure specifications:	Atmospheric to 1000 psia Accurate to 0.1% of full scale

Characteristic	Specification
<b>————— GAS DELIVERY —————</b>	
Inlet ports:	4 each for preparations, carrier, and loop (analysis) gas
Temperature control:	Internal gas lines and valves can be heated up to 150 °C
<b>————— SAMPLE TUBE —————</b>	
Type:	Metal FlowThru tubes*, 316 SS, for use up to 850 °C
Sample size:	0.3 inches diameter (7.5 mm) by 1.5 inches (40 mm)
Pressure:	Up to 1000 psia
Type:	Fused quartz FlowThru tubes, for use up to 1100 °C
Sample size:	Powders and pellets up to 9 mm in diameter
Pressure:	Atmospheric pressure only
<p>* Published material specifications, which include design safety factors, limit these tubes to 650 °C and 1000 psia. However, extensive testing at 850 °C and 1000 psia of numerous tubes of identical design and material have resulted in no failures. Additionally, as a safety measure, the tube in operation is enclosed in a clam-shell furnace. The furnace incorporates a limit switch which cuts the current and releases the pressure if opened to the point of switch actuation.</p> <p>Reactive gases and reaction products, the nature of which cannot be anticipated by Micromeritics, can corrode or embrittle any steel. The user is advised to examine each tube after use and discard any that might appear questionable. It is also essential for maximum safety that, whenever tubes are at elevated pressure even at ambient temperature, the furnace be in place and closed should a failure occur.</p>	
<b>————— ELECTRICAL —————</b>	
Voltage:	85 to 265 VAC
Frequency:	50/60 Hz
Power:	1100 VA, operating, maximum
<b>————— ENVIRONMENT —————</b>	
Temperature:	15 to 35 °C operating; 0 to 50 °C non-operating
Humidity	20 to 80% relative, non-condensing



Characteristic	Specification
<b>PHYSICAL</b>	
Height	24.5 in. (62.2 cm)
Width:	26.0 in. (66 cm)
Depth:	22.75 in. (58 cm)
Weight:	130 lbs (60 kg)
<b>COMPUTER</b>	
Minimum requirements:	Computer capable of running Windows® 2000, Windows XP Professional, or Windows Vista® Business or Ultimate operating system CD-ROM drive 128 megabytes of main memory 1-gigabyte hard drive SVGA monitor (800 x 600 minimum resolution)
<b>WETTED MATERIALS</b>	
<b><u>Standard System</u></b>	
<ul style="list-style-type: none"> <li>• Stainless steel</li> <li>• Gold plated nickel/iron (filaments)</li> <li>• Kalrez® or Chemraz® (most O-ring seals)</li> <li>• Quartz (sample tubes, thermocouple sheath)</li> <li>• Quartz wool (supports sample in tube)</li> <li>• Inconel® - sheathed sample thermocouple (can be placed outside tube or sheathed in quartz)</li> <li>• PEEK (Poly Ether Ether Ketone - some ferrules)</li> <li>• Teflon® tubing (most exhaust lines); TCD exhaust is stainless steel for connection of external devices)</li> <li>• Copper (gas supply lines and BET delay path)</li> <li>• Nickel plated brass and steel (gas inlet valves)</li> <li>• Viton® (seals inside mass flow controllers)</li> <li>• Polyaryletherketone/PTFE composite (rotary valves seals)</li> <li>• Silicone/Teflon sandwich (septum)</li> </ul>	
<b><u>System with Corrosion Resistant Option</u></b>	
As above, but stainless steel manifolds are protected from corrosion with vapor deposited Silcolloy by Silcotek®, Stainless-steel tubing, fittings, ferrules and rotary valves are replaced by Hastelloy-C® versions, Kalrez is used for all O-ring seals and the seals inside the mass flow controllers, stainless-steel grade 316 gas supply lines, stainless-steel gas inlet valves with Kalrez or Chemraz seals.	

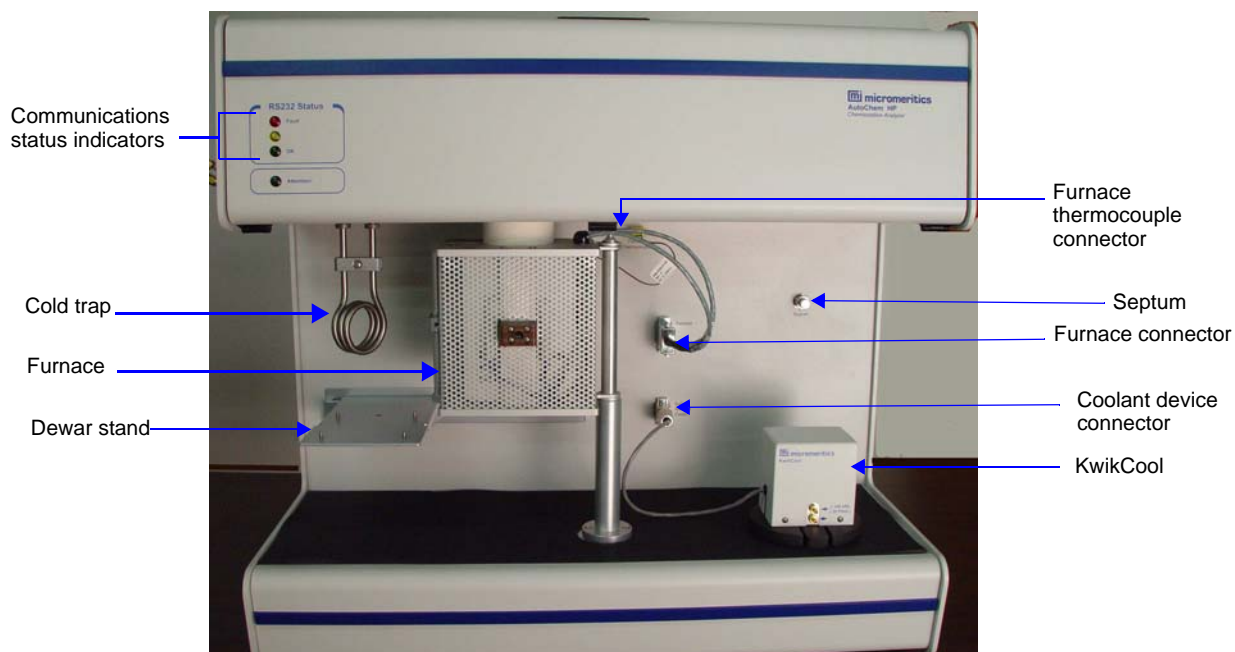


## 2. USER INTERFACE

This chapter describes the controls, indicators, and connectors on the analyzer and provides general instructions for using the software.

### Controls, Indicators, and Connectors

#### Front Panel

**Dewar stand**

Supports the cold trap Dewar.

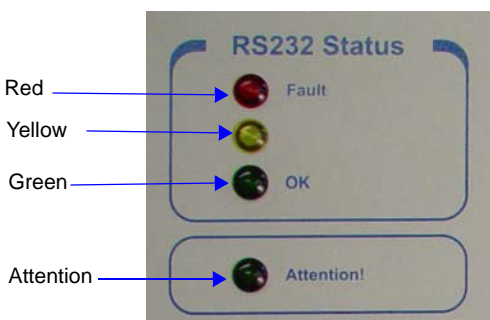
**Furnace**

Heats the sample to a user-selectable temperature.

**Cold trap**

Allows trapping of condensibles in the gas flowing from the sample before it reaches the thermal conductivity detector. A delay loop or dryer may also be attached.

### Communication Status Indicators



The top three indicators show the state of the analyzer's communications with the computer:

**Red (fault):** There is no communication with the computer. (For example, if the 2950 software was exited). When in this state, the Attention! indicator (which is green) is also illuminated.

**Yellow:** No longer used.

**Green (OK):** Normal communications.

The fourth indicator is the **Attention** LED, which blinks to alert the operator that attention is required. For example, if a Wait step requiring action by the operator was included in the list of experiment steps, the LED blinks and the appropriate message appears on the computer screen. After the operator performs the requested action and clicks **OK**, the LED ceases to blink.

### Furnace thermocouple connector

Connects the furnace thermocouple to the analyzer.

### Septum

Used for injecting quantities of gas into the analyzer, such as during a loop calibration.

### Furnace connector

Connects the furnace to the analyzer.

### Coolant device connector

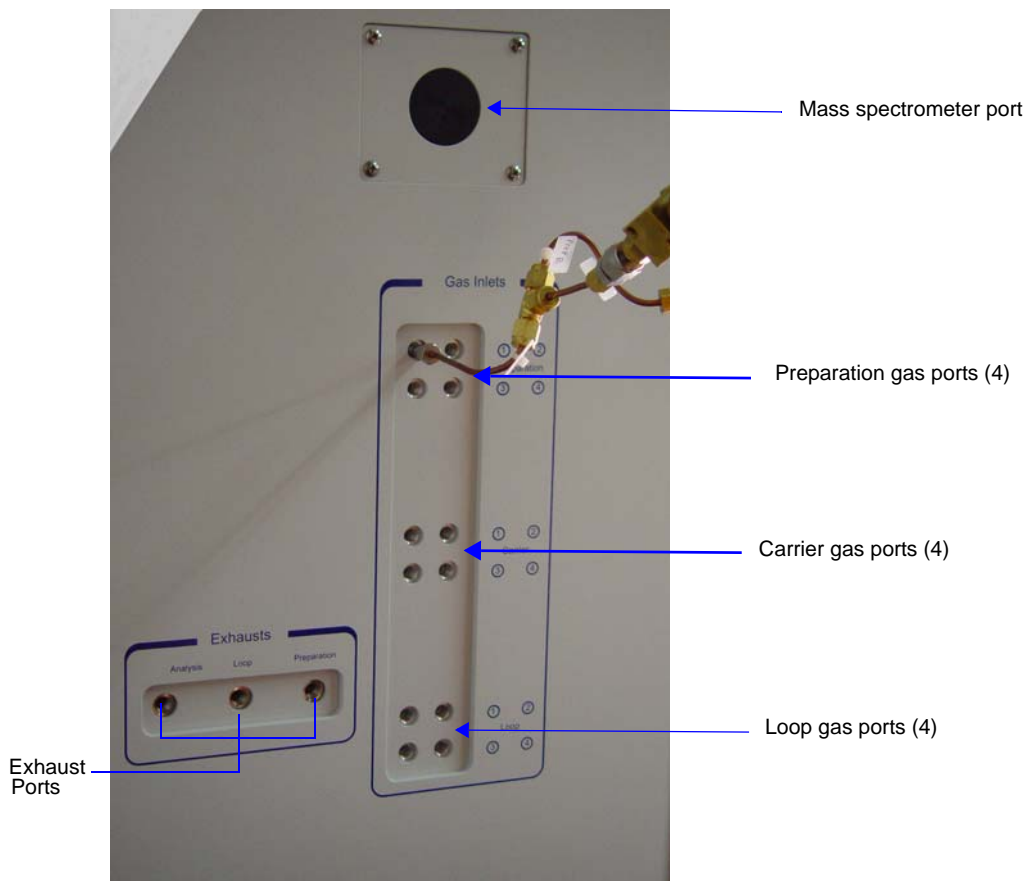
Connects a KwikCool or CryoCooler to the furnace.

### KwikCool

The KwikCool enables you to ramp the temperature rapidly down to near ambient, reducing analysis time and increasing throughput.

The optional CryoCooler enables you to start analyses at subambient temperatures. It can also be used to speed throughput by ramping the the furnace temperature rapidly down to ambient after an analysis.

## Left Side Panel

**Mass spectrometer port**

Used to connect a mass spectrometer to the analyzer.

**Preparation gas ports**

Up to four of these gases can be attached to the Preparation ports: H<sub>2</sub>, O<sub>2</sub>, He, Ar, and others.

**Carrier gas ports**

Up to four of these gases can be attached to the Carrier ports: He, Ar, and other inert gases.

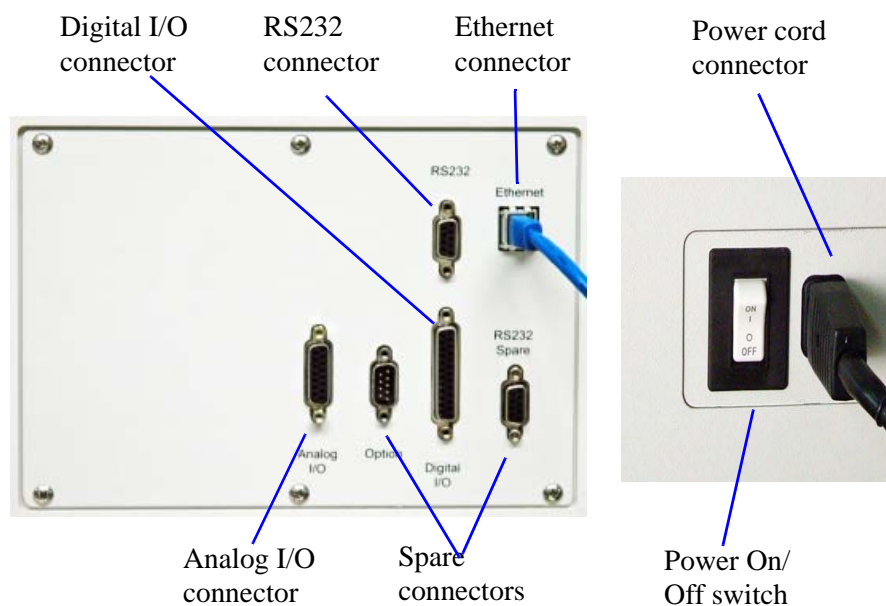
**Loop (analysis) gas ports**

Up to four of these gases can be attached to the Loop ports: H<sub>2</sub>, CO, O<sub>2</sub>, N<sub>2</sub>O, NH<sub>3</sub> (maximum 15% NH<sub>3</sub> in helium), and others.

**Exhaust ports**

Used for venting the gas exhausts. The appropriate method for venting each port depends upon the gases you are using for the experiments you are performing.

## Right Side Panel

**Analog I/O connector**

Used to receive data from auxiliary devices with analog output (refer to Appendix E).

**Digital I/O connector**

Used to receive data from auxiliary devices with digital output (refer to Appendix E).

**Ethernet connector**

Used to connect the analyzer to the computer via an Ethernet cable.

**Power cord connector**

Used to connect the analyzer to the power source.

**Power On/Off Switch**

Turns power to the analyzer on and off.

**RS232 connector**

Not used.

**Spare connectors**

Not used.

## Turning On the Analyzer



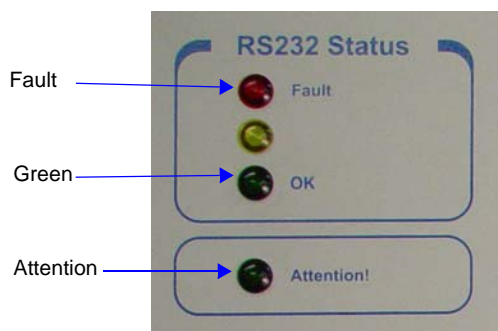
**DO NOT connect or disconnect ANY cables when power is on. Connect and disconnect ALL cables when power is off.**

Place the power switch (main breaker switch) on the side panel of the analyzer in the ON ( | ) position. The power switch is located on the right side panel of the analyzer next to the power cord connector.

When you turn the system on for the first time or after a period of non-use, follow these procedures:

1. Turn on the analyzer.
2. Start the 2950 HP analysis software.
3. Enable manual control, then set the target temperature for the heat zones to 110 °C. Refer to **“Enable Manual Control” on page 6-12.**
4. Flow a purge carrier gas (such as helium or nitrogen) through the system at about 50 cm<sup>3</sup>/min.
5. Allow the system to warm up for at least three hours before analyses are performed.

When the analyzer is turned on (but the software is still not running), the uppermost and lowermost indicator lights (the red Fault light and the green Attention! light) on the upper front panel are illuminated. When the software is subsequently started and communication is established with the analyzer, these lights are turned off and the second light from the bottom (the green OK light) is illuminated.



**A carrier gas must be flowing through the detector whenever the detector filaments are turned on; otherwise, the filaments will deteriorate and lose sensitivity. If the gases are shut off, the filaments are automatically turned off after five minutes.**

The flowing gas also continuously cleans the plumbing of any water vapor which can enter when the sample tube is removed. Ten cm<sup>3</sup>/min is sufficient to perform this function.

## Using the Software

### Keyboard Shortcuts

Shortcut keys can be used to activate some menu commands. Shortcut keys or key combinations (when applicable) are listed to the right of the menu item.

Certain menus or functions can also be accessed using the Alt key plus the underlined letter in the menu command. For example, to access the File menu, press Alt + F, then press the underlined letter on the submenu. For example, Alt + F opens the File menu, then press O to access the File Selector for opening files.

Key(s)	Function
<b>F2</b>	Open a sample information file Clear the selected field (when using the Select Dates dialog)
<b>F3</b>	Open an analysis conditions file Insert the current date (when using the Select Dates dialog)
<b>F4</b>	Open a report options file Display a calendar (when using the Select Dates dialog)
<b>F6</b>	Tile windows
<b>F7</b>	Cascade windows
<b>F8</b>	Start report
<b>F9</b>	Close all open report windows
<b>Alt + F4</b>	Exit the analysis program
<b>Shift + F2</b>	List sample information files
<b>Shift + F3</b>	List analysis conditions files
<b>Shift + F4</b>	List report options files
<b>Shift + F9</b>	Display action(s) available for the selected component on the instrument schematic when manual control is enabled



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**File Status and Description**

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Status	Description
<b>All</b>	All sample information files in the specified directory and within the specified range of dates.
<b>Analyzing</b>	Sample information files that are currently being used for analysis.
<b>Complete</b>	Sample information files that were used in an analysis that has been completed.
<b>No Analysis</b>	Sample information files that have not been used in an analysis.

## File Name Conventions

---

For sample information files, a default file name (the next available sequence number) and a default extension are displayed when the Files list box is opened. For analysis conditions and report options, only a default extension is displayed with an asterisk (\*.ANC, for example). The following table shows the file name extensions for the AutoChem 2950 HP analysis program.

### *Default File Name Extensions*

File Type	Extension
Sample Information	<b>SMP</b>
Analysis Conditions	<b>ANC</b>
Report Options	<b>RPO</b>
Export to disk (ASCII)	<b>EXP</b>
Export to disk (Grams 32)	<b>CGM</b>
Report to disk	<b>RPT</b>
List to disk	<b>LST</b>
Calibration	<b>CAL</b>
The following types are available for reports saved from the Report window:	
Spreadsheet	<b>XLS</b>
ASCII	<b>TXT</b>
Report	<b>REP</b>

---

## Menu Structure

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All functions for the AutoChem 2950 HP are accessed from the Main menu bar. Brief descriptions of each menu are provided below; refer to the chapter given in parentheses for a detailed description of the options contained on that menu.

<b>File</b>	Allows you to manage sample and parameter files.
<b>Unit &lt;n&gt;</b>	Enables you to perform analyses. A unit menu is displayed for each attached unit.
<b>Report</b>	Enables you to generate and close reports.
<b>Options</b>	Allows you to edit sample, gas, active metal, and environmental defaults; and select data presentation formats.
<b>Windows</b>	Enables you to arrange the windows and icons on your screen. It also displays the names of all open windows.
<b>Help</b>	Provides access to the operator manual



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## 3. OPERATING PROCEDURES

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This chapter contains brief step-by-step instructions on how to:

- specify sample file defaults
- create sample information files
- define parameter files
- prepare the sample
- perform an analysis
- generate a list of information on a sample or parameter file
- export data in a sample information file to an ASCII file
- overlay graphs

This chapter does not contain detailed descriptions of the dialogs used to perform these procedures. Refer to Chapters 5 through 8 for dialog descriptions. Use the index to assist you in locating the appropriate dialog.

---

### Specifying Sample Defaults

---

Sample information files include the information required by the AutoChem 2950 HP to perform analyses and collect data. The analysis program automatically generates sample information file names and assigns default values. Sample defaults allow you to customize your sample file as well as the file name sequence. It is best to use defaults for materials that you most commonly analyze. You can always edit the values when the sample file is created.

The sample information presentation can be presented in a Basic, Advanced, or Restricted format. Sample defaults can be specified in the Basic or Advanced formats only; you cannot specify sample defaults using the Restricted format.

Define parameter files before entering sample defaults (refer to [Defining Parameter Files](#) on page [3-5](#)).

## Advanced Format



Select **Option > Option Presentation** and ensure that **Advanced** is selected as the format.

1. Select **Options > Sample defaults**; the 2950 HP Sample Defaults dialog is displayed.

2. In the **Sequence** field, specify a default string. This is the number that is incrementally sequenced and displays in the **File name** field when you select **File > Open > Sample information**.
3. In the field on the right of the **Sample** line, enter a format for the identification. Be sure to include the **\$** symbol if you want the sample file number included as part of the identification.



You can also edit the word **Sample**. For example, you may prefer to use **Test** or **Material**.

4. Edit the **Operator** and **Submitter** lines or omit these lines by selecting **Omit**.
5. If bar code info is not applicable, select **Omit** to omit this field from the sample information dialog. Or, if you prefer to use this line for some other type of information, edit the label.
6. In the **Mass** field, enter an approximate value for the mass of sample you typically use in your analysis. A more accurate value can be entered in the sample file itself.

- Click the **Analysis Conditions** tab and enter appropriate information for your most commonly analyzed sample material. Remember, you can always edit this information in the sample file when it is created.
- Click the **Report Options** tab and select the reports. Again, this selection can be edited in the sample file when it is created.
- Click **Save**, then **Close**.

## Basic Format



Select **Option > Option Presentation** and ensure that **Basic** is selected as the format.

- Select **Options > Sample defaults**; the Basic Sample Information Defaults dialog is displayed.

- In the **Sequence** field, specify a default string. This is the number that is incrementally sequenced and displays in the **File name** field when you select **File > Open > Sample information**.

3. In the field on the right of the **Sample** line, enter a format for the identification. Be sure to include the \$ symbol if you want the sample file name (sequence number) included as part of the identification.



**You can also edit the word *Sample*. For example, you may prefer to use *Test* or *Material*.**

4. In the **Mass** field, enter an approximate value for the mass of sample you typically use in your analysis. A more accurate value can be entered in the sample file itself.
5. Select the down arrow to the right of the **Analysis Conditions** field to choose a default parameter file. The steps included in this file will display in the window below the field. You may view the details of these steps by selecting **View**.
6. Select the down arrow to the right of the **Report Options** field to choose a default parameter file. The reports chosen (preceded with a check mark) for the selected file will display in the list of reports below the field. Edit selections.
7. Click **Save**, then **Close**.



## Defining Parameter Files

---

Analysis Conditions and Report Options files can exist as part of the sample information file or as an individual parameter file. Having these files exist independently allows you to use them repeatedly. For example, unless you expect each analysis to use unique sets of analysis conditions and report options, you can create a group of standard parameter files containing the values you expect to use most frequently. The contents of these files can be copied and imported into multiple sample information files, providing speed and consistency. After these values have been copied and imported into the sample information file, they can be edited. The original parameter file remains intact and ready for its next use.



**Independent parameter files make it possible for one person (such as a laboratory manager or senior chemist) to create standard parameter sets that can be used easily by others (such as laboratory technicians).**

Several predefined parameter files (located in the PARAMS directory) are included with the AutoChem 2950 HP analysis program. These files are examples only and the gases specified in these files may not match the gases connected to your analyzer. To use one of these files as a starting point, simply create a new file and select **Replace**. A dialog is displayed so that you can select the existing parameter file. After the values from this file are copied into the current file, they may be edited.

Save these files to the directory specified as the Parameter File directory (refer to [Parameter Files Directory](#) on page 8-17 for additional information). If you do not save these files to the parameter file directory, they will not display in the drop-down lists on the Basic sample file dialog.

Parameter files can also be created from the Advanced Sample information window using the **Save as** function on the File menu. Select **Save As > Report Options** or **Analysis Conditions**.

---

## Analysis Conditions

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Analysis conditions provide a rapid and intuitive system for entering an analysis protocol. For a description of the Analysis conditions subdialog boxes and fields, refer to [Analysis Conditions](#) on page [5-9](#).

When specifying analysis temperatures in the analysis conditions, follow the guidelines below.

---

### Starting Temperature

---

Experiments should be started at a temperature that is lower than that at which the reaction begins. If the reaction begins below ambient temperature, you should use the optional CryoCooler.

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### Maximum Temperature

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The experiment's maximum temperature is limited to the temperature at which your sample sinters<sup>1</sup> or melts.

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### Ramp Rate

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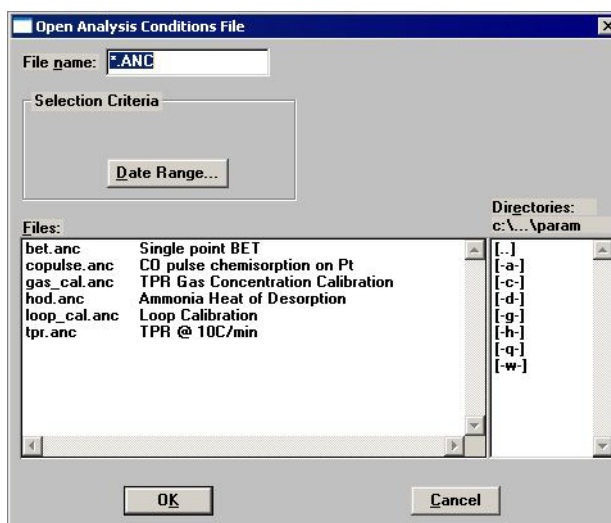
The recommended ramp rate is 5-10 °C/min. However, to use a different ramp rate for various purposes. For example, if you have identified 500 °C as the starting temperature of the reaction being observed, it is more expedient to ramp the temperature rapidly (for example, 50 °C/min.) to a value near 500 °C, then proceed with the experiment at 5-10 °C/min.

A ramp rate of 10 °C/min is commonly used for temperature programmed analyses. Reducing the ramp rate will reduce the rate of reaction and may be beneficial for resolving overlapped peaks. High ramp rates may be used for non-critical heating of the sample to reduce the analysis time.

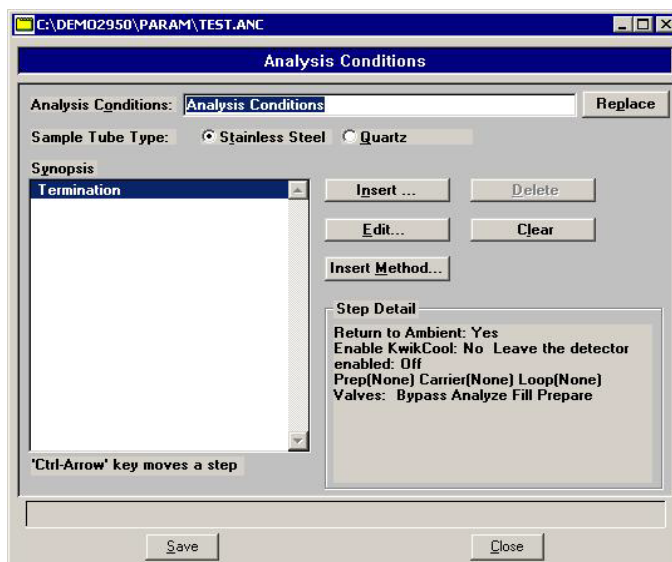
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1. Sintering is the fusing of small particles (or small features of a sample). Sintering tends to reduce the active surface area.

1. Select **File > Open > Analysis conditions**; the Open Analysis Conditions File dialog is displayed.



2. Enter a name for the file (up to eight characters) in the **File name** field, then click **OK**. A verification message is displayed.
3. Click **Yes** to create the file; the Analysis Conditions dialog is displayed.



4. Enter a description in the **Analysis Conditions** field. Use a description that will help you identify the conditions specified.
5. Select the type of sample tube that will be used for the analysis.

Choose **Stainless Steel** for a high pressure analysis. Stainless steel tubes can be used for analyses performed with pressure settings of up to 1000 psia and temperature settings up to 850 °C.



**Do not use stainless steel sample tubes with temperatures above 850 °C\*. Doing so could damage the sample tube.**

\* Published material specifications, which include design safety factors, limit these tubes to 650 °C and 1000 psia. However, extensive testing at 850 °C and 1000 psia of numerous tubes of identical design and material have resulted in no failures. Additionally, as a safety measure, the tube in operation is enclosed in a clam-shell furnace. The furnace incorporates a limit switch which cuts the current and releases the pressure if opened to the point of switch actuation.

Choose **Quartz** for a low pressure analysis. Quartz tubes can be used for analyses performed with pressure settings at ambient and temperature settings up to 1100 °C.



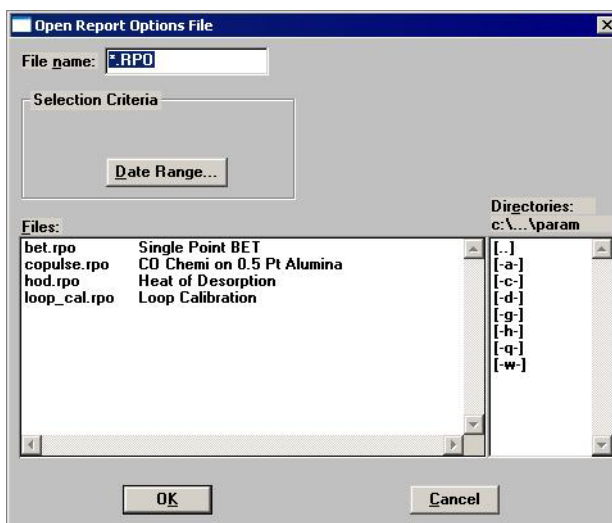
**Do not use quartz sample tubes with pressures significantly above ambient. Doing so could loosen the O-ring seal and allow gas leakage, and could also cause damage to the sample tube.**

6. Click **Insert** to insert steps for the analysis. Each step you insert has an associated dialog, which contains the parameters for that step. You can insert up to 1000 steps in the Synopsis.
7. Choose **Experiment**, or a calibration option, then click **OK**; an associated dialog is displayed.
8. Define the experiment (or calibration), then click **OK**; the experiment (or calibration) will display in the synopsis window.
9. Click **Save**, then **Close**.

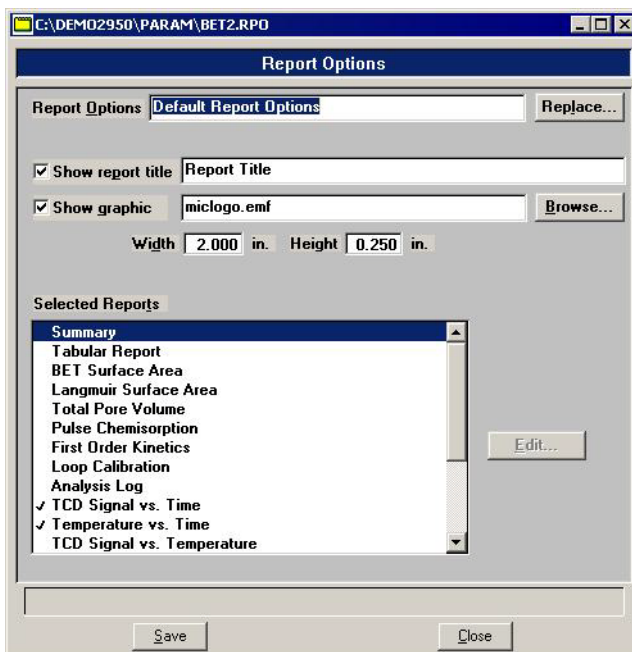
## Report Options

Report options specify which reports will be generated after an analysis. For a description of the Analysis conditions subdialog boxes and fields, refer to [Report Options](#) on page 5-34.

1. Select **File > Open > Report options**; the Open Report Options File dialog is displayed.



2. Enter a name for the file (up to eight characters) in the **File name** field, then click **OK**. A verification message is displayed.
3. Click **Yes** to create the file; the Report Options dialog is displayed.



4. Enter a description in the **Report Options** field. Use a description that will help you identify the types of reports selected.

5. Select **Show report title** and enter the title to appear at the top of the report. Or deselect this option if you do not want a title on your report.
6. If your company logo exists as a bitmap (bmp) or, preferably, an enhanced metafile (emf), you can have it display in the report header, select **Show graphic**. Click **Browse** to select the file; use the Width and Height fields to specify a size.
7. Select (or deselect) the report by highlighting the report and double-clicking. Alternatively, press **Spacebar** after the report is highlighted. A report is selected when it is preceded with a check mark.
8. Edit reports if necessary.
9. Click **Save**, then **Close**.

---

## Creating Sample Information Files

---

Every analysis requires a sample information file. The file consists of information groups which, collectively, identify the sample, guide the analysis and specify the data reduction.

A sample file may be created in any of the three different formats:

- **Advanced**  
Provides complete access to all parts of the sample file, allowing you to quickly edit parameters as required.
- **Basic**  
Presents a single window in which you select predefined parameter files for your analysis. You can also switch to the Advanced format to edit parameters.
- **Restricted**  
Presents a single window in which you select predefined parameter files (identical to the Basic format). You cannot, however, switch to the Advanced format for editing purposes. This format is password-protected.

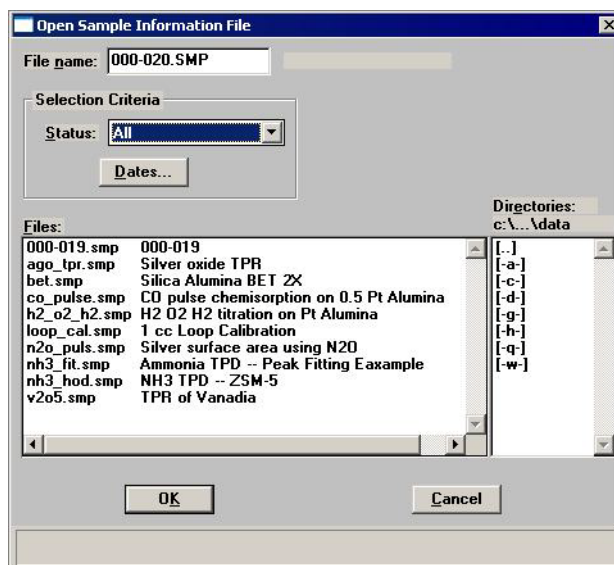
Select **Options > Option presentation** to choose the format.

Sample information files can be created in advance of the analysis or they can be created at the time of analysis, whichever is convenient for you.

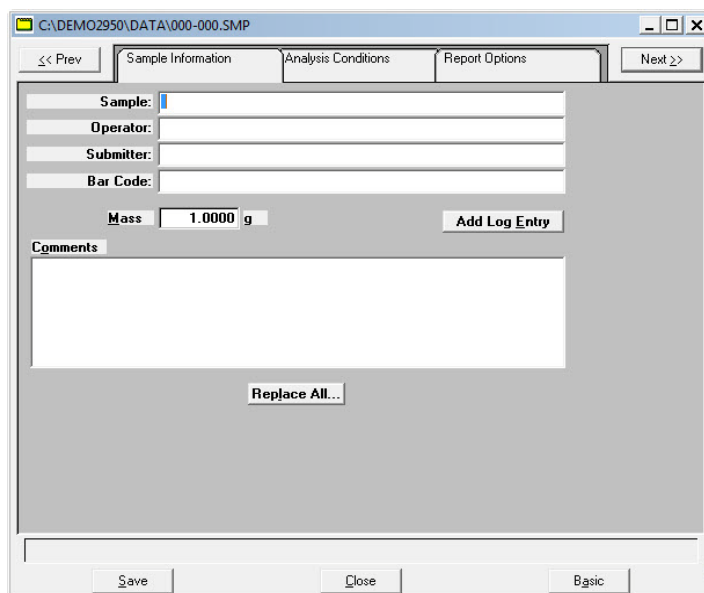
## Advanced Format

The Advanced format presents the sample information editor in a manner similar to that of an index card file. Simply click on the appropriate tab and enter the required information.

1. Select **File > Open > Sample information**; the Open Sample Information dialog is displayed.



2. Accept the next sequenced file number or enter a name for the file (up to eight characters) in the **File name** field, then click **OK**. A verification message is displayed.
3. Click **Yes** to create the file; the Sample Information dialog is displayed.



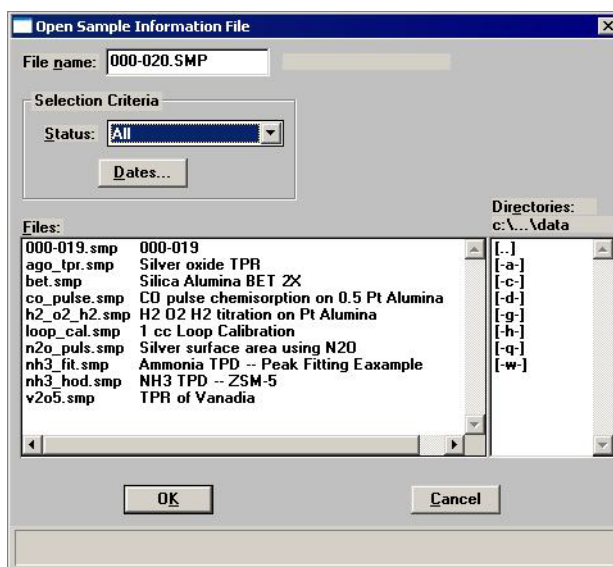


4. In the **Sample** field, enter a description or add to the existing one.
5. Edit the **Operator** and **Submitter** fields if necessary.
6. Enter bar code information (if used). This field will also accept input from a bar code reader (connected to a USB port on the computer).
7. Use the **Comments** field for any comments about the sample or analysis conditions. Anything entered in this field will display in the header of some reports.
8. Click the **Analysis Conditions** tab and define analysis conditions. Refer to [Analysis Conditions](#) on page 3-6 for instructions on completing the dialog.
9. Click the **Report Options** tab and specify report options. Refer to [Report Options](#) on page 3-9 for instructions on completing the dialog.
10. Click **Save**, then **Close**.

## Basic Format

The Basic format allows you to create a sample file by choosing from predefined parameter files. The values in these files can be edited when needed by switching to the Advanced format.

1. Select **File > Open > Sample information**; the Open Sample Information dialog is displayed.



2. Accept the next sequenced file number or enter a name for the file (up to eight characters) in the **File name** field, then click **OK**. A verification message is displayed.

- Click **Yes** to create the file; the Sample Information dialog is displayed.

Basic Sample Information

Sample: CO pulse chemisorption on 0.5 Pt Alumina Replace All...

Mass: 0.6428 g

Analysis Conditions

Analysis Conditions

Report Options

Default Report Options

1.01 Experiment

1.02 Temperature Ramp

1.03 Change Gas Flows

1.04 Temperature Ramp

1.05 Wait

1.06 Temperature Ramp

1.07 Wait

1.08 Start Recording

1.09 Start Repeat

1.10 Dose

1.11 Stop Repeat

1.12 Stop Recording

View

✓ Summary

Tabular Report

BET Surface Area

Langmuir Surface Area

Total Pore Volume

✓ Pulse Chemisorption

First Order Kinetics

Loop Calibration

Analysis Log

✓ TCD Signal vs. Time

Temperature vs. Time

TCD Signal vs. Temperature

Save Close Advanced

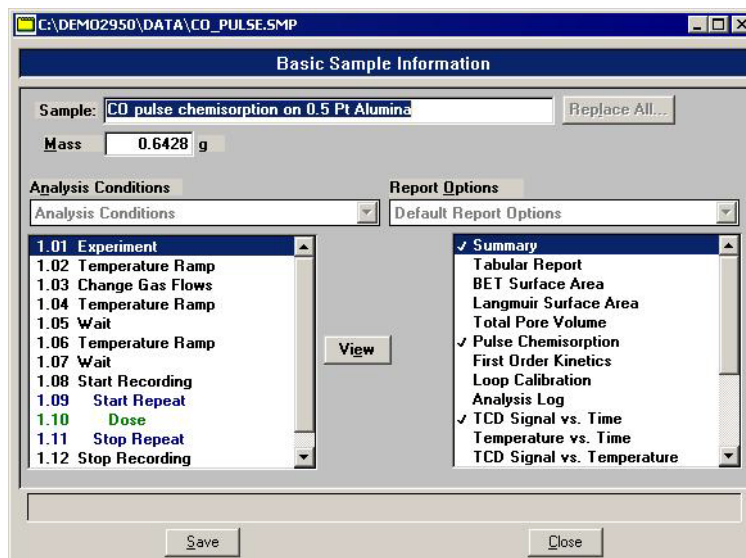


The values contained on the dialog are those specified in Sample Defaults. You can accept these defaults to create the file or continue with the subsequent steps to edit them.

- In the **Sample** field, enter a description or add to the existing one.
- Click the down arrow to the right of the **Analysis Conditions** field and choose the file containing the appropriate analysis conditions. After you choose a file, its steps are displayed in the window below the **Analysis Conditions** field. You can view the details of the steps by highlighting the step and clicking **View**.
- Click the down arrow to the right of the **Report Options** field and choose an appropriate file. You can select additional (or deselect current) reports by double-clicking on the report(s).
- Click **Save**, then **Close**.

## Restricted Format

Sample information files are created using the Restricted format in the same manner as with the Basic format. The name displayed in the title bar even displays as Basic Sample Information. However, the dialog for this format does not allow you to switch to the Advanced format to edit parameters. Certain menu functions are also disabled with this format.



## Preparing for an Analysis

Depending upon the standard procedures at your laboratory, some preparation activities may be performed at the beginning of the day or at the beginning of a shift. Regardless of when you choose to perform them, the following activities must be performed before beginning an analysis:

- If the analyzer has been turned off, allow it to warm up for at least 3 hours after turning on.
- Check all gas regulator pressure settings. (Refer to [Changing Gases](#) on page 9-31.)
- Make sure the sample thermocouple is properly positioned. (Refer to [Selecting the Thermocouple Position](#) on page 3-21.)
- Update the current ambient temperature and pressure. (Refer to [Environmental Defaults](#) on page 8-16.)

## Using the Cold Trap or Delay Path

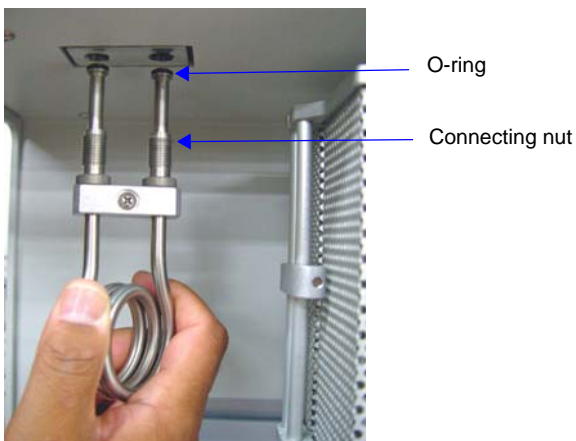
A **cold trap** is used to condense vapors out of the gases flowing through the analyzer before they reach the detector. Specific instructions for using the cold trap for each type of analysis are included in the tutorial (Appendix J, "[TUTORIALS](#)").

A **delay path** may be used to reduce the disturbance of the gas flow caused by large quantities of gas desorbing from the sample, which is often associated with thermal transpiration.

A cold trap or delay path is installed in the set of ports located under the top panel, to the left of the sample tube ports.



1. Make sure the connecting nuts and O-rings are attached as shown below, then insert the open ends of the cold trap (or delay path) into the openings under the upper panel.

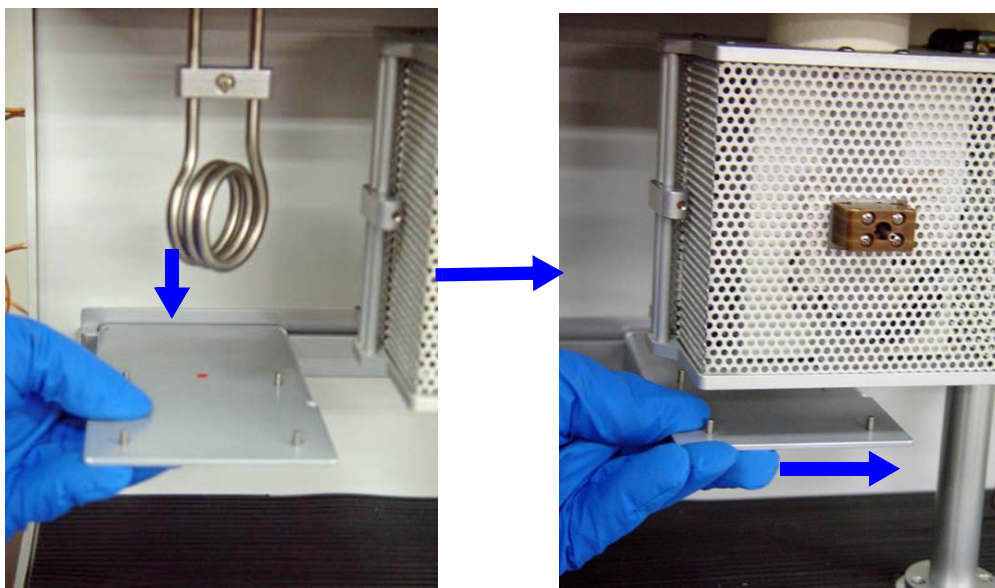


2. Press the cold trap (or delay path) up into the analyzer until it comes to a stop; securely tighten the fittings.



**Do not overtighten the fittings. A sufficient seal is achieved when the fittings are finger-tight.**

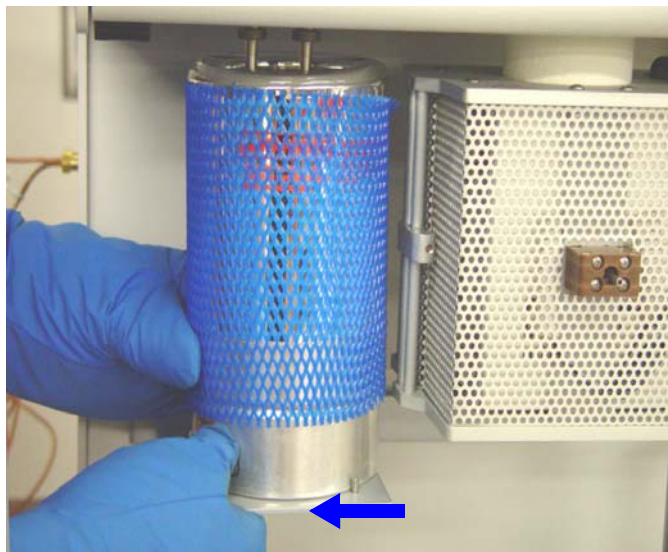
3. To use a Dewar to reduce the temperature of the cold trap, fill the Dewar with the appropriate coolant.
4. Hook the Dewar stand onto the front of the analyzer as shown below, then slide to the right.



5. Carefully place the Dewar under the cold trap, then raise the Dewar up.



6. Slide the stand to the left and place the Dewar on the stand.



## Using a Dewar

### Precautions



**Always handle Dewars with care. Any product incorporating a vacuum is a potential safety hazard and should be treated with caution. Always observe the precautions listed below.**

We recommend the following be observed when handling Dewars containing cryogen:

- Protect yourself by wearing 1) goggles (or a face shield), 2) an insulated or rubber apron, and 3) insulated gloves.
- When pouring cryogen from one container to another: 1) cool the receiving container gradually to minimize thermal shock, 2) pour the cryogen slowly to prevent splashing, and 3) vent the receiving container to the atmosphere.
- Use a plastic stirring rod when stirring substances in a Dewar containing cryogens. Do not use a glass or metal stirring rod unless it is coated with some type of protective coating.
- Do not remove the mesh covering from the Dewar flask. This covering is in place to minimize the risk of flying particles if the Dewar is accidentally knocked over or dropped and broken.



- Do not handle heavy objects above the Dewar. If unavoidable, place a protective cover over the Dewar's opening. If an object of sufficient weight is dropped into the Dewar, it may shatter.

### Mixing an IPA/LN<sub>2</sub> Slurry

An isopropyl alcohol (IPA)/liquid nitrogen (LN<sub>2</sub>) slurry is used to maintain cold trap temperatures of approximately -80 °C. Use the following instructions to mix such a slurry.



**Improperly mixing an IPA/LN<sub>2</sub> slurry could cause injury. If you do not stir the mixture continuously, gas may build up under the surface, causing the liquids to spew out of the Dewar.**

1. Chill a 600-mL Dewar by rinsing it with LN<sub>2</sub>. Allow a small amount of LN<sub>2</sub> to remain in the bottom of the Dewar (approximately 1 cm deep).
2. Stirring constantly, slowly add approximately 500 mL of IPA. For greatest safety, use a laboratory squirt bottle. Squirt the stream of IPA along the inside edge of the Dewar, very near the top, allowing the IPA to flow down the Dewar's inside wall. Do not stop stirring the mixture in the bottom of the Dewar, even if stirring becomes difficult. As you continue stirring and adding IPA, the mixture will loosen and become easier to stir.
3. When all the IPA has been placed in the Dewar, slowly begin pouring LN<sub>2</sub> into the Dewar. Approximately 1 liter is needed. You must continuously stir the mixture as you add the LN<sub>2</sub>. As ice chunks form, break them up and stir them down into the mixture. Avoid splashing. Gently knock ice chunks away from the sides of the Dewar and continue stirring. Add liquid nitrogen until the slurry is within 25 mm (1 in.) of the top of the Dewar.
4. Lift the stirrer out of the slurry and observe as the slurry drips into the Dewar; the stirrer should be thickly coated with slurry.

The goal is to achieve a slurry that has a thick, syrupy consistency without large chunks. When the slurry is cold enough, small pieces of ice may be present (10 - 20% of the mixture). A little ice accumulation along the sides of the Dewar is acceptable.



**The cryogen must not be cold enough to trap (condense) the carrier gas or analysis gas. Do not use liquid nitrogen with a carrier gas containing argon. For example, use an alcohol and liquid nitrogen slurry (-80 °C).**

## Cooling Options

The furnace must cool during temperature-programmed analyses if (1) a low temperature is required as part of the analysis, or (2) to speed cooling of the furnace in order to increase sample throughput, or reduce analysis time.

Cooling options are listed below. To speed the cooling of the furnace to increase throughput, the choice may be one of convenience. If rapid cooling is required for your analysis, the CryoCooler may be employed.

- **Allow the temperature to return to ambient over time**

If time is not a consideration, allow the furnace to cool over time. It may take two hours or more for the furnace to cool from 1100 °C to near ambient. This may be acceptable when time is not a limitation.

The furnace cools more rapidly when it is open. This option requires the availability of an operator to open and close the furnace at the appropriate times during analysis.

- **Connect the KwikCool to the inlet in the front of the furnace**

The KwikCool allows you to ramp the furnace temperature rapidly down near ambient, which reduces the time required to complete analyses and increases throughput.

Instructions for attaching the KwikCool to the furnace are provided in [KwikCool](#) on page 9-48. A KwikCool is included as a standard accessory with every AutoChem 2950 HP.

- **Connect the optional CryoCooler to the inlet in the front of the furnace**

The CryoCooler provides control of the sample temperature from temperatures as low as -100°C. The CryoCooler is required for analyses that include signal recording below ambient. Instructions for attaching the CryoCooler to the furnace are provided in [CryoCooler](#) on page 9-49.



### Safe Operation Guidelines for Stainless Steel Tubes

- External cooling of the tube above 200 °C may be assisted only with air not to exceed 3 cfm @ 30 psi.
- External cooling of the tube below 200 °C may be assisted with the CryoCooler at a rate less than 90 °C/min.
- Do not use other cooling agents below 200 °C because they may embrittle the sample tube.
- Do not submerge the tube in water or oil, or spray the tube with water or oil.

Failure to follow these practices may cause bodily harm and/or damage to the instrument and sample tube. The AutoChem HP has been optimized to use these guidelines for safe operation.



## Protecting the Detector Filaments by Flowing Gas



**A carrier gas must be flowing through the detector whenever the detector filaments are turned on to prevent filament deterioration.**

Flow gas, at a rate of 10 cm<sup>3</sup>/min, through the detector whenever the detector filaments are turned on. Failure to do so may cause the filaments to deteriorate and lose sensitivity. If the gases are shut off, the filaments are automatically turned off after five minutes. Nitrogen, helium, or argon is recommended.

## Selecting the Thermocouple Position

There are three possible configurations for the thermocouple:

- inside the sample tube
- inside the sample tube surrounded by a quartz sheath (for use with quartz sample tubes only)
- outside the sample tube

Best accuracy is obtained when the thermocouple is placed inside the sample tube. In some cases, however, one or more of the gases used during the experiment may react with the Inconel™ material of which the thermocouple is made. A quartz sheath (for use with quartz sample tubes only) is provided to protect the thermocouple when the gases or sample used in the experiment may damage it. In this case, the sheath is placed around the thermocouple and the thermocouple is positioned inside the sample tube. When placed inside the tube, you can adjust the thermocouple so that the end is located approximately 2-3 mm (1/8 in.) above the sample; always try to position the end of the thermocouple as close to the furnace thermocouple as possible.

Positioning the thermocouple along the exterior of the sample tube is less accurate than the other configurations. If the thermocouple is placed outside the sample tube, make sure it is clamped against the sample tube, and that its end does not extend below the point where the sample tube begins to taper.

For instructions on changing the position of the thermocouple refer to [Repositioning, Replacing, or Adjusting the Sample Thermocouple](#) on page 9-14.

## Cleaning Sample Tubes

---

The sample tube should be cleaned (using an ultrasonic cleaning unit) and adequately dried before each analysis to prevent the cross-contamination of samples. The following materials are recommended for cleaning sample tubes:

- Ultrasonic cleaning unit
- Alconox<sup>®</sup> or other suitable detergent
- Latex gloves or lint-free cloth
- Isopropyl alcohol (IPA) is preferred for cleaning; acetone or deionized water may be used if IPA is unavailable
- Nitrogen or helium
- Sample tube brush

Clean the tubes in an ultrasonic cleaning unit as follows:

1. Ensure that the ultrasonic cleaning unit is clean.
2. Using a ratio of 5 grams of Alconox (or equivalent detergent) per 500 mL of warm water, fill the bowl of the ultrasonic unit with enough water to cover the entire sample tube.
3. Ensure that the detergent is dissolved before placing the sample tube into the water. If too much detergent is used, it may be difficult to rinse from the sample tube.
4. Fill the sample tube with warm water and place it in the bowl of the ultrasonic cleaning unit. Turn on the ultrasonic cleaning unit for approximately 15 minutes.
5. Using latex gloves or a lint-free cloth, remove the sample tube from the bowl.
6. Clean the interior of the sample tube with the brush supplied in the accessories kit.



7. Rinse the sample tube thoroughly with hot water. Then rinse it again with isopropyl alcohol, using a waste container to collect used isopropyl alcohol.



**If isopropyl alcohol is unavailable, acetone or deionized water may be used to rinse the sample tube.**

8. Using dry, compressed nitrogen or air, dry the interior of the sample tube. Use a tubing extension long enough and small enough in diameter to fit inside the sample tube.
9. Wipe a stopper with a lint-free cloth; make sure the stopper is dry. Place the stopper on the sample tube. Label the sample tube and stopper for identification.

## Preparing the Sample

---

In order to obtain the most repeatable, accurate results, you should:

- Use approximately the same amount of sample for each analysis (especially if you compare data from sample to sample).
- Make sure the sample is well dispersed in the tube, not against the side or walls of the tube.
- Make sure the material's particle size distribution remains fairly constant from sample to sample. (Large particles of a substance reduce at a different rate than small particles of the same substance.) For samples with a wide particle size distribution, care should be taken that each sample is representative of the entire lot's distribution.

Micromeritics recommends sample masses between 20 mg and 2 g. When determining a sample amount, consider that the sample must be contained within the bottom 20 mm (3/4 in.) of the sample tube.

When determining sample size, consider the percentage of active metals in your sample. Materials with lower percentages of active metals may require larger sample amounts for analysis, while materials with high percentages of active metals can be analyzed using smaller sample amounts.



**Larger amounts of very fine powders may become densely packed in the sample tube restrict, and ultimately block, the gas flow. The resulting increase in gas pressure may force some of the sample out of the sample tube, causing contamination of, or damage to, the analyzer. A possible solution is to expand the sample bed with quartz wool.**

## Determining the Mass of the Sample

1. Remove the stopper from the wide opening of the sample tube and place a small amount of quartz wool into the clean, empty sample tube. A small quantity that provides a 2 to 3  $\mu\text{m}$  filter is adequate for most analyses. Press the quartz wool firmly into place.



2. Replace the stopper.
3. Place the weighing support onto the balance. Zero the balance and allow it to stabilize.



**A form to help you determine the sample's mass is provided in Appendix A. Make copies of this form and use it for your convenience.**

4. Place the sample tube (with stopper) onto the sample weighing support. If you are using the Sample Mass form included in Appendix A, record the value as the mass of the empty sample tube.



5. Remove the sample weighing support and sample tube set (tube and stopper) from the balance.
6. Place a sample container onto the balance. Zero the balance and allow it to stabilize.
7. Slowly add the sample to the container. This step enables you to control the size of the sample before it is poured into the prepared sample tube. Do not record the sample mass at this point.
8. Remove the stopper from the tube. Carefully pour the sample from the container into the sample tube. Use a pipe cleaner to clean the walls of the sample tube before weighing the sample.
9. Replace the stopper on the sample tube.
10. Determine the mass of the sample tube (with stopper) and sample. If you are using the Sample Mass form from Appendix A, record the value as the **Sample plus sample tube mass**.
11. Subtract the sample tube mass from the sample plus sample tube mass. The result is the sample mass.

For some types of analyses, the sample mass measured after analysis is preferred, because many samples contain elevated levels of contaminants and/or moisture which are included in the sample's mass before degassing. This additional mass may affect the analysis results.

For TPR analyses (or other analyses involving small sample quantities), it may be helpful to weigh the sample in a tared container, transfer it to the sample tube, then reweigh the empty sample container after you transfer the sample to the tube. If there is a detectable amount of sample remaining in the container, subtract its mass from that of the sample in the tube.

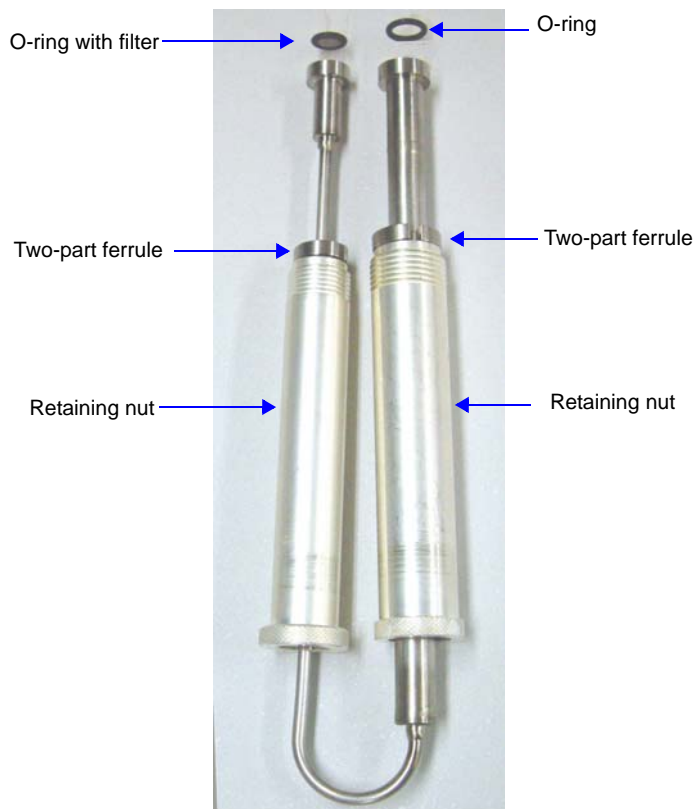
## Performing an Analysis

### Installing the Sample Tube on the Analysis Port



**Make sure the furnace has cooled to ambient temperature before installing tubes. It is a good practice to wear the cotton gloves provided with the instrument when installing sample tubes.**

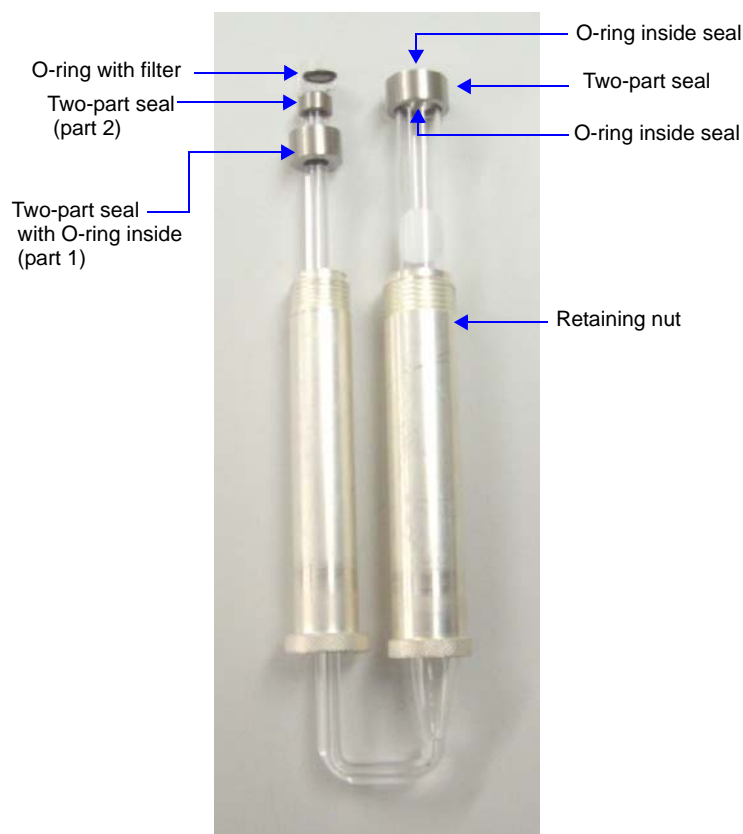
1. Remove the stopper from the sample tube immediately before connecting the sample tube to the analyzer.
2. Assemble the sample tube components as shown in Assembly A or Assembly B below.



*Assembly A: Metal Tube*



**Use only Kalrez™ (or equivalent) O-rings. Kalrez is rated for both chemical and temperature suitability in this application. O-rings of other materials could burn, melt, or decompose.**



*Assembly B: Quartz Tube*



**For quartz tubes: be careful not to allow the nuts to slide rapidly down the tube and into the curved portion of the tube, or the tube may be damaged.**

3. Insert the open ends of the sample tube into the openings under the upper panel.

If the thermocouple is positioned inside the tube, its end should generally be located approximately 2-3 mm (1/8 in.) above the sample surface. In some cases, you may allow the thermocouple to extend into the sample.

If the thermocouple is positioned outside the tube, its end should not extend below the point where the sample tube begins to taper.

When determining the length of the thermocouple, take into account the volume of the quartz wool and sample in the tube.

If you need to increase or decrease the length of the sample thermocouple, refer to [Adjusting the Length of the Thermocouple](#) on page 9-21.



- Press the sample tube up into the analyzer until it comes to a stop. Tighten the fittings by turning the sample retaining nuts to the right until they are finger-tight.

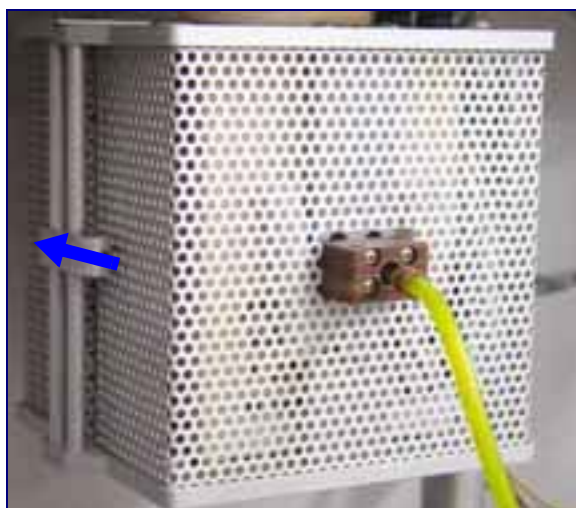


**Do not overtighten the analysis port fittings. Doing so may break a quartz sample tube. The O-rings are sealed when the fittings are finger-tight.**

If the thermocouple is placed on the outside of the sample tube, clamp it to the tube.



- Close and latch the furnace.



**The furnace will not heat unless it is closed. Also, the pressure controller will be disabled when the furnace is open.**

## Starting the Analysis

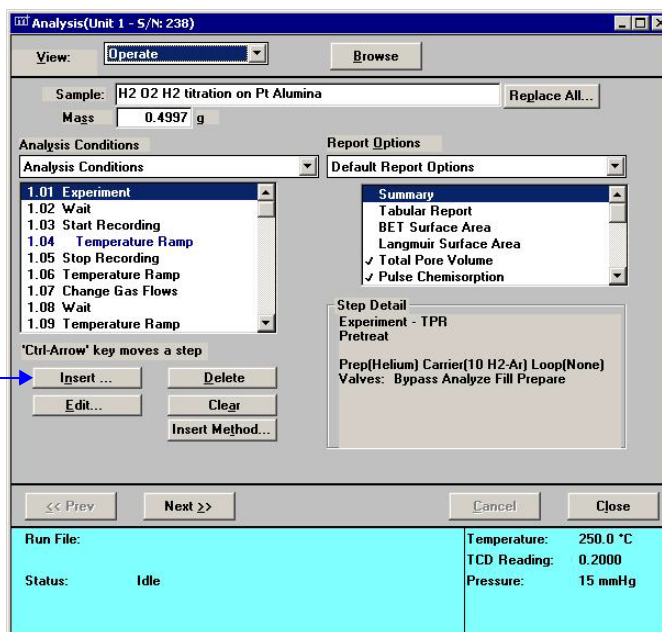
Before starting your analysis, make sure:

- the correct gases are connected and that the Gas Selections dialog (Unit menu) reflects the current gas connections
- the sample has been prepared and the sample tube installed on the analyzer.

If you are performing a loop calibration or some other test that requires injecting gas into the septum, refer to [Using the Septum](#) on page 3-33.

1. In the 2950 HP analysis program, select **Unit > Start analysis**; the Sample Analysis dialog is displayed with the Open Sample information dialog positioned on top.
2. Choose a file for your analysis (or accept the default to create a new one) and click **OK**; the Analysis dialog containing the parameters of the selected file (or the defaults) is displayed.

This group of buttons is not displayed in Basic or Restricted formats; you cannot edit parameters when using these modes.



3. Review and verify analysis conditions and report options; edit if necessary.

4. Click **Next**; the second view of the analysis dialog is displayed. From the drop-down list(s), select the calibrations to be associated with each experiment (if any).

The screenshot shows the 'Analysis(Unit 1 - S/N: demo)' dialog box with the 'View' set to 'Operate'. The main area is titled 'Select a calibration for each experiment.' and contains a table with columns 'Exp.', 'Type', 'Name', and 'Calibration'. The first row is '1. Level Cal. <Description>' with 'None' selected in the 'Calibration' dropdown. Below the table are navigation buttons: '<< Prev', 'Next >>', 'Cancel', and 'Close'. At the bottom, a status bar displays: 'Run File: C:\DEMO2950\DATA\000-027.SMP', 'Sample: 000-027', 'Status: Idle', 'Temperature: 250.0 °C', and 'TCD Reading: 0.2000'.

Exp.	Type	Name	Calibration
1.	Level Cal.	<Description>	None

<< Prev   Next >>   Cancel   Close

Run File: C:\DEMO2950\DATA\000-027.SMP   Temperature: 250.0 °C  
Sample: 000-027   TCD Reading: 0.2000  
Status: Idle

5. Click **Next**; the third view of the analysis dialog is displayed. Specify report output options.

The screenshot shows the 'Analysis(Unit 1 - S/N: demo)' dialog box with the 'View' set to 'Operate'. The main area contains report output options: a checked checkbox for 'Report After Analysis', a 'Number of Reports' spinner set to '1', a 'Destination' dropdown set to 'Screen', and a 'File name' text box containing 'C:\DEMO2950\\*.RPT'. Navigation buttons at the bottom are '<< Prev', 'Next >>', 'Cancel', and 'Close'. The status bar at the bottom is identical to the previous screenshot: 'Run File: C:\DEMO2950\DATA\000-027.SMP', 'Sample: 000-027', 'Status: Idle', 'Temperature: 250.0 °C', and 'TCD Reading: 0.2000'.

☒ Report After Analysis

Number of Reports: 1

Destination: Screen

File name: C:\DEMO2950\\*.RPT

<< Prev   Next >>   Cancel   Close

Run File: C:\DEMO2950\DATA\000-027.SMP   Temperature: 250.0 °C  
Sample: 000-027   TCD Reading: 0.2000  
Status: Idle

6. Click **Next** to review the safety information.

**Analysis(Unit 1 - S/N: demo)**

View: Operate

**Warning:** A dangerous condition can occur when incompatible gases are combined inside the analyzer or when the exhausts are improperly vented.

1. Check the Analysis Conditions in the sample file to make sure that dangerous combinations of gases within the analyzer are not created.  
You may press the Prev button to get back to the screen which shows the Analysis Conditions, you can edit the conditions there.
2. Check the Gas Selections dialog (Unit menu) to make sure that the gas connected to each port is correctly identified.
3. Check the method for venting exhausts to make sure that it is safe for all the products of the analysis.

For additional information, see the "Purging Gases", "Identifying Gases/Ports in the Software", or "Venting Exhausts" topics in the Operator's Manual.

Note: The "Start" button submits this sample to the instrument for analysis.

<< Prev Start Cancel Close

Run File:	C:\DEMO2950\DATA\000-011.SMP	Temperature:	250.0 °C
Sample:	000-010	TCD Reading:	0.2000
Status:	Idle	Repeat Count:	1

7. Click **Start** to begin the analysis. A dialog allowing you to monitor the progress of the analysis is displayed.

**Analysis(Unit 1 - S/N: demo)**

View: Operate Browse

Sample: 000-018 Replace All...

Mass: 1.0000 g

**Analysis Conditions**

Analysis Conditions

- 1.01 Experiment
- 1.02 Wait
- 1.03 Start Recording
- 1.04 Temperature Ramp
- 1.05 Stop Recording
- Done

**Report Options**

Default Report Options

- Summary
- Tabular Report
- BET Surface Area
- Langmuir Surface Area
- Total Pore Volume
- Pulse Chemisorption

**Step Detail**

Experiment - TPR  
ref 1

Prep(Argon) Carrier(Helium) Loop(None)  
Valves: Trap Analyze Fill Prepare

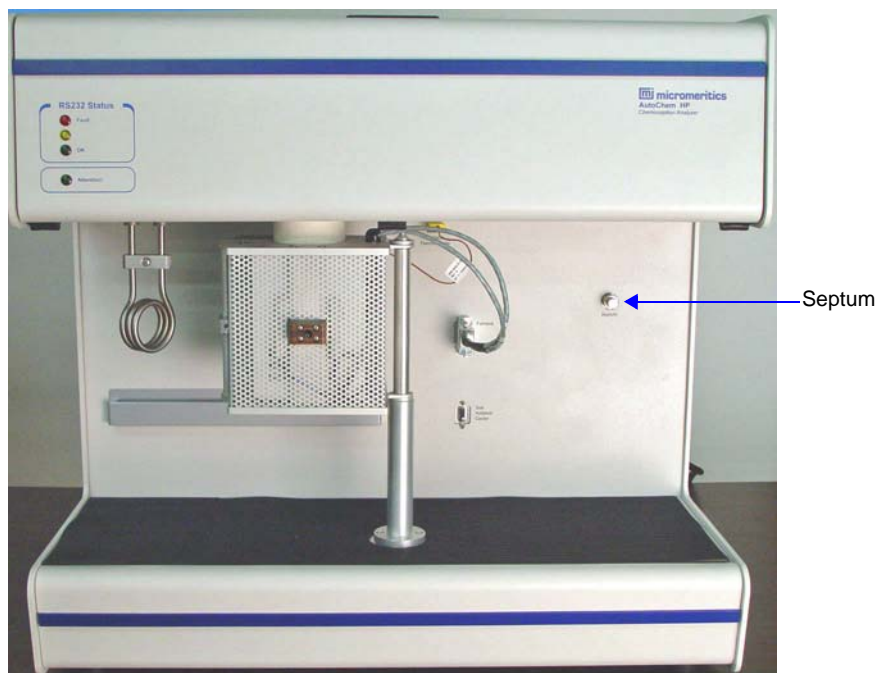
<< Prev Next >> Cancel Close

Run File:	Temperature:	250.0 °C
Status:	TCD Reading:	0.2000

You can also use this dialog to edit any step after the current one. Simply suspend the analysis; this enables the editing buttons. After editing is completed, resume the analysis.

## Using the Septum

The septum is used with a syringe to inject quantities of gas into the analyzer, such as during a loop calibration. It should be used for work at atmospheric pressure only. The septum is located in the right side of the recessed portion of the front panel.



**Do not use the septum at pressures above ambient. Gas may escape at elevated pressures.**

Data accuracy is diminished when you use poor techniques for injecting gas through the septum. The following guidelines should help you maximize the accuracy of your data.

Injecting the gas through the septum causes a peak to appear, but it also causes a disturbance in the flow of gas through the analyzer. This disturbance is visible in the peak data. You can minimize this disturbance by injecting the gas more slowly into the septum. However, prolonging the injection causes the peak to spread.

Develop an injecting method that balances the disturbance with the need for sharper peaks. Use the Peak Editor (described on page 7-16) to adjust peak data to reduce the effects of disturbance.

## Filling the Syringe with Gas

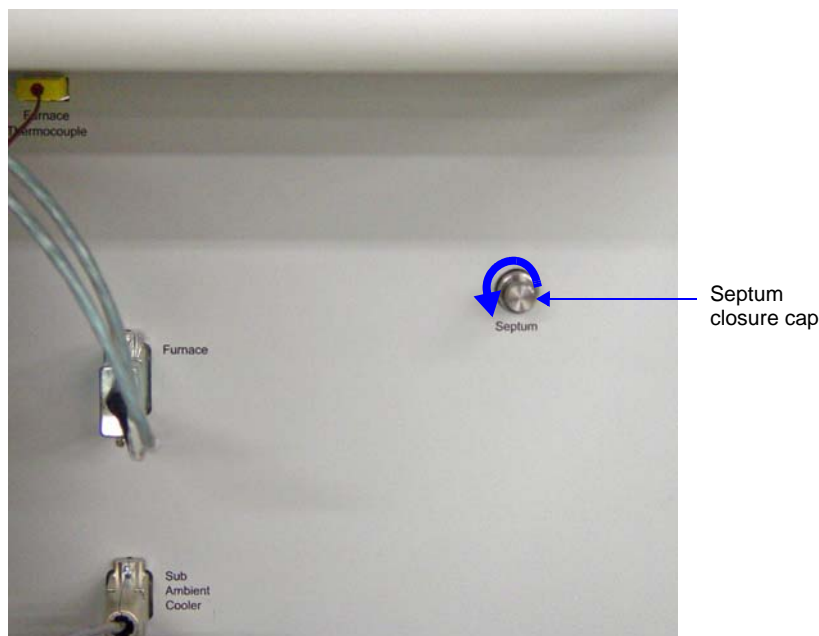
- Always hold the syringe by its metal parts away from the needle. Holding the syringe by the glass allows your body heat to affect (reduce) the quantity of gas in the syringe.
- To fill the syringe: first empty it completely, then insert it into a septum accessory installed on the gas regulator. Draw the syringe plunger back until the syringe is completely filled with gas. Remove the syringe from the septum, and allow it to return to room temperature as described above. Then, press the plunger into the syringe until the correct amount of gas is contained in the syringe.
- An alternative way to fill the syringe with nitrogen (evaporating from liquid nitrogen), is to empty the syringe, then hold the tip of the needle just above the surface of a quantity of liquid nitrogen. Draw the syringe plunger back to fill the syringe with the gas. Allow the syringe to stabilize at room temperature, then push the plunger into the syringe until the correct amount of gas is contained in the syringe.

## Injecting Gas into the Analyzer



**The system must be at atmospheric pressure to safely use the septum. Attempting to use the septum at a higher pressure may cause the septum to eject from the analyzer, resulting in damage to the septum and, possibly, bodily injury.**

1. Turn the septum closure cap counterclockwise to loosen, then remove the nut.



2. Insert the needle fully into the septum.



3. Inject the entire quantity of gas that you have measured into the septum. (Press the plunger into the syringe completely.)
4. Remove the syringe.
5. Replace the septum cap and tighten it finger-tight.



## Removing the Sample Tube



**The furnace and sample tube may remain very hot for some time after analysis has ended. The sample tube retaining nuts are extremely hot, approximately 110 °C. Wear the cotton gloves provided with the instrument and use extreme caution!**

It is recommended that you allow the furnace and sample tube to return to ambient temperature before touching them.

1. If the furnace is closed around the sample tube, unlatch and open it.



**For quartz tubes, support the sample retaining nuts so that they do not fall rapidly out of the analyzer. This may cause damage to a quartz sample tube.**

2. Loosen the sample retaining nuts, being very careful to support them. As you loosen the nuts, also support the sample tube and pull it gently down out of the analyzer. When the nuts are sufficiently loose, the sample tube, retaining nuts, ferrules, and O-rings should slide out of the analyzer.





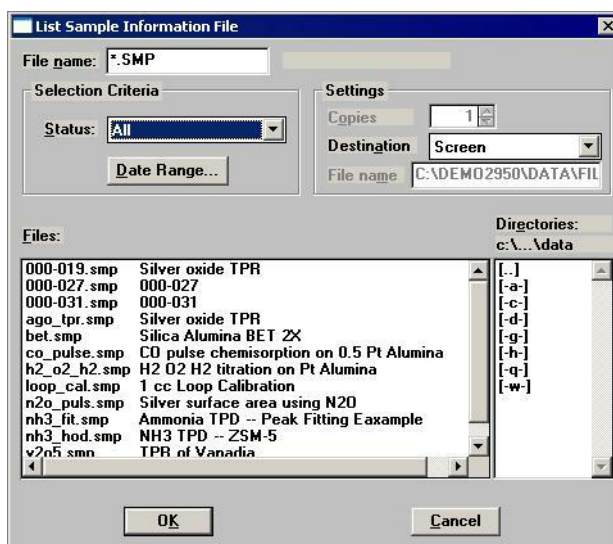
## Listing File Statistics

You can generate a list of the following information on one or more sample or parameter files:

- File name
- Date the file was created (or last edited)
- Time the file was created (or last edited)
- File identification
- File status (sample information files)

Perform the following steps to generate a list:

1. Select **File > List**.
2. Select the type of file; a dialog similar to the one shown below is displayed.



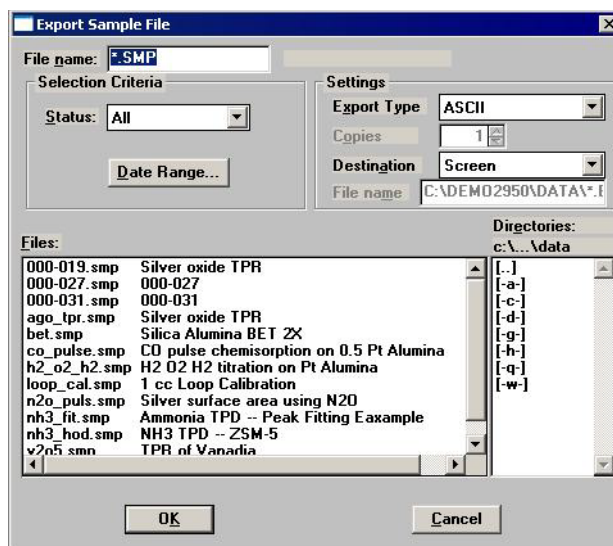
3. From the **Files** list box, choose the file(s). To include all files in the list, leave all files deselected.
4. At the **Destination** field, click on the down arrow and choose an output destination. If you choose File as the destination, enter a name in the **File name** field (or accept the default).
5. Click **OK**; a list for the requested file(s) is sent to the specified destination.

## Exporting Data from a Sample Information File

Data from a sample information file can be exported in ASCII or Grams/32 format. ASCII data can then be imported into other applications, such as spreadsheets. Grams/32 data can be imported into the Grams/32 program (a peak-editing program used with earlier versions of the AutoChem software) and manipulated independent of the AutoChem 2950 HP analysis program. Refer to Appendix D, **FORMAT OF EXPORTED DATA** on page **D-1** for a description of exported data.

Perform the following steps to export a sample information file:

1. Select **File > Export**; the Export Sample File dialog is displayed.

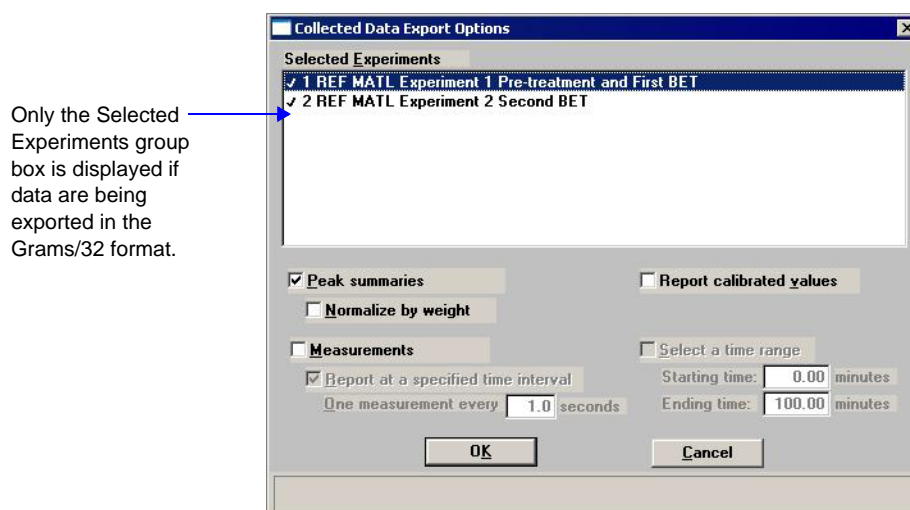


2. From the **Files** list box, choose the file(s) to export. You can choose multiple files by holding down **Ctrl** and clicking on the files.
3. From the **Settings** group box:
  - a. Choose whether you are exporting in ASCII or Grams/32 format.
  - b. Choose a destination for your exported file.

If you choose **File**, enter a name in the **File name** field (or accept the default). If you choose **Printer**, the **Copies** field is enabled allowing you to print up to 4 copies.

You must choose **File** as the destination when exporting in Grams/32 format.

- Click **OK**; the Collected Data Export Options dialog is displayed.



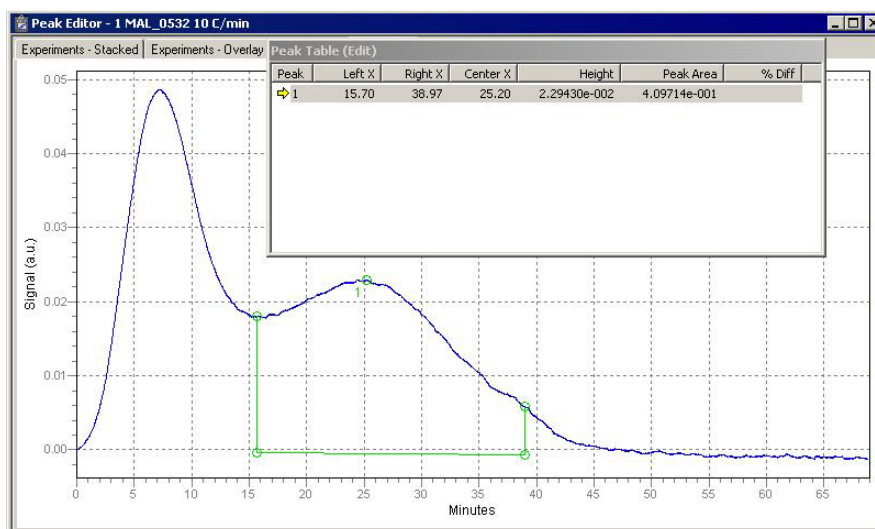
- Choose the experiments to export. Then select any other options. Refer to [Export](#) on page [5-57](#) for additional information on this dialog.
- Click **OK**; data are exported to the specified destination.

## Creating and Assigning a Calibration File

After you have completed an analysis, you can edit the peaks to create a calibration file and assign the file to an experiment.

### Editing the Peaks

1. Select **Reports > Peak Editor** from the main menu. The Edit Sample Peaks dialog is displayed.
2. Select the sample file and click **OK**; the Peak Editor, Stacked view dialog is displayed. This view of the dialog displays all calibrations and experiments contained in the file, stacked one after the other.
3. Click a calibration experiment; the name of the experiment displays in the title bar of the dialog.
4. Click the **Edit Peaks** tab; the Peak Editor dialog and table for the selected calibration are displayed.



5. Right-click in the graph area and select **Find All Peaks**; this option finds all peaks and marks them.



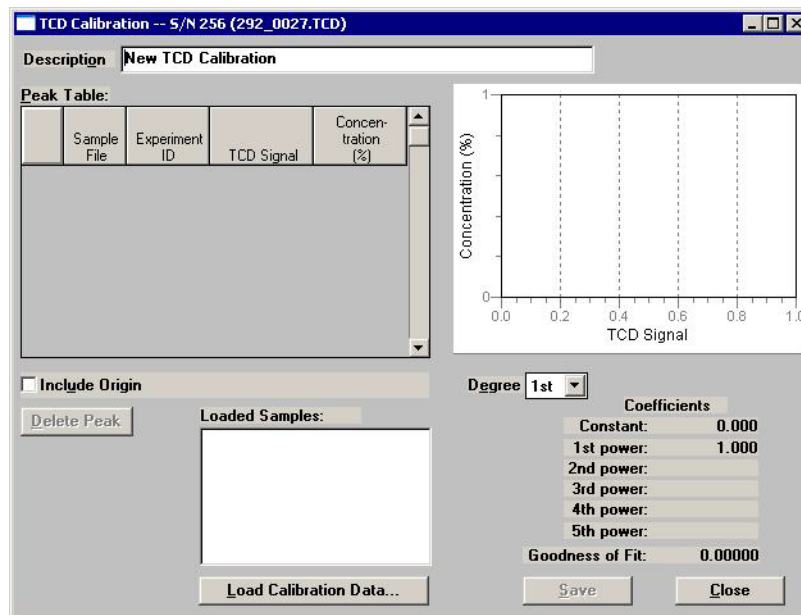
You can further define peaks. Select the peak (the baseline area of the peak displays in green when selected), right-click, and use the **Mark Left Edge** and **Mark Right Edge** options.

6. Right-click and select **Save**.

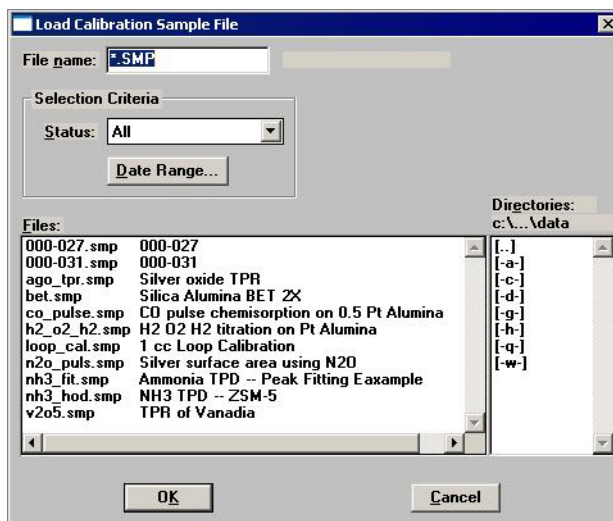
## Creating the Calibration File

After you have defined calibration peaks, you can create the calibration file.

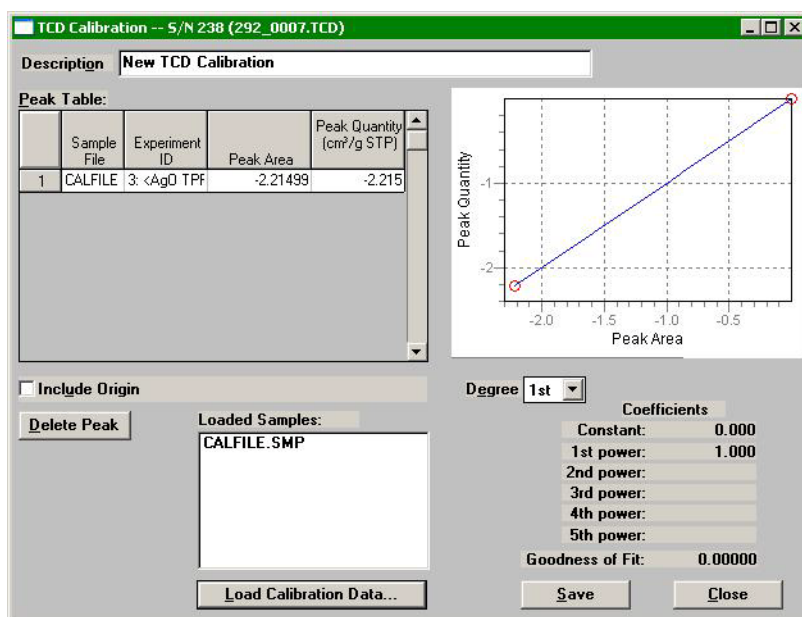
1. Select **Unit > TCD Calibration > New**; the TCD Calibration dialog is displayed.



2. Click **Load Calibration Data**; the Load Calibration Sample File dialog is displayed.



3. Select the sample file containing the calibration data, then click **OK**.



4. In the TCD Calibration dialog, enter an intuitive description in the **Description** field. It is important that you use a description that you will easily recognize.
5. Use the **Delete Peak** button to delete from the Peak Table any data not associated with your calibration experiment.
6. Using the formula given below, calculate the peak volume for each peak in the table; enter the results into the appropriate fields.

$$V_{inj} \times \frac{273.15}{T_{amb} + 273.15} \times \frac{P_{amb}}{760 \text{ mmHg}} = V_{Peak}$$

**Example:** Calculations for 1cm<sup>3</sup> injection, ambient temperature of 22 °C, ambient pressure of 740 mmHg

$$1 \times \frac{273.15}{22 + 273.15} \times \frac{740}{760 \text{ mmHg}} = 0.901 \text{ cm}^3$$

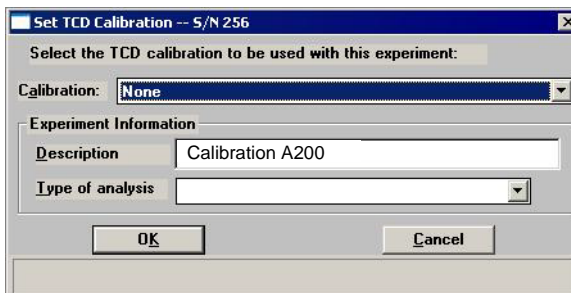
7. Click **Save** to save the file, then **Close** to close the dialog.

Repeat steps 1 through 7 for each gas concentration in the calibration. For example, if you have gas concentrations of 10%, 30%, and 20%, you should mark your calibration peaks and create calibration files for all three concentrations; a total of three files.

## Assigning the Calibration File to the Experiment

Now you may assign the calibration to the appropriate experiment.

1. Click back into the Peak Editor dialog and select the **Experiments-Stacked** tab.
2. Click a sample experiment. Remember, you can view the experiment name in the title bar.
3. Click the **Edit Peaks** tab; the Edit Peaks dialog and table for the selected sample experiment are displayed.
4. Right-click in the graph area and select **Find All Peaks** from the shortcut menu.
5. Right-click in the graph area and select **Set Calibration** from the shortcut menu; the Set Calibration dialog is displayed.



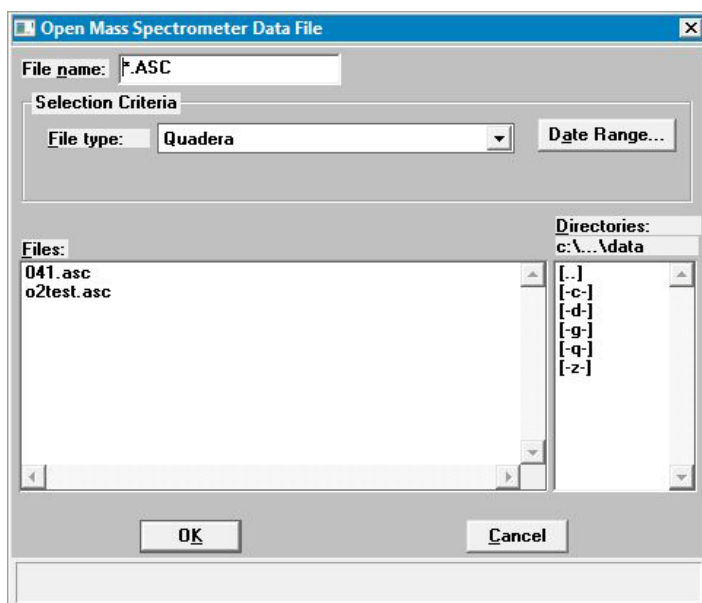
6. Click on the down arrow to the right of the Calibration field and choose the calibration file you created for the concentration of the selected experiment. Ensure that the carrier gas and flow rates match for the experiment and calibration.
7. Right-click in the graph area and select **Save** from the shortcut menu.

## Creating and Assigning a MS Calibration File

After completing the analysis, data collected can be imported with a ThermoStar or MicroStar Mass Spectrometer, create a calibration file, and assign the file to the experiment.

### Importing the MS Data

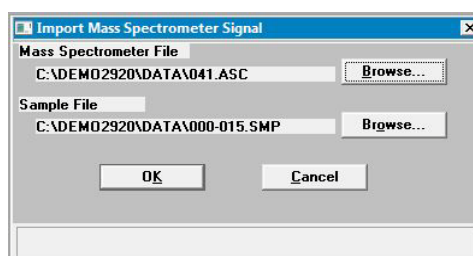
1. Select **File > Import MS Signal**; the Open Mass Spectrometer Data File dialog is displayed.



2. Select the software used to generate the file from the **File type** drop-down list and click **OK**; the Import Mass Spectrometer Signal dialog is displayed.

Displays file and directory path of the file you are importing. \_\_\_\_\_

Displays the next sequenced file number (default) and the directory



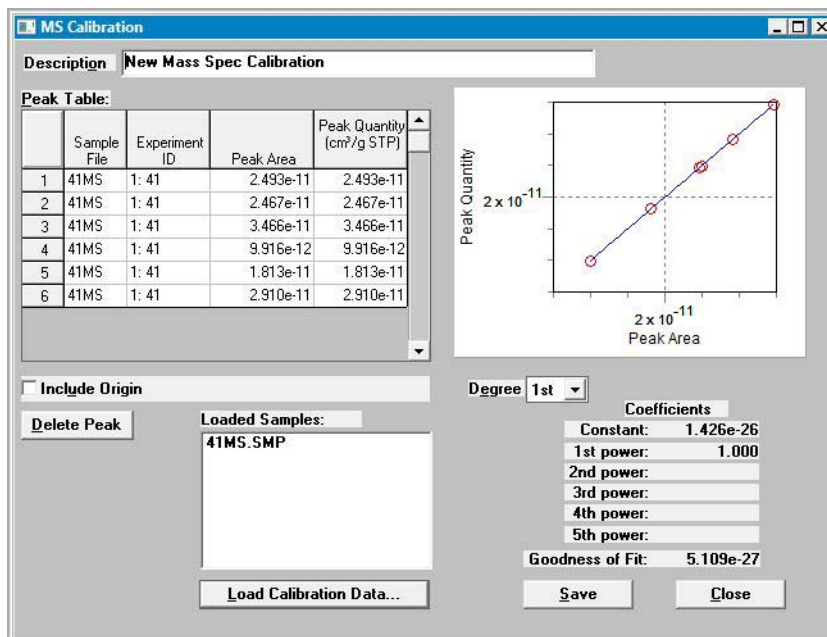
You can click **Browse** to the right of the **Sample File** field to rename the file or save into a different directory.

3. Click **OK**; the file is created and stored as specified.



## Creating the Calibration File

1. Select **File > MS Signal Calibration > New**; the MS Calibration dialog is displayed.



2. Enter an intuitive description in the **Description** field. It is important that you use a description that you will easily recognize.
3. Click **Load Calibration Data**; the Load Calibration Sample File dialog is displayed.
4. Select the sample file you created when importing the MS data and click **OK**; the MS Calibration dialog is populated with the data (shown above).
5. Delete from the Peak Table any data not associated with your calibration experiment.
6. Using the formula given below, calculate the peak quantity for each peak in the table; enter the results into the appropriate fields.

$$V_{\text{syrrinj}} \times \frac{273.15}{T_{\text{amb}} + 273.15} \times \frac{P_{\text{amb}}}{760 \text{ mmHg}} = V_{\text{Peak}}$$

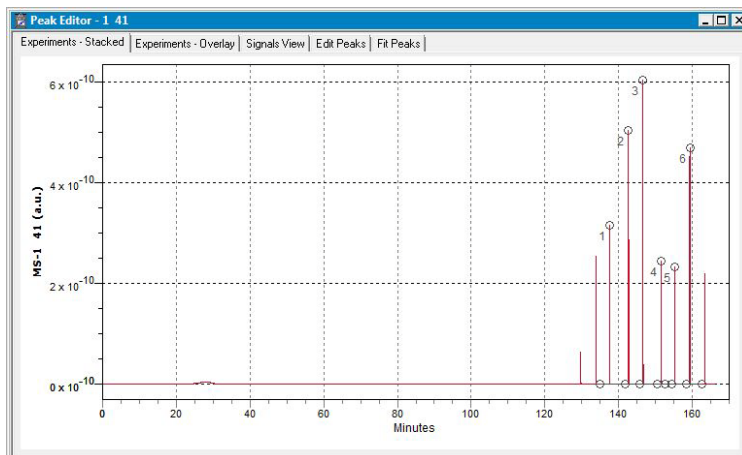
**Example:** Calculations for 1 cm<sup>3</sup> injection, ambient temperature of 22 °C, ambient pressure of 760 mmHg:

$$1 \times \frac{273.15}{22 + 273.15} \times \frac{740}{760 \text{ mmHg}} = 0.901 \text{ cm}^3 \text{ STP}$$

7. Click **Save** to save the file, then **Close** to close the dialog.

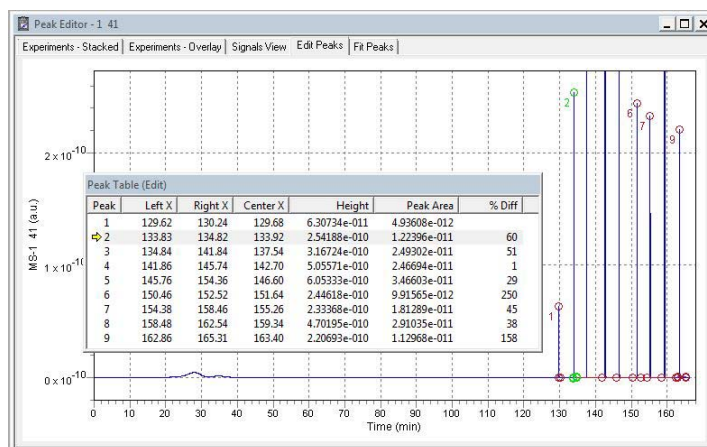
## Editing the Peaks

1. Select **Reports > Peak Editor** from the main menu.
2. Select the sample file and click **OK**; the Peak Editor, Stacked view dialog, is displayed.



This view of the dialog displays all calibrations and experiments contained in the file, stacked one after the other.

3. Click a calibration experiment; the name of the experiment displays in the title bar of the dialog.
4. Click the **Edit Peaks** tab; the Edit Peaks dialog and table for the selected calibration are displayed.



Peaks should be in a positive position before integrating. If you have any negative peaks: right-click in the graph area, choose *View, Display* and click the *Invert* option

5. Right-click in the graph area and select **Find All Peaks**; this option finds all peaks and marks them.



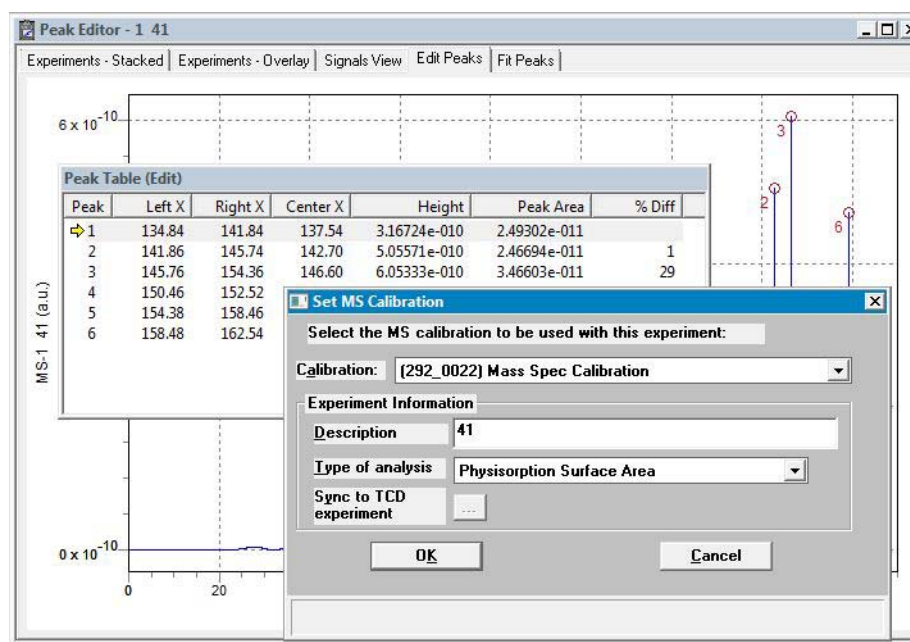
Peaks can be further defined. Select the peak (the baseline area of the peak displays in green when selected), right-click, and use the *Mark Left Edge* and *Mark Right Edge* options.

6. Right-click and select **Save**.

## Assigning the Calibration File to an Experiment

Now you must assign the calibration to the appropriate experiment.

1. Click back into the Peak Editor dialog and select the **Experiments-Stacked** tab.
2. Click a sample experiment. Remember, you can view the experiment name in the title bar.
3. Click the **Edit Peaks** tab; the Edit Peaks dialog and table for the selected sample experiment are displayed.
4. Right-click in the graph area and select **Set Calibration** from the shortcut menu; the Set MS Calibration dialog is displayed.



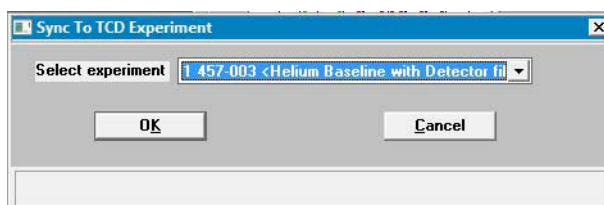
5. Click on the down arrow to the right of the **Calibration** field and choose the calibration file you created for the experiment. Be sure that the Experiment and the Calibration match.

6. Select the type of analysis from the drop-down list. If you select **Pulse Chemisorption**, perform the following steps before proceeding.



**When mass spec data are imported, some parameters (loop temperature, physical injection volume, active concentration, and active metals table) used to generate the pulse chemi report are unavailable and remain at default values. These parameters must be copied into the experiment using the Sync to TCD experiment option.**

- a. Click the **Sync to TCD experiment** ellipsis (...) button; the Load Pulse Chemisorption Experiment dialog is displayed.
- b. Select the sample file associated with the current mass spec experiment, then click **OK**; the Sync to TCD Experiment dialog is displayed.



- c. Click the down arrow and select the experiment and click **OK**.
7. Click **OK** on the Set MS Calibration dialog to close it and return to the Peak Editor.
  8. Right-click in the graph area and select **Save** from the shortcut menu.

## 4. SOFTWARE AND SETUP MODIFICATIONS

The analyzer allows other computer programs to run while an automatic operation is in progress. The Help menu provides access to this operator manual and tutorials on using the software.

Report options can be specified when creating the sample information file. When running an analysis, data gathered during the analysis process are compiled into the predefined reports. Reports can also be defined and generated after an analysis has been run. Each selected report is displayed on its own tab and reflects data collected during the analysis.

### Software Setup

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The Setup program is located on the installation CD and is used to reinstall the software and make analyzer changes - such as adding or removing a unit, etc..



If the computer is to be connected to a network, a second Ethernet port on the computer must be used for that purpose.



If the IP address needs to be changed on the computer connected to the analyzer, refer to the computer's operating system manual or the internet for instructions. The IP address for the computer and the IP address specified in the setup program must match. The IP address must be 192.168.77.100.

### Uninstall the Software

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When the software is uninstalled, only the files required to run the application are removed. Parameter files, sample files, reports, calibration files, and data files are not removed.

To uninstall the software, double click the uninstall.EXE file located in the software installation directory, then follow the screen prompts.

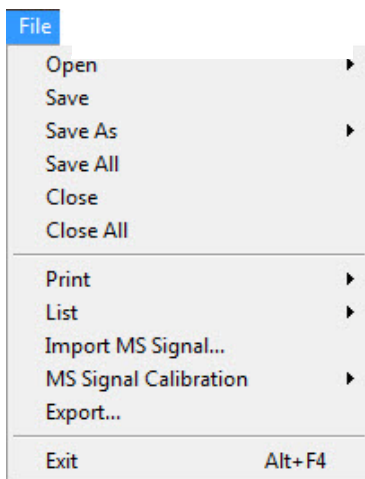


## 5. FILE MENU

The File menu contains options that allow you to manage sample and parameter files.

### Description

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Listed below are brief descriptions of the File menu options. Detailed descriptions follow this section.

<b>Open</b>	Opens an existing file or creates a new file. When you select this item, a drop-down menu is displayed containing three types of files: sample information, analysis conditions, and report options.
<b>Save</b>	Saves the file in the active window..
<b>Save As</b>	Allows you to save the file that is in the active window under a different name. It also can be used to save a subset of a sample file as an independent parameter file.
<b>Save All</b>	Saves all open files.
<b>Close</b>	Closes the file in the active window.
<b>Close All</b>	Closes all open files.
<b>Print</b>	Prints details of the selected file(s).
<b>List</b>	Generates a list of certain information for the selected sample file(s) or parameter file(s).

---

<b>Import MS Signal</b>	Allows you to import data collected on a Pfeiffer Vacuum (formerly Balzers) or MKS Instruments mass spectrometers. Exported ASCII data from Quadstar, Quadera, and Process Eye software are supported.
<b>MS Signal Calibration</b>	Displays a series of dialogs used to create, edit, and manage calibration files created from imported mass spec signals. Calibration files enable the software to convert mass spec signals and peak area data to volume data.
<b>Export</b>	Exports a sample file in ASCII format or in a format compatible with the Grams/32 application (a peak editing program used previously with the AutoChem).
<b>Exit</b>	Exits the analysis program.



## Open a File

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### Sample Information

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Sample Information files contain information used to control the analysis, as well as collected data. A sample information file consists of four parts:

- Sample information
- Analysis conditions
- Report options
- Collected data (this part does not appear until analysis is complete)

Portions of the sample file can also exist as parameter files separate from the sample information file itself. These parameter files contain frequently used analysis conditions and report options that can be conveniently loaded into a new sample information file.

Sample information files are presented in three different formats, Basic, Advanced, and Restricted. You can select any one of these formats as your default. A convenient toggle button makes it easy to switch between Basic and Advanced formats, regardless of which is selected as your default.

- **Basic Format**

Presents the entire sample information file in a single dialog box. Basic format makes it easy to select previously created, standard parameter files from convenient drop-down lists.

- **Advanced Format**

Presents all four parts of the sample information file as a group of dialog boxes with index card-type tabs (although the Collected Data tab does not appear until after analysis). Clicking on each tab opens the associated dialog. Use this format to create customized sample information files.

- **Restricted Format**

Restricted format is identical to Basic format, except it prevents the operator from switching to the Advanced format. This format, which is password protected, limits an operator's ability to change analysis conditions and report options.

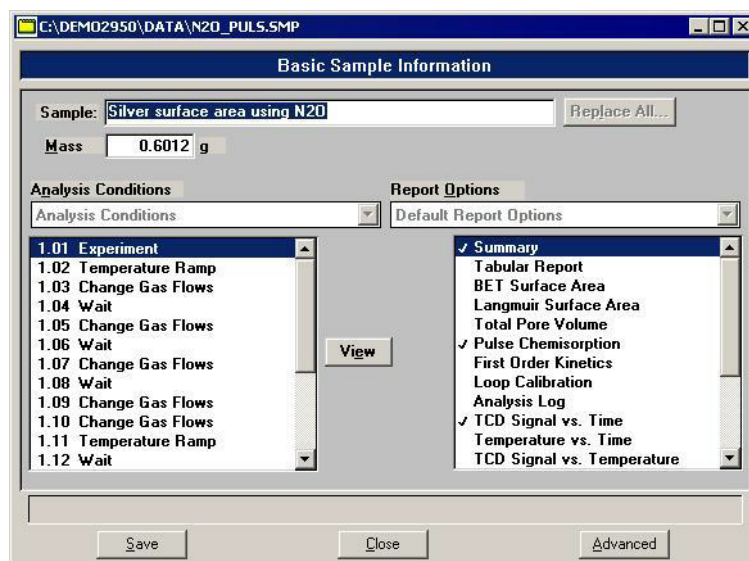
Select **Options > Option presentation** to specify your default format.



**If you are creating a new sample information file, the dialogs display the default values specified in the Sample Defaults. Refer to “[Sample Defaults](#)” on page 8-7.**

## Basic Format

The Basic format displays all parameters of the sample information file on a single dialog. With the Basic format, you can quickly create a sample information file using predefined parameter files. To view or edit specific parameters, you can switch easily to the Advanced format by clicking **Advanced**.



### Sample

Contains the description of the current sample file.

If this is a new file, this field contains the next sequenced description based on the format you specified in Sample defaults.

### Mass

Allows you to enter the mass of the sample.

### Analysis Conditions

Displays the name of the current or default Analysis Conditions file.

To choose a different file, click the down arrow to the right of the field, then click the file.

### View

Allows you to view details about the currently selected step in the analysis conditions set.

### Report Options

Displays the name of the current or default Report Options file.

To choose a different file, click the down arrow to the right of the field, then click the file.

**Reports List Box**

Displays a list of available reports. The checked reports are the ones requested for the current Report options file. If this is a new file, the checked reports are the ones you specified for the default(s). You can select (or deselect) reports by double-clicking on the report.

**Replace All**

Allows you to copy an existing sample file's values into the sample information file you currently have open. A file selection dialog is displayed. Choose the file containing the values to copy, then click **OK**; the values are copied into the new file automatically. You may then edit the values in the new sample file without altering the sample file from which they were copied.

**Save**

Saves the currently open file.

**Close**

Closes the currently open file.

**Advanced**

Switches the presentation format to the Advanced format.

## Advanced Format

The Advanced format displays the sample information file in a manner similar to that of an index card file. Each parameter has its own tab and is accessed by clicking the tab. Or, you can use **Prev** and **Next** to move through the dialogs. This format allows you to create customized sample information files.

The prompts for the **Sample**, **Operator** and **Submitter** fields may be customized by selecting **Sample defaults** from the Options menu. Refer to **“Advanced” on page 8-10** for instructions on customizing these prompts.

### Sample

Contains the description of the current sample file.

If this is a new file, this field contains the next sequenced description based on the format you specified in Sample Defaults.

### Operator Submitter

Displays the Operator and Submitter names (if specified) of the current sample file.

If this is a new file, these fields contain the names specified in Sample defaults.

**Bar Code**

This field enables you to enter bar code information. If bar code information is not used, you can use this field to enter additional information about the sample; for example, to enter the lot number of your sample.

This field will also accept data from a bar code reader.

You can use up to 40 alphanumeric characters.

**Mass**

Allows you to enter the mass of the sample.

**Add Log Entry**

Displays a dialog box allowing you to enter information relating to the sample.



Any information you enter here is printed as part of the Analysis Log report (select in Report Options).

**Comments**

Use this field to enter any comments about the sample or the planned analysis conditions. Comments entered here are printed in the header of some reports.

**Replace All**

Allows you to copy the values for all parameters of an existing sample file into the sample information file you are creating. A file selection dialog is displayed. Choose the file containing the values to copy, then click **OK**; the values are copied into all parameters of the new file automatically. You can then edit the values in the new sample file without altering the sample file from which they were copied.



**Each of the individual parameter files that make up the sample information file can also be replaced separately using the Replace button that appears on the associated dialog.**

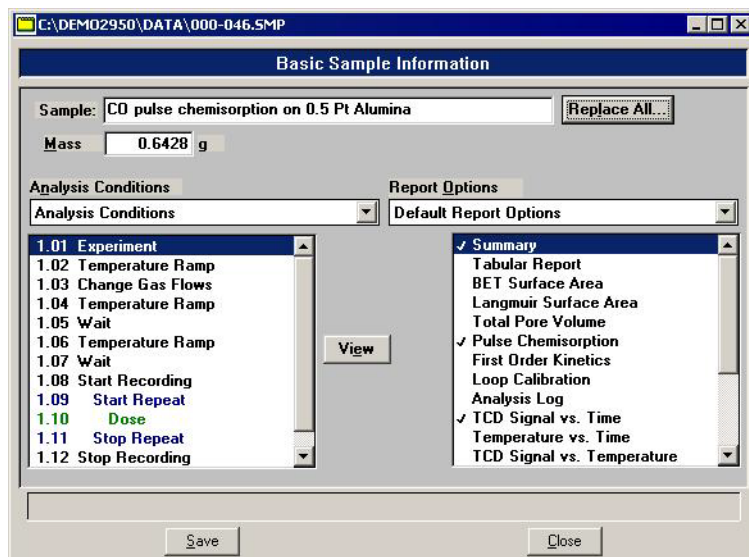
**Basic**

Switches the presentation format to the Basic format.

The Analysis Conditions and Report Options tabs of the Advanced format sample information file are described in subsequent sections.

## Restricted Format

A third format is provided to control access to some portions of the sample information file. Restricted format displays in the same manner as the Basic format.



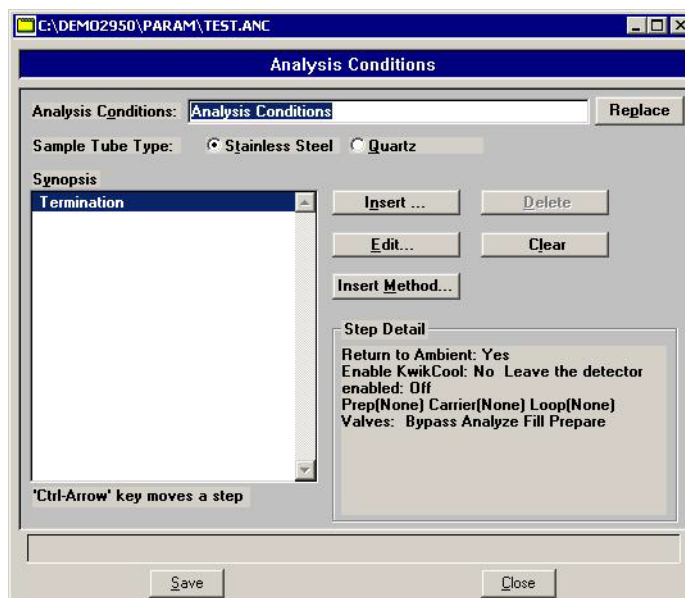
The Restricted format does not allow switching to the Advanced format for editing.

In Restricted format, the **Advanced** button does not appear on the screen, so the operator cannot switch to the Advanced format. This format is also password-protected, preventing the operator from making changes to analysis conditions and report options sets (refer to [“Restricted Format” on page 8-4](#)).

Restricted format is ideal for laboratories in which standard analysis procedures are established by a lab manager, while one or more lab technicians actually perform the analyses. The lab manager can create independent analysis conditions and report options files containing standard sets of operating conditions, then the operators can use Restricted format for daily operations, selecting the standard parameter files from the drop-down lists.

## Analysis Conditions

Analysis conditions files specify each step in the analysis, including temperatures, pressures, and gases. You can create an Analysis Conditions file as an independent parameter file or as part of the sample information file. You can also save the analysis conditions portion of the sample information file as a parameter file.



When creating parameter files to display in the drop-down list on the Basic sample information dialog, be sure to save them to the directory specified as the Parameter File directory (see page 8-17).

### Analysis Conditions

If you are opening an existing analysis conditions file, this field contains the description.

If you are creating a new analysis conditions file, this field contains the description specified in the Sample defaults. Use a description that will help you identify this set of analysis conditions in the future.

### Replace

Allows you to copy values from an existing file to the one you are creating. A dialog box is displayed allowing you to choose the file.

**Sample Tube Type**

Enables you to choose the type of sample tube that will be used for the analysis.

Choose **Stainless Steel** for a high pressure analysis. Stainless steel tubes can be used for analyses performed with pressure settings of up to 1000 psia and temperature settings up to 850 °C.

Choose **Quartz** for an analysis performed with pressure settings at ambient and temperature settings up to 1100 °C.

**Do not use stainless steel sample tubes with temperatures above 850 °C. Doing so could weaken, corrode or break the sample tube.**

**Do not use quartz sample tubes with pressures above ambient. Doing so could loosen the O-ring seal and allow gas leakage.**

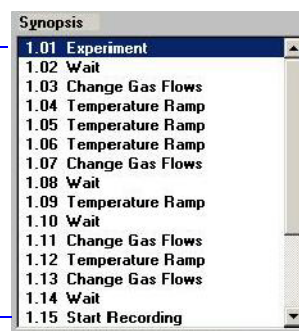
**Synopsis**

Displays a list of the steps the analyzer will follow during the analysis.



**A set of analysis conditions may consist of up to 99 separate *experiments*, such as TPR or TPO and each experiment may consist of up to 99 *steps*.**

Steps within  
Experiment 1



The Synopsis is the key to the AutoChem 2950 HP's high level of automation. This list enables the software to control and change the gas flow, temperature, and data recording



## Synopsis (continued)

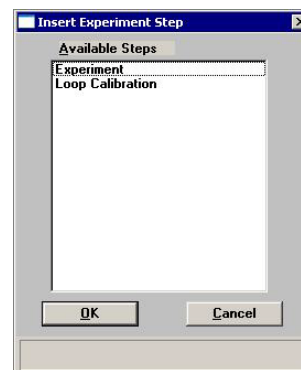
process. It causes the analyzer to progress to the next step, or to wait until specific criteria are met before proceeding. In all, this list provides a step-by-step guide for the analysis.

## Insert

Allows you to insert a new experiment or a step within an experiment.



This dialog displays if you are inserting a new experiment.



This dialog displays if you are inserting a step within an experiment.



At some points in the sequence of steps, all steps are available. At other points, certain steps cannot be performed. For example, you cannot start a new experiment inside a **Repeat** loop.

The steps that appear in the list box are those which are currently available. To select an experiment or step to insert: click on it, then click **OK**. A dialog enabling you to specify instructions for the step or experiment is displayed. Each of the specific step and experiment dialogs are discussed in subsequent sections.

The new step is inserted directly before the step that was highlighted when **Insert** was selected. After you insert a **Repeat** step or **Record** step, the automatically entered **Stop Repeat** or **Stop Record** step is highlighted. Any steps inserted at that point will occur within the Repeat or Record loop.



**You can also insert steps after the analysis has started by suspending the analysis.**

**Insert**

*(continued)*

You can easily move a step from one position in the list to another. Click on it, then use **Ctrl** + ↑ or **Ctrl** + ↓ to move the highlighted step.

**Delete**

Deletes the selected item from the synopsis list. If you click this button when an experiment is selected, the entire experiment (including all its steps) is deleted after a confirmation.

**Edit**

Enables you to edit the selected experiment or step; an associated dialog is displayed.

You can also edit a step or experiment during analysis. However, you must suspend the analysis before any editing can be accomplished. You can edit only the steps or experiments following the current one; all completed steps will be disabled.

After an analysis has been completed, **Edit** displays as **View**. Steps may be viewed but not edited.

**Clear**

Clears the list of experiments and steps for this Analysis Conditions dialog. A confirmation message is displayed.

**Insert Method**

Allows you to insert the entire list of steps from another analysis conditions file. After you click this button, select an analysis conditions file from the list. All the steps in that file are inserted directly above the highlighted step. Inserted methods can be edited without altering the original analysis conditions file(s) from which they were copied.

For example, if you have three different standard sample preparation methods that you use under varying circumstances, create an analysis conditions file for each preparation method. Then use **Insert Method** to insert the appropriate preparation method in the analysis conditions file you create when you are ready to analyze a sample.

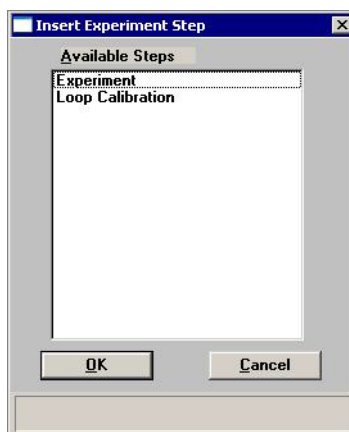
You can also create several analysis conditions files containing individual experiments, then use **Insert Method** to combine them into a comprehensive analysis comprised of these individual analyses.

**Step Detail**

Displays information about the currently highlighted step in the Synopsis. The information displayed for each step is a summary of the information contained on that step's dialog.

**Insert Experiments**

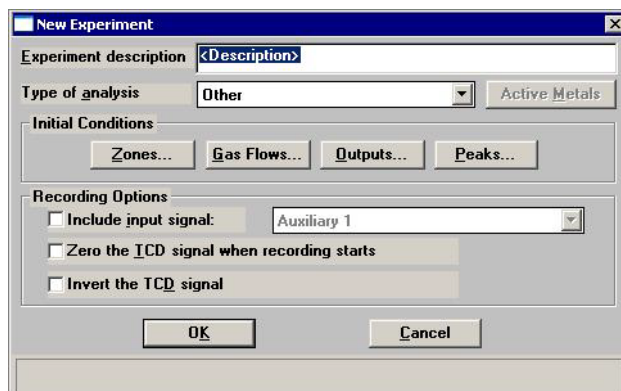
An analysis set is created by inserting a series of experiments in the sample file Synopsis. You can insert up to 99 experiments. There are two types of experiments that you can insert in an analysis conditions set. The following dialog is displayed when you choose to insert an experiment:



The first type is the customizable Experiment. You will insert into this Experiment the individual steps which control the reaction(s) in the analysis. When you insert an experiment, you specify the Initial Conditions, then insert individual steps. Each step can be customized in detail. The second type is Loop Calibration. The sequence of steps in Loop Calibration cannot be altered.

## Experiment

Inserts a basic experiment into the list of steps.



The 'New Experiment' dialog box contains the following fields and buttons:

- Experiment description:** A text field with the placeholder '<Description>'.
- Type of analysis:** A drop-down menu currently set to 'Other', with an 'Active Metals' button to its right.
- Initial Conditions:** A section containing four buttons: 'Zones...', 'Gas Flows...', 'Outputs...', and 'Peaks...'.
- Recording Options:** A section with three checkboxes:
  - ☐ Include input signal: Auxiliary 1 (with a drop-down arrow)
  - ☐ Zero the TCD signal when recording starts
  - ☐ Invert the TCD signal
- Buttons:** 'OK' and 'Cancel' at the bottom.

### Experiment description

Allows you to enter a description or identifier for this experiment. The description you enter in this field appears in the legend of the report.

### Type of analysis

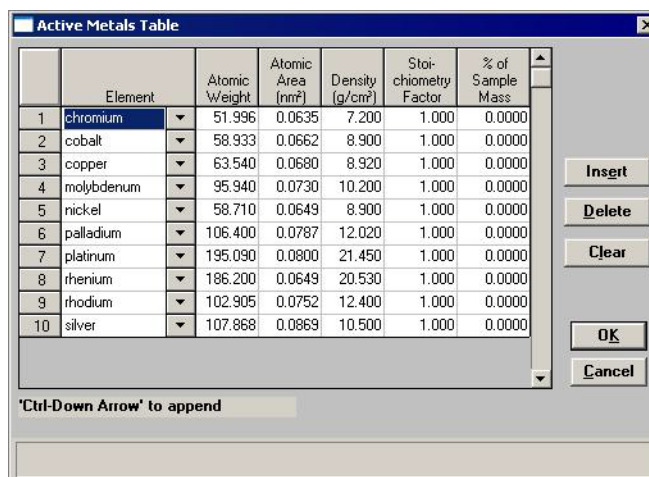
Select the type of analysis from the drop-down list.



**It is important to select the correct experiment type because the type is referenced during some other operations. For example, a Pulse Chemisorption report can only be generated for those experiments specifying *Pulse Chemisorption* as the type.**

### Active Metals

Opens the Active Metals Table dialog, which contains the metals entered in the default Active Metals Table. You can edit the values for metal characteristics shown in the table (the default table will not be affected).



The 'Active Metals Table' dialog box displays a table of metal characteristics. The table has 10 rows and 7 columns. To the right of the table are buttons for 'Insert', 'Delete', 'Clear', 'OK', and 'Cancel'. At the bottom, there is a text field with the instruction '\*Ctrl-Down Arrow' to append'.

	Element	Atomic Weight	Atomic Area (nm <sup>2</sup> )	Density (g/cm <sup>3</sup> )	Stoichiometry Factor	% of Sample Mass
1	chromium	51.996	0.0635	7.200	1.000	0.0000
2	cobalt	58.933	0.0662	8.900	1.000	0.0000
3	copper	63.540	0.0680	8.920	1.000	0.0000
4	molybdenum	95.940	0.0730	10.200	1.000	0.0000
5	nickel	58.710	0.0649	8.900	1.000	0.0000
6	palladium	106.400	0.0787	12.020	1.000	0.0000
7	platinum	195.090	0.0800	21.450	1.000	0.0000
8	rhenium	186.200	0.0649	20.530	1.000	0.0000
9	rhodium	102.905	0.0752	12.400	1.000	0.0000
10	silver	107.868	0.0869	10.500	1.000	0.0000

\*Ctrl-Down Arrow' to append



The contents of this table are copied from the default table on the Options menu. After this sample file is created, however, the contents of this table become specific to the file. Changes made in this table do not affect the default table and changes made to the default table do not affect this table.

**Insert**

Inserts a new row into the table; the row is inserted above the selected one. The row consists of a drop-down list containing the active metal defaults. Select a metal from the list.

Note that you cannot add a metal at this point. To add a metal select **Options > Active Metal defaults**.

**Delete**

Deletes the selected row.

**Clear**

Clears the table of all entries except one; one is required.

**Initial Conditions**

The buttons in this group box allow you to specify initial conditions for the analysis.

**Zones**

Displays the Set Temperature Zones dialog allowing you to set the heat zone temperatures.

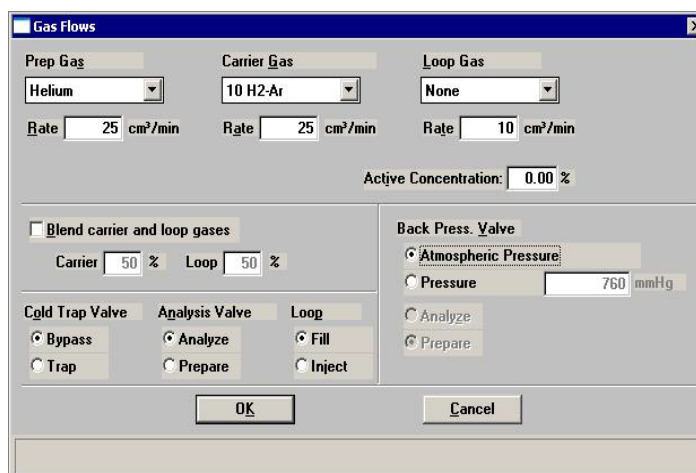
The dialog box titled "Set Temperature Zones" contains two sections. The "TCD Detector" section has two rows: "Block zone" with a value of 100 °C and "Filament" with a value of 175 °C. The "Valve Zones" section has four rows: "Cold trap" with a value of 110 °C, "Analysis" with a value of 110 °C, "Loop" with a value of 110 °C, and "Back Pressure" with a value of 110 °C. At the bottom are "OK" and "Cancel" buttons. A status bar at the very bottom reads "Enter a value between 20 and 150."

For the TCD block and filament zones, higher temperatures may improve signal to noise ratios but can also reduce filament life.

The **Filament** temperature must be set at least 20 °C higher than the **Block zone** temperature.

## Gas Flows

Displays the Gas Flows dialog.



This dialog allows you to choose the gases for this step, specify the flow rates, specify the percent of the carrier gas mixture which is composed of the reactive, and set the state of the rotary valves.

### Prep Gas Carrier Gas Loop Gas

For each set of inlet ports, you may select a gas from the drop-down list.

*Rate: 5 to 175 cm<sup>3</sup>/min.*

The gases that appear in each drop-down list are those specified in **Gas Defaults** (accessed from the Options menu).

Select **None** when no gas is to be flowing (the flow rate is ignored).

When you start an analysis, the system checks to ensure that the selected gases are connected to the appropriate ports. If there is a discrepancy between a gas selected for the current sample file and the gas indicated in the Gas Selections dialog, an error message is displayed when you attempt to begin the analysis.



**Make sure the appropriate gases are attached to the appropriate ports, and that the Gas Selections dialog accurately shows which gas is connected to each port.**

<b>Active Concentration</b>	Specify the percent of the gas mixture which is composed of the reactive gas versus an inert filler. This value is used for pulse chemisorption data reduction.
<b>Blend carrier and loop gases</b>	Select this option to have the carrier and loop gases blended. Then enter the percentage for the gases. Whichever field you make an entry in, the other field automatically defaults to a percentage totaling 100.
<b>Cold Trap Valve</b> <b>Analysis Valve</b> <b>Loop Valve</b> <b>Back Press. Valve</b>	Choose a status for each valve.



The following two concerns do not arise when you insert the first Experiment in the Synopsis, but they must be considered when selecting gases for subsequent experiments and for subsequent Change Gas Flows steps.

- Avoid placing incompatible gases in the analyzer in immediate sequence.

When you change the gas flowing into the analyzer, the new gas enters the analyzer while the previous gas is still present.

You can “purge” the previous gas before starting to flow the new gas by selecting an inert gas for the initial conditions gas in an experiment step, or by inserting an extra **Change gas flows** step in which you flow an inert gas between the flows of incompatible gases. If you do so, you must also insert a **Wait** step specifying how long to flow the inert gas. You can specify a specific time period or select one of the other **Wait** step criteria. (Each of these steps is described later in this chapter.)

- Changes in the gas cause a brief adjustment of the Mass Flow Controller(s) involved in the change.

This adjustment may be detected by the TCD, and may be visible in the data (if data are being recorded when the gas flow is changed).

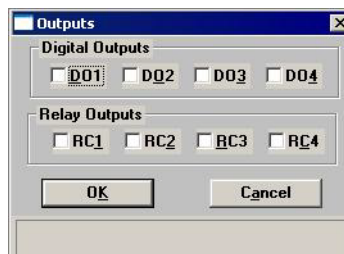
Either disregard such disturbances in the data or stop recording before you change the gas flows, then insert a **Wait for stable baseline** step after the **Change gas flows** step. Resume recording after a stable baseline is detected. A suggested sequence of steps then might be as follows:

- Start recording
- Experiment steps as needed
- Stop recording
- Change gas flows

- Wait for stable baseline
- Start recording
- Experiment steps as needed

## Outputs

Displays the Outputs dialog allowing you to specify the state of the digital and relay outputs.

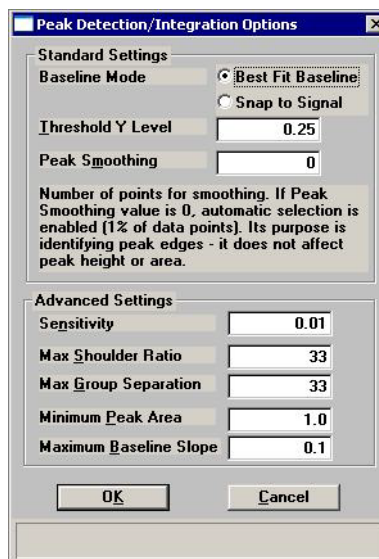


Ports are provided for connecting auxiliary inputs and outputs. For example, the digital outputs can be used to control a mass spectrometer.

Refer to **“AUXILLIARY INPUTS AND OUTPUTS” on page E-1** for a complete description of the auxiliary input/output function.

## Peaks

Allows you to control peak detection during analysis. After analysis, you can further control peak detection using the Peak Editor (refer to **“Peak Editor” on page 7-16**).



The TCD detects and records all deviations from baseline, but only those which satisfy the criteria established in this dialog are reported as peaks. For more information about this dialog, refer to Appendix H, **PEAK DETECTION / INTEGRATION OPTIONS**.



**Recording Options****Include input signal**

These options allow you to use external electrical input from an auxiliary port, or you can choose to report an instrument-specific parameter from the drop-down list.

Refer to **[“AUXILLIARY INPUTS AND OUTPUTS” on page E-1](#)** for additional information on auxiliary input/outputs.

**Zero the TCD signal when recording starts**

Allows you to zero the TCD signal automatically the first time recording starts in an experiment.

**Invert the TCD signal**

Inverts the TCD signal. For example, if you expect to obtain only negative peaks, this option can be used to record the peaks in the positive direction.

## Loop Calibration

**Loop Calibration** enables you to calibrate the gas injection loop. This experiment verifies the exact volume of the loop for use in calculations on analyses in which the loop is used. Sample analysis data yield signal vs. temperature data and peak areas. Associating the sample information file with a loop calibration file makes it possible for the software to convert peak area to peak volume.



Appendix J provides a tutorial for performing a loop calibration (page J-38.)

The screenshot shows the 'Loop Volume Calibration Experiment' dialog box. It has a title bar with a close button. The 'Description' field contains '<Description>'. Under 'Calibration Gases', 'Gas 1' is set to 'Choose a Gas' (Attached as a carrier gas) with a flow rate of 10 cm<sup>3</sup>/min. 'Gas 2' is also set to 'Choose a Gas' (Attached as a loop gas) with a flow rate of 10 cm<sup>3</sup>/min. 'Loop Thermal Equilibration' is set to 2.00 min. 'Physical Syringe Volume' is set to 1.00000 cm<sup>3</sup>. Under 'Recording Options', there are two unchecked checkboxes: 'Zero the ICD signal when recording starts' and 'Invert the TCD signal'. At the bottom are buttons for 'Zones...', 'Gas Flows...', 'Outputs...', 'OK', and 'Cancel'.

### Description

Enter a description or identifier for this calibration. It is important to use a description that will help you identify this data when displayed in sample reports.

### Calibration Gases

Allows you to select the carrier and loop gases to be used in this calibration. The gases available are those specified in the **Gas defaults** accessed from the **Options** menu.

### Flow Rate

Specify the flow rate for the loop and carrier gases.

Typically, the default of 10 cm<sup>3</sup>/min is appropriate

### Loop thermal equilibration delay

Allows you to specify the duration of the equilibration delay. This delay allows extra time for the instrument to stabilize thermally after the baseline has fully stabilized.

**Physical Syringe Volume**

Enter the volume of gas to be injected from the syringe you are using. The exact quantity should be calculated.

Typical values are: 0.45 cm<sup>3</sup> for a 0.5 cm<sup>3</sup> loop, 0.9 cm<sup>3</sup> for a 1.0 cm<sup>3</sup> loop, and 4.5 cm<sup>3</sup> for a 5.0 cm<sup>3</sup> loop.

**Zero the TCD signal when recording starts**

Select this option to zero the TCD signal automatically the first time recording starts in an experiment.

**Invert the TCD signal**

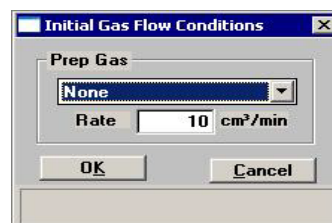
Inverts the TCD signal. For example, if you expect to obtain only negative peaks, this option can be used to record the peaks in the positive direction.

**Zones**

This button functions in the same manner as the Zones button described earlier in **Experiment**. Refer to [“Zones” on page 5-15](#).

**Gas Flows**

Enables you to specify the Preparation gas and its flow rate. This feature is useful if you will have a sample loaded while calibration is occurring.



Select a gas from the drop-down list. The gases that appear in the drop-down list are those specified in the **Gas defaults** accessed from the **Options** menu

When you start an analysis, the software checks to be sure that the gases you select are connected to the appropriate ports by comparing them to the Gas Selections dialog of the Unit menu. If there is a discrepancy between a selected gas and the gas indicated in the Gas Selections dialog, an error message is displayed when you attempt to begin the analysis.

Enter a flow rate in the **Rate** field.

## Outputs

This button functions in the same manner as the Outputs button described earlier in **Experiment**. Refer to **“Outputs” on page 5-18**.

## Insert Experiment Step

After you have set up the initial conditions for your basic Experiment, the Analysis Conditions dialog is again displayed showing the Experiment inserted. Clicking **Insert** now allows you to insert a step in the Experiment you just added to the Synopsis. You can insert steps in a wide variety of sequences. You can also use steps repeatedly within the Experiment.



Unlike the basic Experiment, Loop Calibration is a specialized experiment, which is pre-programmed so that you can insert it in your analysis as a single step.



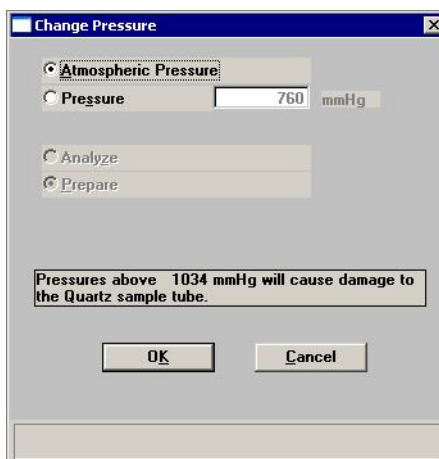
**You cannot change the steps included in a Loop Calibration, but you can include it in analyses that contain other experiments.**

## Change Gas Flows

Displays the Gas Flows dialog. This dialog is identical to the one that is opened when **Gas Flows** is selected from the Insert Experiment Step dialog. A complete description of this dialog is found on page **5-16**.

## Change Pressure

Displays the Change Pressure dialog.



These options allow you to set target pressures for analysis or preparation, or to return to atmospheric pressure.



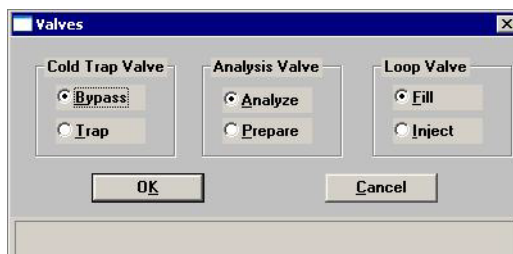
**Do not use quartz sample tubes with pressures above 1034 mmHg. If you do so, the O-rings will not seal and the sample tube can be released from its fitting.**

## Change Temperature Zones

Displays the Set Temperature Zones dialog. This dialog is identical to the one that is opened when **Zones** is selected from the Insert Experiment Step dialog. A complete description of this dialog is found on page [5-15](#).

## Change Valves

Displays the Valves dialog, allowing you to choose the state in which each of the rotary valves is to be placed.



## DDE Command

The DDE (Dynamic Data Exchange) Command experiment step enables the 2950 application to communicate with another DDE-aware application, such as a mass spectrometer or blender program, running on the computer.



<b>Service</b>	Enter the name of the service provided by the DDE-aware application (refer to the DDE server application's user manual).
<b>Topic</b>	A service will contain one or more topics as described in the application's user manual. Enter the topic.
<b>Transaction Type</b>	Enables you to choose a type of transaction.
<b>Execute a remote command</b>	Select this option to execute the command you enter in the Data field.
<b>Poke data into another application</b>	Select this option to send the text string you enter in the Data field to the specified application.
<b>Retrieve and log a data item</b>	Select this option to add the response to the transaction you specify in the Item field to the analysis log.
<b>Item</b>	If you selected <b>Retrieve and log a data item</b> or <b>Poke data into another application</b> , enter the name of the transaction (refer to the DDE server application's user manual).
<b>Data</b>	If you selected <b>Execute a remote command</b> or <b>Poke data into another application</b> , enter the command or data as a text string.

## **Dose**

**Dose**

☒ **Inject Loop Gas**

☐ **Manual Injection**

Physical Injection Volume:  mL

☐ **Get Physisorption Point** Baseline...

**After Injection**

☒ **Wait for change from Baseline or 2.00 minutes, then wait for a return to Baseline**

☐ **Wait for 2.00 minutes, then wait for a return to Baseline**

☐ **Wait for 2.00 minutes**

OK Cancel

### **Dosing Choices**

Click a radio button to select the appropriate dosing method for this experiment step.

#### **Inject Loop Gas**

Automatically injects the contents of the loop into the path that leads to the sample. The contents of the loop are pushed out of the loop by the Carrier Gas.

#### **Manual Injection**

Prompts you to inject a dose of gas into the septum using a syringe.

#### **Get Physisorption Point**

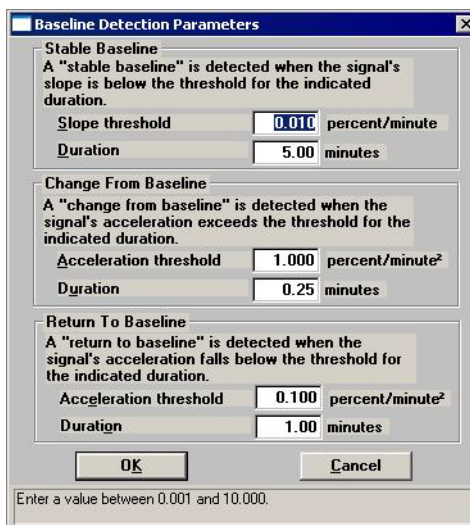
Prompts you through the steps necessary to collect data for a physisorption point. These steps include placing a Dewar of liquid nitrogen around the sample tube, waiting for a return to baseline, then replacing the Dewar with water at room temperature. You can take repeated points by placing the Dose step within a Repeat Loop (discussed later in this chapter).

### **After Injection**

Allows you to specify the conditions for completion of this step.

## Baseline

Click to specify Baseline settings if you have selected a **Wait** step that depends upon the Baseline.



The dialog box is titled "Baseline Detection Parameters" and contains three sections for setting thresholds and durations. The "Stable Baseline" section has a slope threshold of 0.010 percent/minute and a duration of 5.00 minutes. The "Change From Baseline" section has an acceleration threshold of 1.000 percent/minute<sup>2</sup> and a duration of 0.25 minutes. The "Return To Baseline" section has an acceleration threshold of 0.100 percent/minute<sup>2</sup> and a duration of 1.00 minutes. At the bottom, there are "OK" and "Cancel" buttons, and a note: "Enter a value between 0.001 and 10.000."

Section	Parameter	Value	Unit
Stable Baseline	Slope threshold	0.010	percent/minute
	Duration	5.00	minutes
Change From Baseline	Acceleration threshold	1.000	percent/minute <sup>2</sup>
	Duration	0.25	minutes
Return To Baseline	Acceleration threshold	0.100	percent/minute <sup>2</sup>
	Duration	1.00	minutes

Establish the **Slope** or **Acceleration threshold** and **Duration** for determining what constitutes a **Stable Baseline**, a **Change From Baseline**, and a **Return To Baseline**. These values control whether a particular change in the signal is significant to the current experiment; in other words, they define a stable baseline.

Some **Wait** steps are contingent upon the values you select here. For example, if your experiment contains a **Wait until Baseline is stable** step, the signal is compared to these values to determine if a stable baseline has been established. Lower slope/acceleration values and longer durations create a more rigorous definition of these factors than higher values and shorter durations. Normally the default values are satisfactory.

The range for each field on this dialog is displayed in the information bar at the bottom of the dialog when the field is selected.



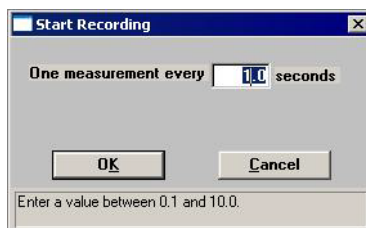
### **Experiment**

Enables you to enter a new experiment within the current experiment. Refer to **“Experiment” on page 5-14**.

### **Loop Calibration**

Enables you to enter a loop calibration within the current experiment. Refer to **“Loop Calibration” on page 5-20**.

### **Recording**



Allows you to specify how frequently the signal reading is recorded.

Recommended values are 1.0 for TPR, TPD, and TPO experiments, and 0.2 for pulse chemisorption.

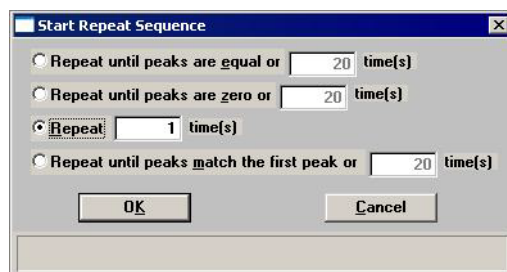
The **Start Recording** dialog starts the recording of the signal for this analysis. A **Stop Recording** step is inserted in the list of steps automatically on the Analysis Conditions dialog when you insert a **Start Recording** step.



**If a Start Recording step is immediately followed by a step that prompts an immediate peak, peak data are recorded before any baseline readings can be collected. To collect some baseline data before the first peak in such an experiment, insert a Wait for X minutes step after the Start Recording step, but before the step which causes the peak.**

## Repeat

When you insert a Repeat step in your analysis conditions, the Start Repeat Sequence dialog opens allowing you to specify the duration of the repeat sequence. After you complete the Start Repeat Sequence dialog and click **OK**, a Start Repeat step and a Stop Repeat step are automatically inserted in the list of steps for this analysis. You may insert multiple experiment steps within the Repeat loop.



**Repeat until peaks are equal or \_\_\_\_ times.**

Stops repeating the steps within the loop when the last four peaks of this experiment have equal volumes or when the maximum number of repeats is reached.

(Equal means the last four peaks are within 15% of their average, and the average of the last four peaks is larger than the first peak or the average of the last four peaks is larger than

$$\frac{N+5}{N} \times \text{Size of first peak}$$

where N is the number of peaks.)

This option is useful when performing H<sub>2</sub> or CO pulse chemisorption on supported metal catalysts.

**Repeat until peaks are zero or \_\_\_\_ times.**

Stops repeating the steps within the loop when the last four peaks of this experiment have zero volume or when the maximum number of repeats is reached. (Zero volume is defined in the software as four peaks with volumes within 10% of each other and all volumes less than 50% of the first peak.)

This option is useful when performing an N<sub>2</sub>O decomposition for characterizing copper catalysts.

**Repeat \_\_\_\_ times.**

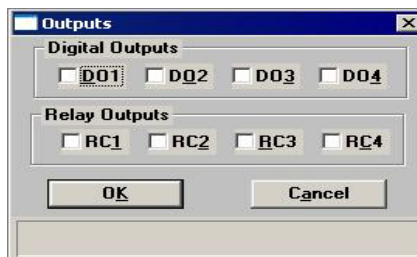
Stops repeating the steps within the loop when the specified number of times is reached.

**Repeat until peaks match the first peak or \_\_\_\_ times.**

Stops repeating the steps within the loop when the peaks match the first peak or when the maximum number of repeats is reached.

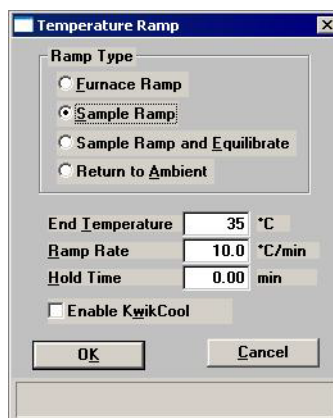
### **Set Outputs**

When this step is selected, the Outputs dialog opens. Select each signal to enable. A complete discussion of the auxiliary input/output options is contained in Appendix E, page [E-1](#).



### **Temperature Ramp**

Insert this step to change the sample temperature.



#### **Furnace Ramp**

Ramps the furnace temperature directly to the End temperature, ignoring the sample temperature.

#### **Sample Ramp**

Ramps the sample temperature to the End temperature. The actual furnace temperature is adjusted to meet this target.

#### **Sample Ramp with Equilibration**

Ramps the sample temperature to the End temperature and waits for it to equilibrate before proceeding.

#### **Return to Ambient**

Allows the furnace temperature only (not the sample temperature) to return rapidly to a temperature between 14 and 50 °C.

**End Temperature**

Enter the ending temperature for this ramping procedure.

An ending temperature below 20 °C enables the CryoCooler (if installed) automatically.

This field is disabled when **Return to Ambient** is selected.

**Ramp Rate**

Enter the ramp rate for this ramping procedure.

This field is disabled when **Return to Ambient** is selected.

**Hold Time**

Enter the amount of time to hold the temperature at the conclusion of this step. If **Equilibrate** is selected, the hold time is not started until the temperature is equilibrated.

This field is disabled when **Return to Ambient** is selected.



**If you are ramping from subambient temperatures and the CryoCooler is installed, the target temperature should be held for several minutes. This ensures that the entire furnace has reached thermal equilibrium before the ramp begins. Otherwise it may take several minutes for the temperature ramp rate to stabilize.**

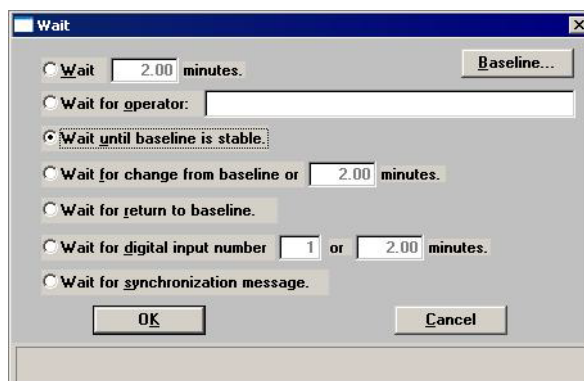
**Enable KwikCool**

Select this to use the KwikCool to reduce the furnace temperature more rapidly during cool-down. If this option is checked and the KwikCool is not attached to the analyzer when the analysis is started, an error message is displayed.

If the CryoCooler (rather than the KwikCool) is attached, the cool-down operation is performed by the CryoCooler

## Wait

Allows you to specify a waiting routine.



**Wait \_\_\_\_ minutes.**

Specify an exact number of minutes to wait.

**Wait for operator**

Enter a description of the operator's task, such as "Fill the Dewar", then click **OK**.

During an analysis, the message you enter is displayed on the monitor at the appropriate time and the attention LED on the upper front panel will flash or blink. The analysis continues after the operator clicks **OK**. Be sure to specify whether the operator should first perform the task or first click **OK**.

**Wait until baseline is stable.**

Waits until the baseline becomes stable. If you choose this option, click **Baseline** to specify the settings.

**Wait for change from baseline or \_\_\_\_ minutes.**

Enter the number of minutes to wait, then click **Baseline** to specify the settings.

**Wait for return to baseline.**

Waits for a return to Baseline. If you choose this option, click **Baseline** to specify the settings.

**Baseline**

Click to specify Baseline settings if you have selected a Wait step that depends upon the Baseline.

This dialog is described on page [5-26](#).

**Wait for digital input  
number \_\_\_\_\_ or  
\_\_\_\_\_ minutes.**

Enter the number of the digital input source being awaited, then specify the maximum number of minutes to wait for input. If the digital input is not received before the time elapses, the analysis will continue, and a warning message is displayed. Digital inputs are electrical connections to the right side of the instrument. See Appendix E, page [E-1](#) for additional information.

**Wait for synchronization  
message.**

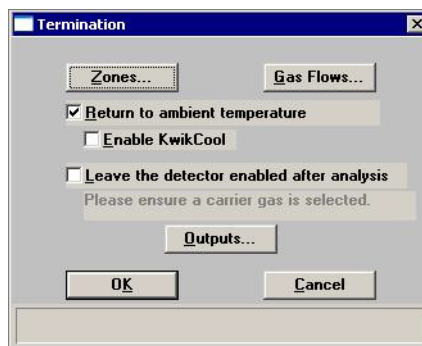
Select this option to direct the 2950 software to act as a DDE server. When the 2950 program receives a message from another DDE application (the client), this Wait will be completed and logged into the sample log.

The DDE client must use the following parameters in an Execute a Remote Command in order to provide the synchronization message.

*Service*    *WIN2950*  
*Topic*     *Command*  
*Item*      *Sync*

## Termination

This step is automatically inserted in every experiment. The contents of this step are the post-analysis conditions specified using Sample defaults on the Options menu. You can view and/or edit the details of this dialog by clicking **Edit** when **Termination** is selected (highlighted); the Termination dialog is displayed.



### Zones

Displays the Set Temperature Zones dialog allowing you to view or edit current temperature zones. Refer to page [5-15](#) for information on this dialog.

### Gas Flows

Displays the Gas Flows dialog allowing you to view or edit current gasses and their flows. Refer to page [5-16](#) for information on this dialog.

For a high pressure analysis, select the same valve setting as selected at the start of the analysis. If the valve settings are not the same, you will receive an error message.

### Return to ambient temperature

Select this option to have the sample cooled to ambient temperature.

### Enable KwikCool

Select this option to enable the KwikCool during the cool-down operation. If the CryoCooler rather than the KwikCool is attached, the cool-down operation is performed by the CryoCooler.

**Leave the detector enabled after analysis**

Several hours may be required for the instrument to reach thermal stability after the detector is enabled. This option keeps the detector enabled after analysis, allowing the instrument to remain stable.

Gas flow through the detector is required while the detector is enabled but is not necessary if the detector is disabled. However, it is good practice to flow an inert gas to continuously purge the system.

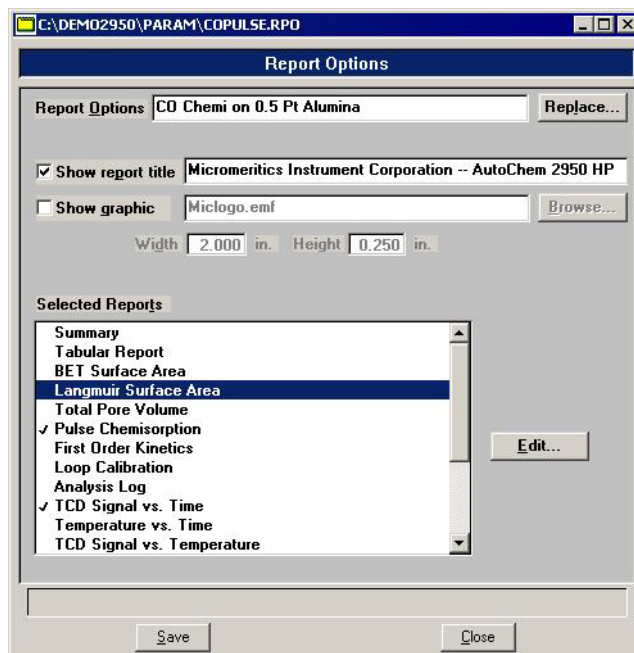
Should the analysis be cancelled due to an instrument error, the detector will be left enabled according to this option. Should the analysis be cancelled by the operator, this option is ignored, and the detector is left enabled.

**Outputs**

Displays the Outputs dialog allowing you to view or edit the digital and relay outputs. Refer to page 5-18 for additional information on this dialog.

**Report Options**

This dialog is used to edit existing report options, or to create new report options to be used in sample information files.



**When creating parameter files to display in the drop-down list on the Basic sample information dialog, be sure to save them to the directory specified as the Parameter File directory (see Chapter 8).**



**Report options**

If you are editing an existing file, this field displays the description or identification of the report options file.

If this is a new file, this field contains the description specified in Sample defaults. Use a description that will help you identify this set of report options in the future.

**Replace**

Allows you to copy values from an existing file to the one you are creating. A dialog is displayed so that you may choose the file containing the values to copy. Since the values are copied into the new file, you can edit them without changing values in the original file.

**Show report title**

This option allows you to have a title appear on your report and to enter text for it.

If this is a new file, the title you specified as the default is displayed. Accept the default title or enter a different one.

**Show graphic**

Select this option to have a graphic (bitmap or enhanced metafile) display above the report title. For example, you to display your company logo. Click **Browse** to select the graphic file, then enter the Height and Width in the fields provided. This image can also be edited from the report window.

**Selected Reports**

Contains a list of available reports. Included in the list are 10 user-definable graphs. These reports are described on page [5-43](#).

A report is selected when it is preceded with a check mark. Select reports by double-clicking the report(s). Alternatively, you can highlight the report and press **Edit**. Reports are deselected in the same manner. Certain reports can be edited by highlighting the report and clicking **Edit**.

**Edit**

Allows you to edit the highlighted report. Clicking **Edit** opens the appropriate dialog box. The dialog(s) associated with each type of report are described in the following sections.

The Summary Report and the Analysis Log cannot be edited.

## Summary Report

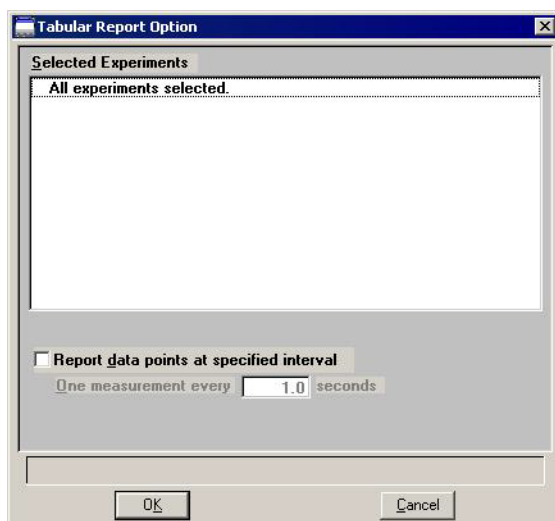
---

The Summary report provides a condensed listing of analysis statistics and data results. This report cannot be edited.

## Tabular Report Option

---

Clicking **Edit** when Tabular Report is selected opens the following dialog:



### Selected Experiments

All experiments are selected by default. When the report options file is used in a sample information file, each experiment in the sample file is listed.

### Report data points at specified interval

Select this option to report data points at a specified interval, then specify the frequency of reported data (the number of seconds between reported data readings).

This feature is useful if you anticipate a large amount of data to be collected during the analysis (such as in a lengthy analysis with readings taken every second). If you do not specify an interval for data reporting, all points are reported, regardless of the length of the report.

## BET Surface Area Report Option

Clicking **Edit** when BET Surface Area is selected opens the following dialog:



### Selected Experiments

All applicable experiments are selected by default. When the report options file is used in a sample information file, each physisorption experiment with active concentration < 40% is listed.

### Report Type

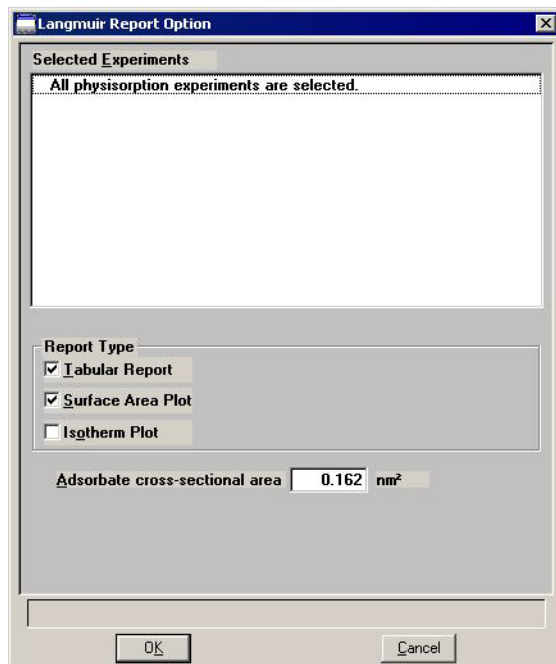
From this group box, choose the type of report(s). If you choose Multipoint or Multipoint plus one single point, the choices on the right side of the group box are enabled.

### Adsorbate cross-sectional area

Enter the appropriate value. The adsorbate cross-section is the area occupied by a single adsorbed molecule on the adsorbent surface.

## Langmuir Surface Area Report Option

Clicking **Edit** when Langmuir Surface Area is selected opens the following dialog:

**Selected Experiments**

All applicable experiments are selected by default. When the report options file is used in a sample information file, each physisorption experiment is listed.

**Report Type**

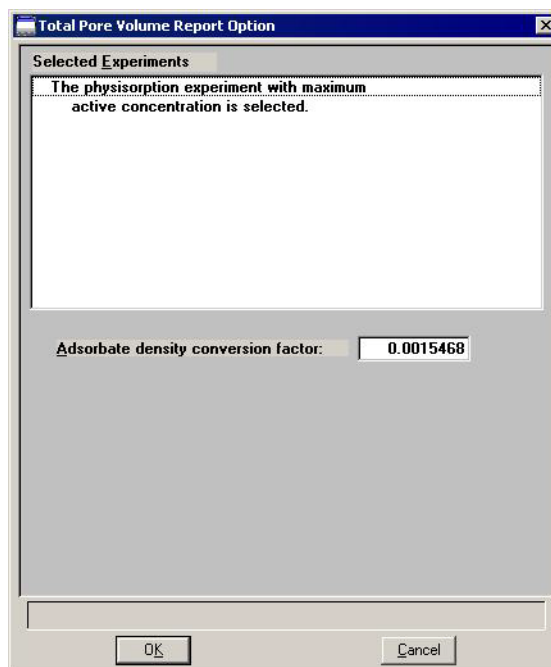
From this group box, choose the type(s) of reports to generate.

**Adsorbate cross-sectional area**

Enter the appropriate value. The adsorbate cross-section is the area occupied by a single adsorbed molecule on the adsorbent surface.

## Total Pore Volume

Clicking **Edit** when Total Pore Volume is selected opens the following dialog:



### Selected Experiments

All applicable experiments are selected by default. When the report options file is used in a sample information file, any experiment may be deselected.

### Adsorbate density conversion factor

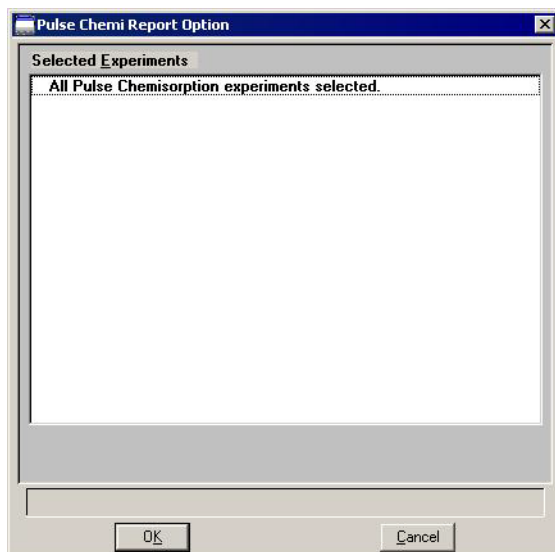
Enter the appropriate value for converting the gas volume to liquid volume. This value can be obtained by dividing the gas density by the liquid density at the adsorption temperature.

$$\text{Density conversion factor} = \frac{\rho_{gas}}{\rho_l} \times \frac{T_{BATH}}{T_{STP}}$$

## Pulse Chemisorption Report Option

---

Clicking **Edit** when Pulse Chemisorption is selected opens the following dialog:



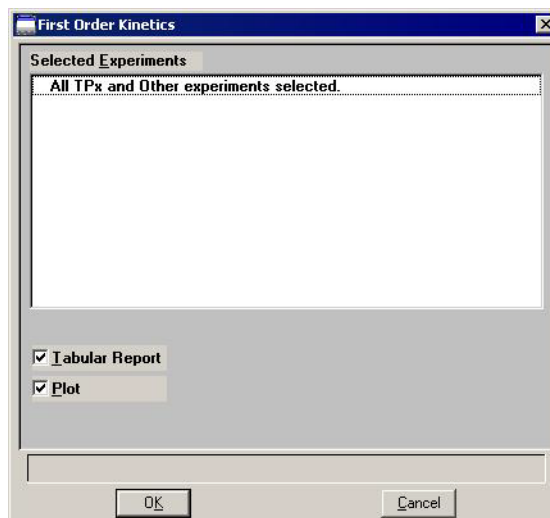
### Selected Experiments

All applicable experiments are selected by default. When the report options file is used in a sample information file, each pulse chemisorption experiment is listed. Any experiment may be deselected.

## First Order Kinetics Report Option

---

Clicking **Edit** when First Order Kinetics (heat of adsorption) is selected opens the following dialog:



### Selected Experiments

All applicable experiments are selected by default. When the report options file is used in a sample information file, each TPx and Other experiment is listed. Any experiment may be deselected.

### Tabular Report

Select this option to have a tabular (data in a table form) report generated.

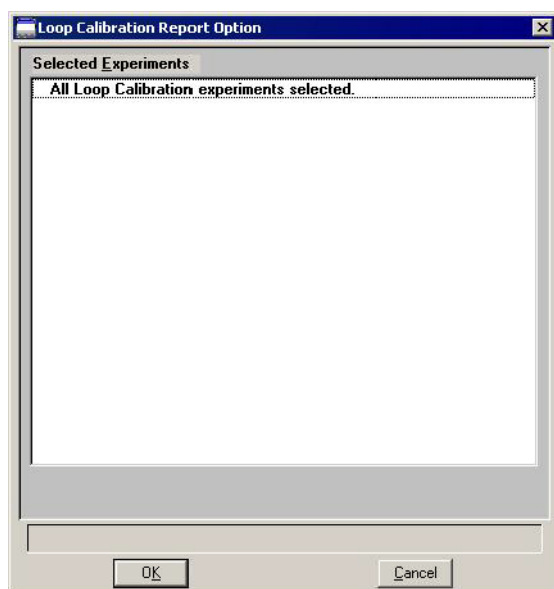
### Plot

Select this option to have the data plotted in a graphical format.

## Loop Calibration Report Option

---

Clicking **Edit** when Loop Calibration is selected opens the following dialog:



### Selected Experiments

All loop calibrations are selected by default. When the report options file is used in a sample information file, each loop calibration is listed. Any calibration may be deselected.

## Analysis Log Report

---

When **Analysis Log** is selected, a log of analysis operations is included in the reports. The Analysis Log cannot be edited.



## Graph Report Options

The AutoChem 2950 HP program contains 10 user-defined graphs, each definable with regard to the x-axis, y-axis and y-overlay. The graphs are named as follows:

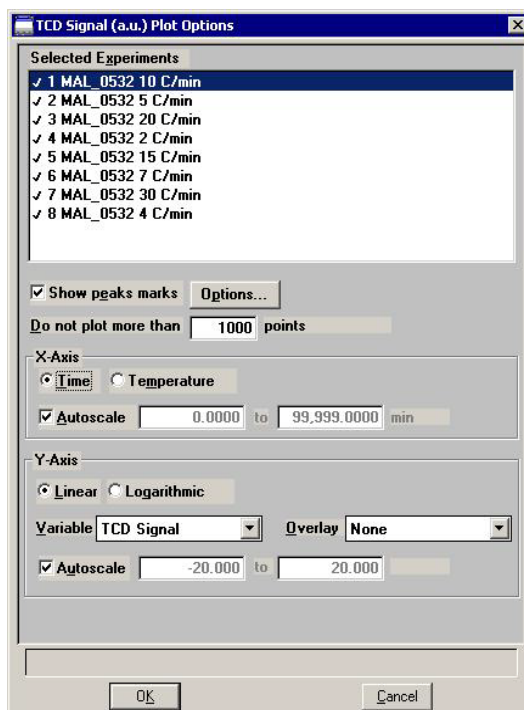
<Y-axis choice> and <Y-axis Overlay Choice> vs. X-axis choice>. For example: TCD Signal and Pressure vs. Time.

Ten default files, which may be edited, are included in the program. They are:

### Report

Pressure vs. Time  
Pressure vs. Temperature  
Input Signal vs. Time  
Input Signal vs. Temperature  
TCD Signal and Pressure vs. Temperature  
Input Signal 1 vs. Time  
Input Signal 1 vs. Temperature  
Input Signal 2 vs. Time  
Input Signal 2 vs. Temperature  
TCD Signal and TCD Concentration vs. Time

All graph report options are similar and are edited in the same manner. Click **Edit** on a selected graph report; a dialog similar to the following is displayed:



Any signal (the detector signal or the 1 or 2 auxiliary signal) may be plotted against time or temperature. Any signal can be overlaid onto the primary signal.

For color output to a monitor or printer, signals are displayed in different colors. For black and white output, different symbols are used.

**Selected Experiments**

All experiments are selected by default. When the report options file is used in a sample information file, each experiment in the sample file is listed. Any experiment may be deselected.

**Show peak marks**

Select this option to:

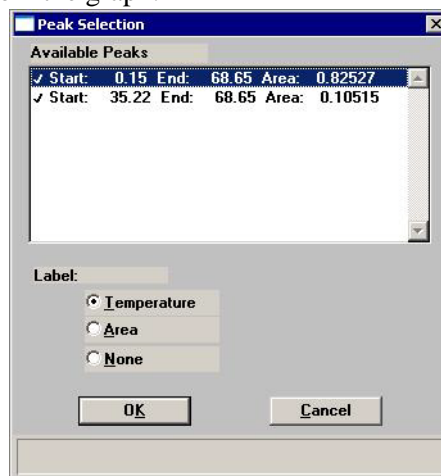
- display the areas and baselines on the graph
- draw a straight baseline between the selected peaks
- enable the **Peaks** button, allowing you to specify the peaks to include in the experiment

**Do not plot more than \_\_\_\_ points**

Allows you to specify a maximum number of points to plot. Typically, the default of 1000 is sufficient. You can specify up to 8000 if higher resolution and more detail is needed. The speed at which graphs are plotted on some computers may be affected when large numbers of points are requested.

**Options**

Opens a dialog that contains a list of all the peaks for the results of the experiment highlighted in the **Experiments** list box. All peaks are selected by default; deselect any peak to exclude from the graph.



The **Label** radio buttons enable you to choose the peak label that will be displayed on graphs.

**Temperature** is often used for TPR, TPO, and TPD. **Area** is often used for pulse chemisorption.

<b>X-Axis</b>	Specify <b>Time</b> or <b>Temperature</b> for the X-axis variable.
<b>Autoscale</b>	<p>Select this option to have the X-Axis scaled automatically. As the default, this axis begins at zero, and the system uses the highest value collected during analysis as the ending point. The Axis from and to fields are disabled if you choose to autoscale data.</p> <p>If you choose not to autoscale data, you must enter from and to values. The value entered in the <b>To</b> field must be greater than the value entered in the <b>From</b> field.</p>
<b>Y-Axis</b>	Select <b>Linear</b> or <b>Logarithmic</b> for the Y-axis.
<b>Variable</b>	Select a Y-axis variable from the drop-down list.
<b>Overlay</b>	You can use this feature to overlay another variable with the current one. Select a variable from the drop-down list.
<b>Autoscale</b>	<p>Select this option to have the Y-Axis scaled automatically. This axis begins at zero by default; the system uses the highest value collected during analysis as the ending point. The Axis <b>From</b> and <b>To</b> fields are disabled if you choose to autoscale data.</p> <p>If you choose not to autoscale data, you must enter from and to values. The value entered in the <b>To</b> field may be less than the value entered in the <b>From</b> field if you plan to invert negative-going peaks.</p>

## Collected Data

Collected Data is a part of a sample information file that does not appear on the Advanced Sample Information dialog until after analysis.

C:\DEMO2950\DATA\CO\_PULSE.SMP  
 << Prev   Sample Information   Analysis Conditions   Report Options   **Collected Data**   Next >>

Po of adsorbate:  mmHg    Ambient temperature:  °C  
 Ambient pressure:  mmHg

Experiment:

Physical Injection Volume:  mL  
 Active Concentration:  %

	Experiment Type	Start Time (min)	Stop Time (min)	Temperature at Maximum (°C)	Area
1	Chemisorption	0.74	1.74	34.6	-0.00806
2	Chemisorption	4.58	6.24	34.4	-0.04606
3	Chemisorption	8.56	10.21	34.4	-0.04668
4	Chemisorption	12.57	14.23	34.5	-0.04699
5	Chemisorption	16.58	18.22	34.5	-0.04723
6	Chemisorption	20.56	22.16	34.5	-0.04718

☐ Use fitted peaks    **Active Metals**

Enter a value between 0.10 and 99999.38.

Save    Close    Basic

**Po of adsorbate**      Displays the saturation pressure of the adsorbate at the time of analysis.

**Ambient temperature**      Displays the room temperature at the time of analysis.

**Ambient pressure**      Displays the room pressure at the time of analysis.

**Experiment**      Defaults to the first experiment of the current file. Click the down arrow to choose other experiments in the file.

**Physical injection volume**      Displays the volume of the syringe used to perform the manual injections.

**Active Concentration**      Displays the percent of the gas mixture that is composed of the reactive gas versus an inert filler.

**Peak table**      Displays the peak summaries for the experiment shown in the Experiment field.

**Use Fitted peaks**

Select this option to report peak data obtained using the Fit peak option in the Peak Editor (refer to **Peak Editor** on page 7-16).

**Active Metals**

Enabled for Pulse Chemisorption reports. Opens the Active Metals Table dialog, which contains the metals entered in the default Active Metals Table. You can edit the values for metal characteristics shown in the table.

	Element	Atomic Weight	Atomic Area (nm <sup>2</sup> )	Density (g/cm <sup>3</sup> )	Stoichiometry Factor	% of Sample Mass
1	chromium	51.996	0.0635	7.200	1.000	0.0000
2	cobalt	58.933	0.0662	8.900	1.000	0.0000
3	copper	63.540	0.0680	8.920	1.000	0.0000
4	molybdenum	95.940	0.0730	10.200	1.000	0.0000
5	nickel	58.710	0.0649	8.900	1.000	0.0000
6	palladium	106.400	0.0787	12.020	1.000	0.0000
7	platinum	195.090	0.0800	21.450	1.000	0.5000
8	rhenium	186.200	0.0649	20.530	1.000	0.0000
9	rhodium	102.905	0.0752	12.400	1.000	0.0000
10	silver	107.868	0.0869	10.500	1.000	0.0000

Ctrl-Down Arrow' to append



The contents of this table are copied from the default table on the Options menu. After this sample file is created, however, the contents of this table become specific to the file. Changes made in this table do not affect the default table and changes made to the default table do not affect this table.

**Insert**

Inserts a new row into the table; the row is inserted above the selected one. The row consists of a drop-down list containing the active metal defaults. Select a metal from the list.

Note that you cannot add a metal at this point. To add a metal select **Options > Active Metal defaults**.

**Delete**

Deletes the selected row.

**Clear**

Clears the table of all entries except one; one is required.

## List

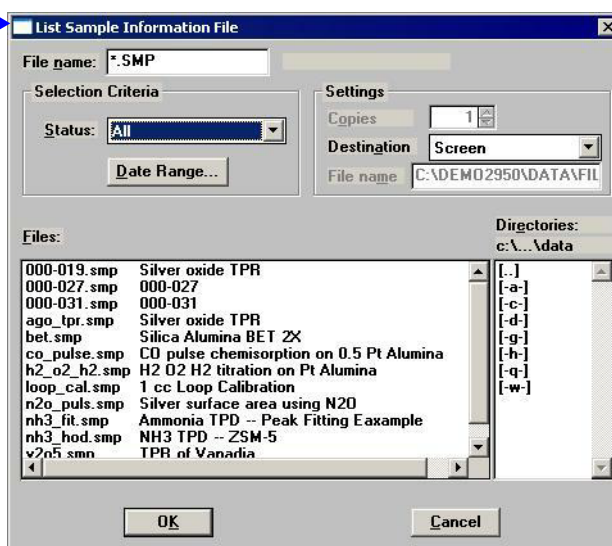
List enables you to generate a listing of the following information on one or more files:

- File name
- Date the file was created (or last edited)
- Time the file was created (or last edited)
- File identification
- File status

When you select **List** from the File menu, three choices are presented: **Sample information**, **Analysis conditions**, and **Report options**.

Regardless of which file type you select, a dialog similar to the one shown below is displayed.

Displays the type of file requested; in this example, a Sample Information File.



### File name

If you select only one file from the Files: list, the name is copied to this field. If multiple files are selected, the last one selected displays.



You may request a nonconsecutive list of multiple files by holding down Ctrl while selecting files. If no files are selected, a list is generated for all files.

### Status

Select the option for the file(s) to be listed.

### Dates

### Files

### Directories

**Settings group box**

The **Destination** field contains a drop-down list of the available print destinations

If you select **Printer**, the **Copies** field is enabled allowing you to enter up to four copies.

If you select **File**, the **File name** field is enabled allowing you to enter a name (or you can accept the default).

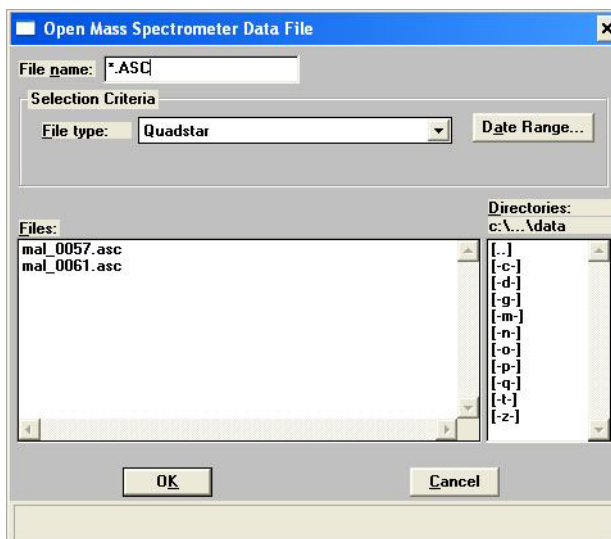
## Import MS Signal

**Import MS Signal** allows you to import data collected by a ThermoStar or MicroStar Mass Spectrometer.



When using a ThermoStar mass spectrometer, in order to have the analog signals import properly, they must be configured in a certain manner in the QuadStar software. Refer to the next page for information on configuring the signals.

Select **Import MS Signal** from the **File** menu; the Open Mass Spectrometer Data File dialog is displayed.

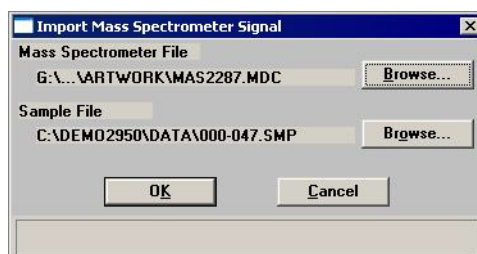
**File name**

Displays the wildcard (\*) with the extension of the selected file type. The file type extensions are:

- MDC (Quadstar)
- ASC (Quadera)
- XLS (TAMS)
- TXT (MKS)

<b>File type</b>	Displays a drop-down list of file types. The file types are those produced from the Mass Spectrometer software used with the 2950.
<b>Date Range</b>	Use to limit the files displayed in the <b>Files</b> list to a specified date range.
<b>Files list</b>	Displays the files available in the current directory.

Select the file and click **OK**, the Import Mass Spectrometer Signal dialog is displayed.



<b>Mass Spectrometer File</b>	Displays the mass spectrometer file you selected from the previous dialog to import into the AutoChem program.
<b>AutoChem Sample</b>	<p>Displays the AutoChem sample file into which the mass spectrometer file will be imported.</p> <p>If you have a sample file open, its name is displayed in this field and the data will be imported into this file when you click <b>OK</b>.</p> <p>If you do not have a sample file open, the next sequenced file number is displayed in this field. When you click <b>OK</b>, the data will automatically import and assume the next sequenced number.</p>
<b>Browse</b>	Use to select a different file from the one(s) displayed.



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## Configuring the Analog Signals

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Analog signals must be configured depending on the software used. The import utility reads the mass spectrometer data and any analog signals collected by the mass spectrometer. The analog signals must be configured for them to be properly imported.

---

### Quadstar

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1. Launch the Parset program using Windows Explorer, double-click on **Parset.exe**.
2. Refer to the Instrument Configuration section of the ThermoStar operator's manual and configure the characteristic curves as follows:

A1 = T  
A0 = TCD

---

### Quadera

---

When selecting the Chart Label name in the Analog I/O Characteristics for a scan to export, ensure that the label for the analog temperature signal is consistent with one of the following rules:

- Is either "T" or "t",
- Begins with "T" or "t" followed by a non-alphabetic character (e.g., "T\_1"),
- Begins with the letters "temp" (e.g., "Temp," "TEMPdegC," "TEMP\_1").

---

### TAMS

---

No additional configuration required.

---

### MKS

---

When exporting from Process Eye Professional--Recall, ensure that the following configuration options are selected for Data:

- Include column headings
- Include scan time
- Millisecond data

## MS Signal Calibration

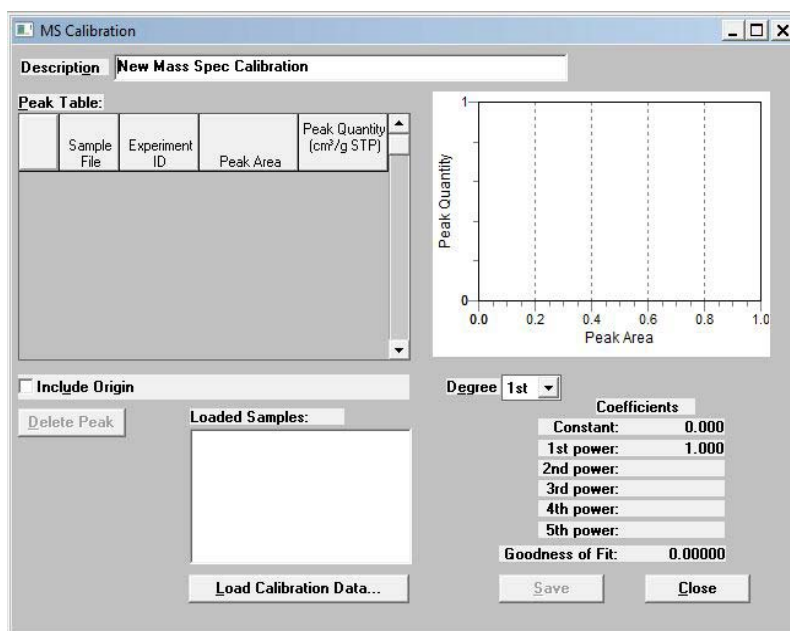
Similar to TCD calibrations (see page 6-23), mass spec calibrations can be created from imported mass spec signals collected during a calibration run.

After importing the mass spec signal using File > Import MS Signal, the created sample files containing the imported mass spec signals can be loaded into the MS Calibration dialog, which is analogous to the TCD Calibration dialog (see page 6-25).

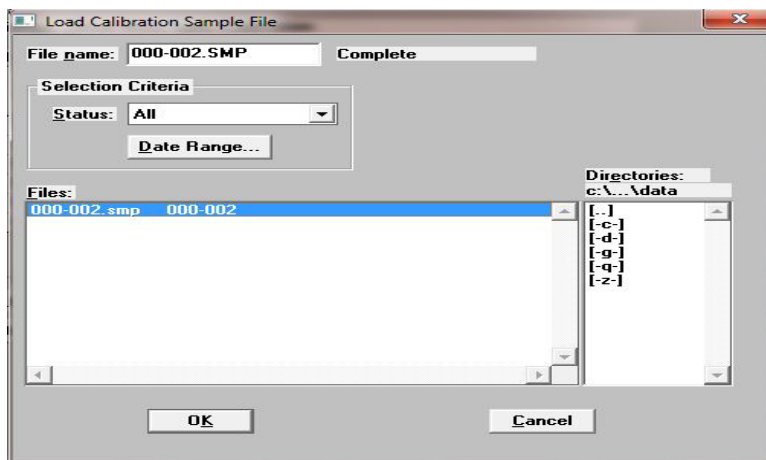
You may then associate the mass spec calibration to a sample file containing imported mass spec signals by clicking Set Calibration on the Peak Editor dialog (described in Chapter 7, [REPORT MENU](#)).

### New

Allows you to create a new MS Signal calibration file; the New MS Signal Calibration dialog is displayed.



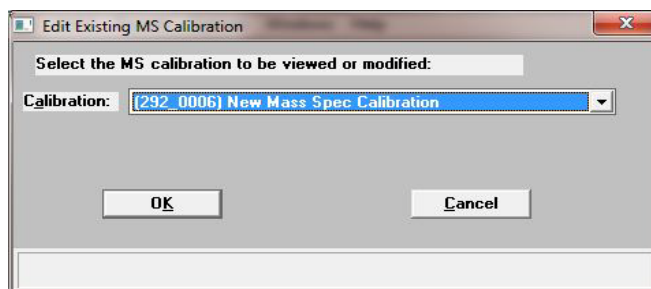
Click **Load Calibration Data** to load a calibration sample. The Load Calibration Sample dialog is displayed.



After you select a sample file, click **OK** to populate the MS Calibration dialog with sample data. MS Signal calibration files are used to convert sample data to volume data. Examples of these calibrations are provided in the tutorials (Appendix J).

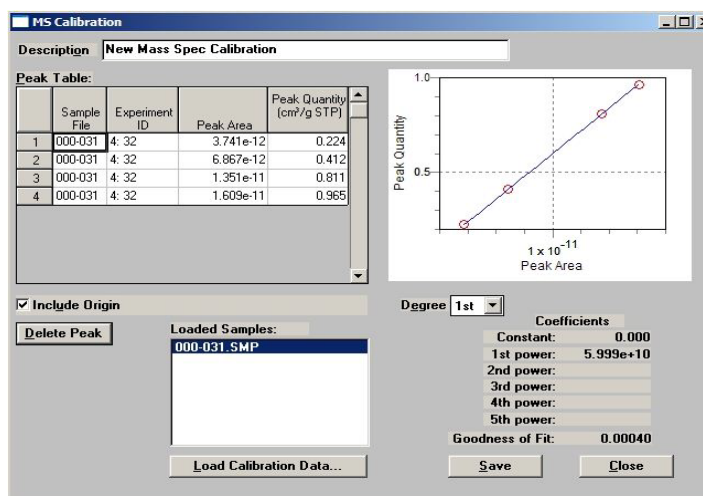
## Open

Allows you to open an existing calibration file for editing; the Edit Existing MS Calibration dialog is displayed. This dialog contains a drop-down list of existing MS calibration files.



After choosing the file, click **OK**; the MS Calibration dialog is displayed.

This type of peak table is displayed for an MS Signal calibration.



### Description

Displays the name of the current calibration file. This field can also be used to enter a new name when changes have been made.

### Load Calibration Data

Allows you to load another calibration file. This sample file must include imported mass spec signals. If a file is selected with no imported mass spec signals, an error message displays.

### Delete Peak

Deletes the selected peak.

### Degree

Allows you to choose a different degree of polynomial; usually the 2nd degree is adequate.

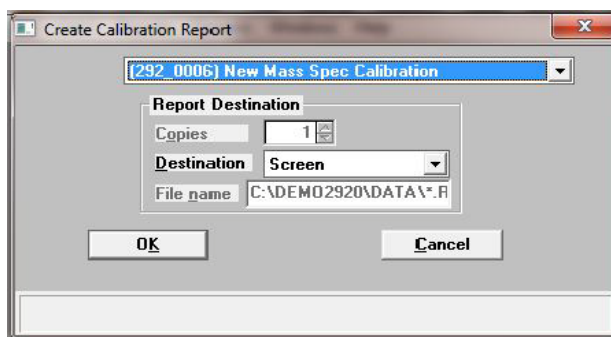
<b>Coefficients</b>	Shows the fit of data to the polynomial.
<b>Save</b>	Enabled when the current file has been edited, allowing you to save the changes. To save it under a different name, enter a new one in the <b>Description</b> field.
<b>Close</b>	Closes the dialog.

---

## Report

---

Select this option to print a report of a calibration file.



<b>Calibration</b>	From the drop-down list, select the file to generate a report.
<b>Report Destination Copies</b>	Enabled if you choose <b>Printer</b> as the report destination. You can print up to four copies.
<b>Destination</b>	Allows you to choose a destination for the report.
<b>File name</b>	Enabled if <b>File</b> is selected as the report destination, allowing you to enter a file name (or accept the default).

## List

Select this option to display a list of the files in the Calibration Directory, including details such as the calibration file's identifier (as entered in the sample information file) and the date the file was created (or last edited).

**Next  
Prev**

Allow you to view the next or previous page of the list (if they exist).

**Close**

Closes the window.

**Print**

Prints the list.

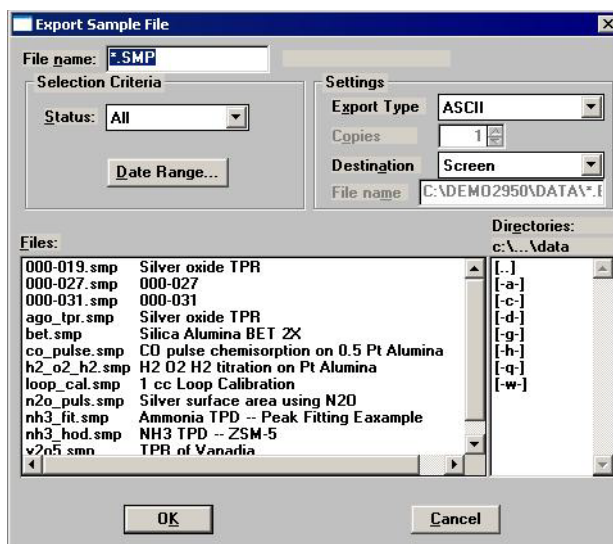
**Header**

This toggle button hides/shows the header of this list. If the header is selected, it appears on every page of the list when it is displayed and/or printed.

## Export

**Export** allows you to copy data in a sample information file and reformat it in ASCII format or Grams/32 format. ASCII data can be imported into other applications, such as spreadsheets. Grams/32 data can be imported into the Grams/32 program (the peak editing program used in previous versions of the software) and then manipulated without use of the AutoChem 2950 HP analysis program. You may select multiple files by holding down **Ctrl** and clicking the left mouse button once on each file. See Appendix D **FORMAT OF EXPORTED DATA** for a description of exported data.

Select **File > Export**; the Export Sample File dialog is displayed.



**File name** The name of the file you select from the Files: list box is copied to this field.

**Status** Select the option for the file(s) to be exported.

**Dates**

**Files**

**Directories**

**Export Type** You can export the data from a 2950 sample information file in an ASCII or Grams/32 format.

Choose **ASCII** to create a data file that can be imported into an application such as a spreadsheet program.

Choose **Grams/32** to create a data file that can be imported into the Grams/32 program.

**Destination** Contains a drop-down list of the available destinations.



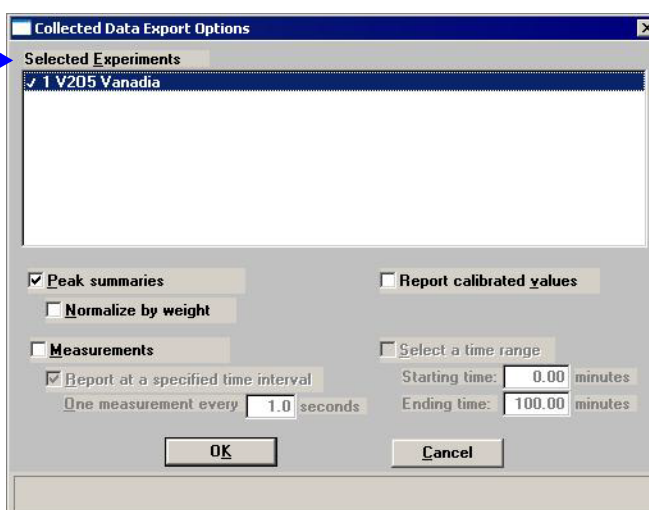
A sample information file exported as a Grams/32 type can only be generated to a File destination. When you export a Grams/32 type, five separate files (which can be read only by the Grams/32 software) are generated. Refer to Appendix D, **FORMAT OF EXPORTED DATA** for information on the contents of these files.

If you select **Printer**, the **Copies** field is enabled allowing you to enter up to four copies.

If you select **File**, enter a name in the **File name** field of the Settings group box (or accept the default).

When you click **OK**, the Collected Data Export Options dialog is displayed.

Only the Selected Experiments portion of this dialog displays if you choose Grams/32 as the type of Export.



### Selected Experiments

Displays a list of the experiments included in this sample file. All experiments are selected by default.

This portion of the dialog is the only field displayed if you are exporting a file as a Grams/32 type.

### Peak summaries

Exports a summary of the peaks contained in the selected experiment(s).

### Normalize by weight

This choice should be used for normalizing peak summaries. Divides the peak area by the sample weight to correct the signal strength for different sample sizes.

### Report calibrated values

Exports the calibrated values for the TCD signal if calibration data are available.



**Measurements**

Selecting this option enables the time interval and time range fields.

**Report at a specified interval**

Select this option to report measurements at a specified interval; a field is enabled so that you may enter an interval (the number of seconds between reported data readings).

Use this feature if a large amount of data was collected during the analysis (such as in a lengthy analysis with readings taken every second). If you do not specify an interval for data reporting, points are reported as follows:

- Up to 500 lines per experiment if destination is to the **screen** or to a **printer**.
- Up to 10,000 total lines if destination is to a file.

**Select a time range**

Select this option to specify relative starting and ending times of the measurements to export. This feature allows you to specify a region of interest to be extracted from a large data set.

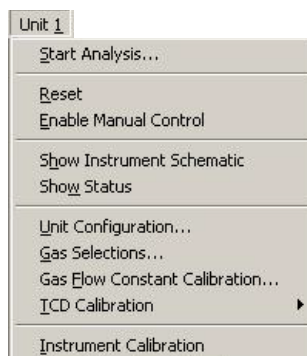


## 6. UNIT MENU

The Unit menu contains options that enable you to perform analyses, display the system schematic and status bar, manually control the analyzer, and perform calibration procedures.

### Description

---



Listed below are brief descriptions of the Unit menu options. Detailed descriptions follow this section.

**Start Analysis**

Opens a series of dialog boxes that enable you to start an analysis. Page [6-2](#).

**Reset**

After confirmation, resets the system to a safe state. Page [6-11](#).

**Enable Manual control**

Enables you to control various instrument components manually, using the instrument schematic (disabled during an analysis that is not suspended). Page [6-12](#).

**Show Instrument schematic**

Displays the instrument schematic. Page [6-17](#).

**Show Status**

Displays only the Status Bar of the operational window. Page [6-19](#).

**Unit Configuration**

Displays the hardware configuration, as established when the instrument was installed, including software version, IP address, and installed options. Page [6-20](#).

Also allows you to enter the loop (if present) volume.

<b>Gas Selections</b>	Allows you to specify which gases are connected to the analyzer ports. Page <a href="#">6-21</a> .
<b>Gas Flow Constant calibration</b>	Displays a dialog enabling you to obtain the conversion constant of a gas. The conversion constant is used by the Mass Flow Controllers to ensure accurate flow control. Page <a href="#">6-22</a> .
<b>TCD Calibration</b>	Displays a series of dialogs allowing you to create, edit, and manage calibration files. Calibration files enable the software to convert sample signal, temperature, and peak area data to volume data. Page <a href="#">6-23</a> .
<b>Instrument Calibration</b>	Displays a dialog for calibrating various instrument components. This dialog is used to calibrate the sample thermocouple. Page <a href="#">6-30</a> .

## Start Analysis

When you select **Start Analysis**, the Analysis dialog is displayed with a file selection dialog positioned on top. You can either select a sample file that was created and saved earlier, or you can create a new sample file now.

Which option you choose depends on the method you prefer for your laboratory. The analysis process is the same either way. For example, your lab may prefer to create numerous sample files in advance, then analyze the samples when they become available. Alternatively, your lab may prefer to create sample files for each sample as you begin the analysis.

After you choose a sample file and click **OK**, the Analysis dialog becomes totally visible.

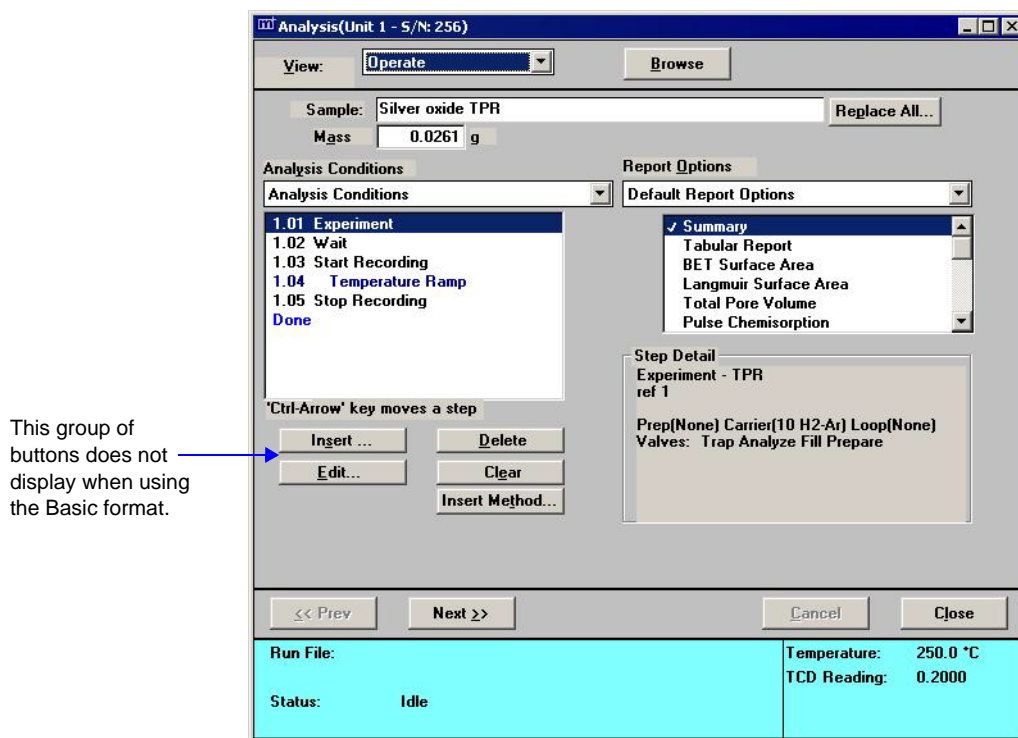


**If there are any inconsistencies between the sample information file you choose and the way the instrument is configured, a message is displayed instructing you to correct them. For example, if the sample information file specifies *helium* for the Preparation gas, but the Gas Selections dialog indicates that helium is not connected to a Preparation gas port, the software informs you of the discrepancy**



**The software is unable to determine if the wrong gas is physically connected to the port. It can only check that the gas selected agrees with the gas identified for each port in the Gas Selections dialog. It is important ensure that the Gas Selections dialog accurately reflects which gas is physically connected to each port.**

The appearance of the Analysis dialog depends on whether you are using the Advanced or Basic mode. The same fields appear on both dialogs; however, the Advanced dialog contains additional buttons that allow you to insert and edit individual steps.

**View**

Allows you to choose from four views to display in the current window

**Operate**

Displays the dialogs associated with the current operation.

**Instrument schematic**

Displays the instrument schematic. Refer to **Show instrument schematic** on page 6-17 for a description of the components contained on the schematic.

**Results**

Displays analysis results as they are collected.

**Sample log**

Displays a log of each step as it is completed, as well as any error messages or manual operations that occur during the analysis.

**Sample**

Displays the description for the current file.

**Mass**

Enables you to enter an accurate weight of the sample to be analyzed.

<b>Browse</b>	Allows you to choose a different sample file for the current analysis.
<b>Replace All</b>	Allows you to replace the contents of the current file with those of another one. After the values have been copied into the current file, they may be edited. Editing the values will not affect the file from which they were copied.
<b>Analysis Conditions</b>	<p>Displays the current Analysis Conditions file. This drop-down list contains predefined parameter files, some of which are supplied with the analysis program. For newly created files to display in this list, they must be saved to the directory specified as the parameter files directory (refer to <a href="#">“Parameter Files Directory” on page 8-17</a>). Click the down arrow to the right of the field to choose a different file.</p> <p>The window directly below this list contains the conditions specified in the Analysis Conditions file. If you are using the Advanced format for the current analysis, you can edit the conditions using the buttons provided below the Analysis Conditions window. Refer to page <a href="#">5-11</a> for details on these buttons.</p> <p>You can also edit analysis conditions after the analysis has begun. Click <b>Suspend</b> to suspend the analysis; this enables the editing buttons. You can edit only the steps or experiments following the current one. After editing is complete, resume the analysis.</p>
<b>Report Options</b>	<p>Displays the current Report options file. This drop-down list contains predefined parameter files, some of which are supplied with the analysis program. For newly created files to display in this list, they must be saved to the directory specified as the parameter files directory (refer to <a href="#">Parameter Files Directory on page 8-17</a> for additional information). Click the down arrow to the right of the field to choose a different file.</p> <p>The window directly below this list contains the reports requested for the current Report Options file. You can select additional reports or deselect currently selected ones. A report is selected when it is preceded with a check mark. Simply double-click to select (or deselect) a report. Alternatively, you can highlight the report and press the <b>Spacebar</b>.</p>

Click **Next** to advance to the second view of the Analysis dialog. This view enables you to choose a TCD calibration for each experiment in this sample's analysis conditions set. A TCD calibration allows the areas of the peaks recorded by the instrument to be converted into volumes. A TCD calibration is not required by all analysis types, nor is it necessary to select the calibration from this dialog. A calibration can be associated with an experiment during peak editing (after analysis has ended).

Exp.	Type	Name	Calibration
1.	Level Cal.	<Description>	None
2.	Other	<Description>	None

<< Prev    Next >>    Cancel    Close

Run File: C:\DEMO2950\DATA\000-052.SMP    Temperature: 250.0 °C  
Sample: 000-052    TCD Reading: 0.2000  
Status: Idle

The default(s) for the calibration file(s) are those specified using the TCD Calibration Defaults option on the Unit menu (discussed later in this chapter).

The files available in each drop-down list are those available for the instrument.

Click **Next** to advance to the next view of the Analysis dialog. This view enables you to generate reports automatically when the analysis is complete. The reports generated are those selected in the report options portion of the sample information file.

Analysis(Unit 1 - S/N: 256)

View: Operate

☒ Report After Analysis

Number of Reports: 1

Destination: Screen

File name: C:\DEMO2950\\*.RPT

<< Prev    Next >>    Cancel    Close

Run File:	C:\DEMO2950\DATA\000-052.SMP	Temperature:	250.0 °C
Sample:	000-052	TCD Reading:	0.2000
Status:	Idle		

**Report After Analysis**

Choose this option to have the reports specified in the sample file generated automatically after the analysis is complete.

**Number of Reports**

Enabled when **Printer** is selected as the report destination. You can print up to four copies.

**Destination**

Allows you to choose a destination for the reports.

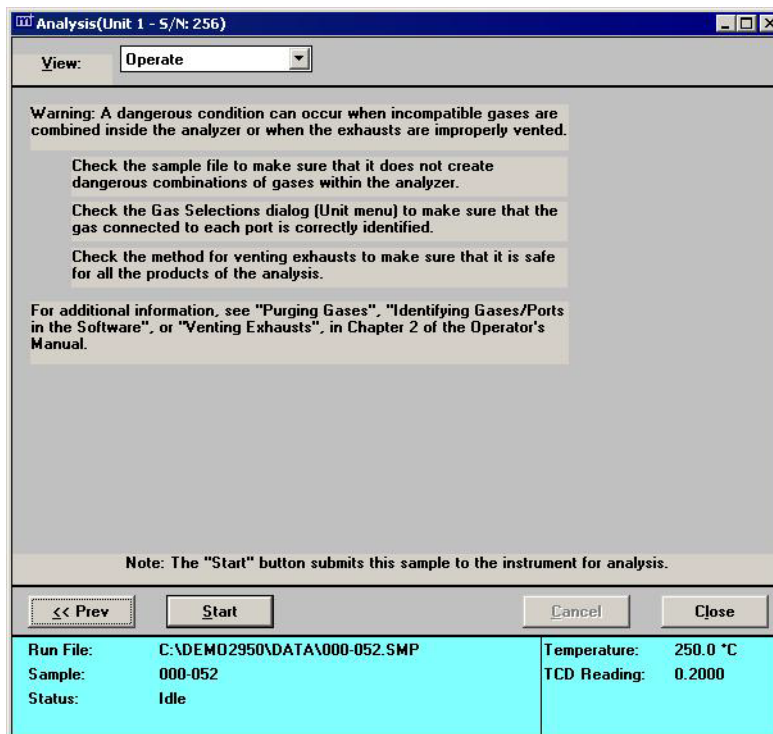
If you choose **Screen** for the destination, many options are available for manipulating and customizing your reports.

**File name**

Enabled when **File** is selected as the report destination, allowing you to enter a file name (or you can accept the default).

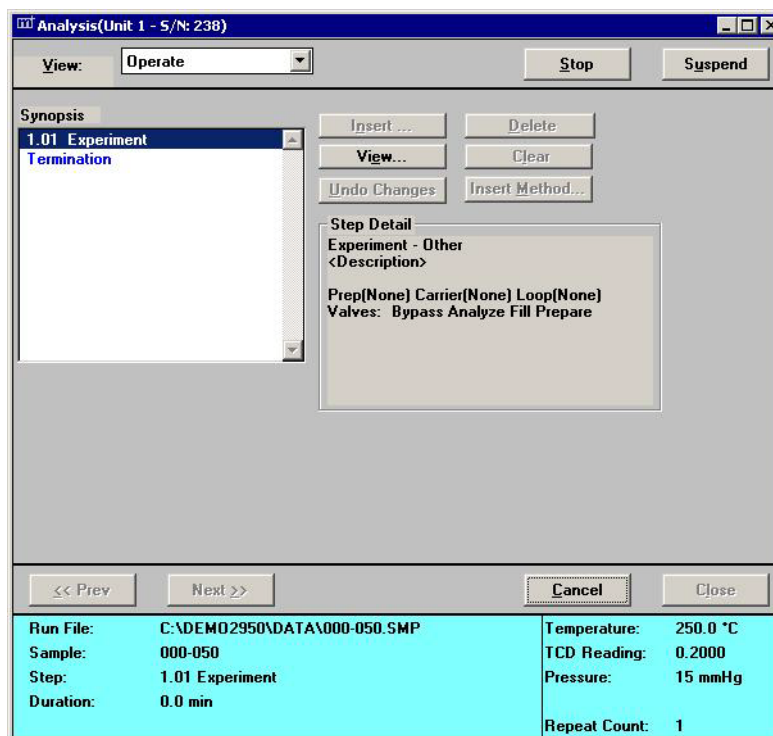


Click **Next**; a dialog instructing you to verify the following is displayed:



- Verify that the gas bottles connected to the analyzer are correctly identified in the software.
- Verify in the sample file that the combinations or sequence of gases in the analyzer are not dangerous, and that the sample will not react with any of the gases in a dangerous way.
- Verify that the current method for venting the analyzer's exhausts is adequate and safe for all products of the analysis.

After you have verified that all conditions are safe for the analysis, click **Start** to begin the analysis. The original view of the Analysis window is displayed, except that now it tracks the progress of the analysis.



### Synopsis

Shows the list of experiment steps in the sample information file. Upcoming steps are shown in blue or black. The current step is highlighted and shown in light blue; completed steps are shown in light green.

You may scroll through the list to review the analysis steps. You can also edit steps that have not been started; the analysis must be suspended to do this (see Suspend below).

### Step Detail

Shows detailed information about the current step.

### Stop

Stops operation of the current step and automatically suspends the analysis. Click **Resume** to restart the analysis. The analysis resumes at the beginning of the next step. This button is used to skip the current step.

Manual Control is allowed while an analysis is suspended.

**Cancel**

Cancels the analysis; the current step is not completed. After confirmation, the analysis ends and leaves the analyzer in the current state. The analysis cannot be resumed. To return the analyzer to a safe state automatically, select **Reset** from the **Unit** menu.

**Suspend**

Suspends the analysis. This button is a toggle button; it reads **Resume** when the analysis is suspended.

To suspend the analysis to edit steps in the list or add steps to the list. Steps can only be added or edited in the portion of the list that has not yet been executed; such editing is available only if you are using Advanced presentation format. The current step cannot be edited. Click **Resume** to continue the analysis after editing has been completed.

Manual Control is allowed while an analysis is suspended.



During analysis, analysis conditions can be edited only using this view of the Start Analysis dialogs. Analysis conditions cannot be edited in the sample file during analysis.



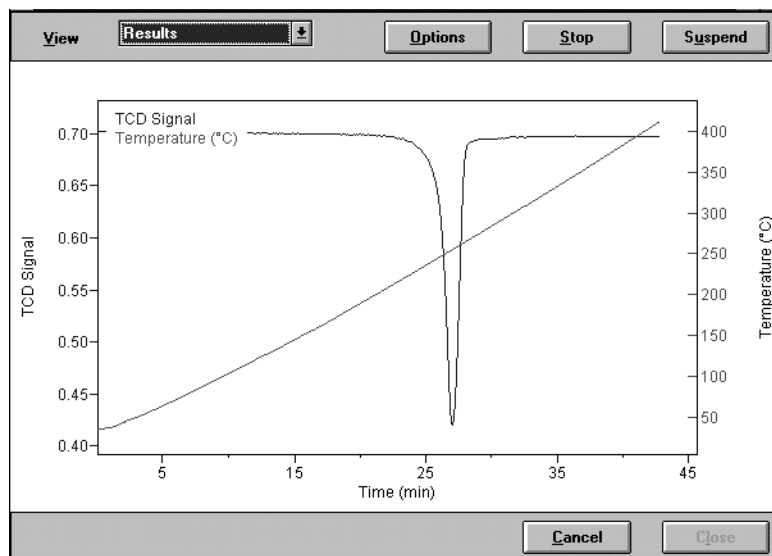
There is a very brief pause while the analyzer suspends or resumes the analysis. Do not relick the button.

**Messages**

If you have included an experiment step that requires a message to the user, the message is displayed on the monitor when the appropriate point in the analysis is reached. For example, if you have included a Dose step that takes a BET measurement, you are prompted to change the Dewar at the appropriate time. The operator performs the required function, then clicks **OK** to resume analysis.

## Viewing the Results

To view data as they are collected, select **Results** from the View drop-down list. Up to four signals may be displayed on the graph: TCD, Sample Temperature, Auxiliary 1 and Auxiliary 2. The scales for the first two signals are shown on the right and left Y-axes, respectively. If more than two signals are selected, the additional signals are shown without scales.

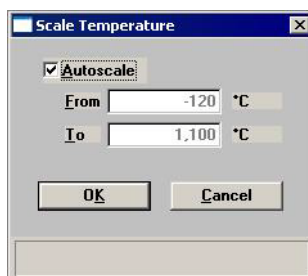


Click **Options** to customize the presentation of data in the Results view. You can change the axes and select or deselect the data that are presented. If you specify a value for Starting at (X-Axis), only those data collected after that point are shown. If the Starting at value is greater than the experiment duration, then all data are shown.

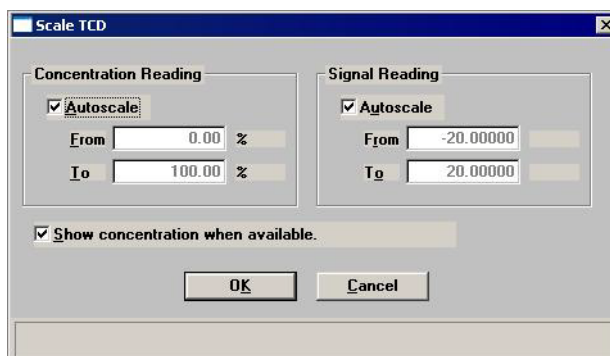
The Smoothing option uses the Savitsky-Golay smoothing filter, which may improve data presentation.

The screenshot shows the 'Results Options' dialog box. It has a title bar with a close button. The dialog is divided into several sections. The 'Y-Axis' section has four checkboxes: 'TCD Reading' (checked), 'Temperature' (checked), 'Pressure' (unchecked), and 'Input Signal' (unchecked). Each checkbox has a 'Scaling...' button next to it. The 'X-Axis' section has two radio buttons: 'Time' (selected) and 'Temperature' (unselected). Below this is a 'Starting at' field with a value of '0.00' and a unit of 'min'. The 'Smoothing Options' section has a 'Smoothing' checkbox (unchecked) and two input fields: 'Window Size' with a value of '11' and 'Order Of Smoothing' with a value of '3'. At the bottom are 'OK' and 'Cancel' buttons.

Click **Scaling** to adjust the scale of a selected signal. The example shown below is the Scaling dialog associated with the Temperature signal.



In addition to allowing you to adjust the scale of the Concentration and Signal Results, the Scale TCD dialog lets you indicate (by checking the box) whether or not to display the Concentration. (The concentration will be displayed if a Gas Concentration calibration has been associated with the experiment.)



## Reset

Use this option to abort the current analysis and return the analyzer to a safe state. The following actions occur:

- All gas flows stop
- The detector is disabled
- The heat zones are reset to 0 °C
- The target furnace temperature is reset to 20 °C

Do not select this option unless you are sure you want to stop the current analysis. The analysis cannot be resumed after **Reset** is selected.

## Enable Manual Control

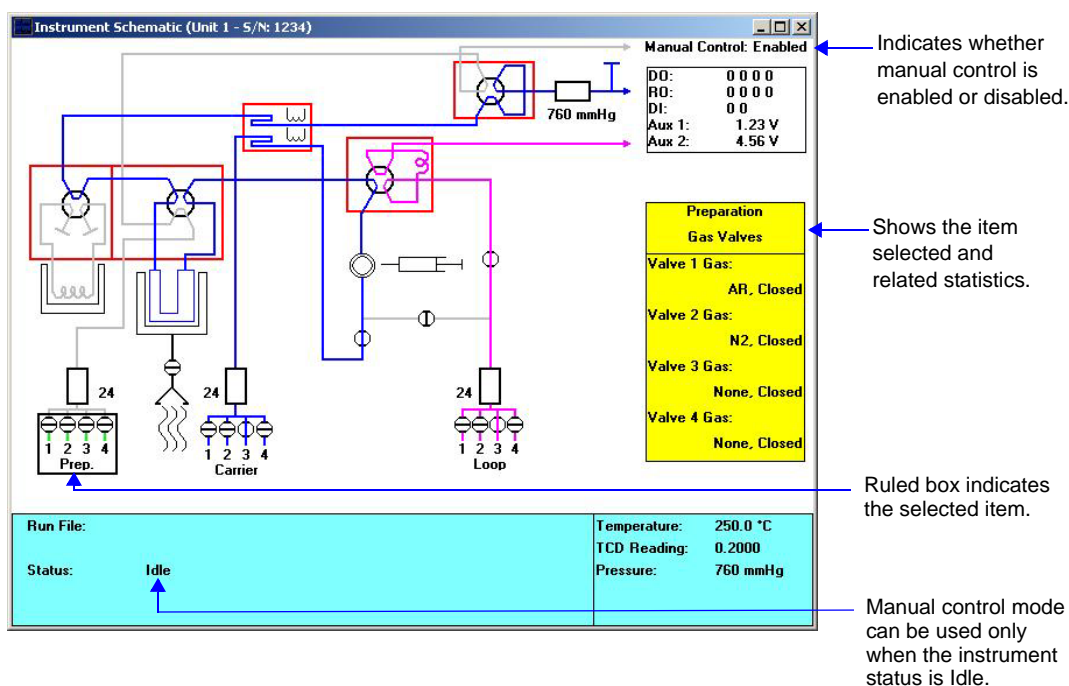
Select **Unit > Enable Manual Control** to manually control components such as the valves, Mass Flow Controllers, heat zones, etc. If the instrument schematic is not displayed, select **Unit > Show Instrument Schematic**.



**Extreme caution should be used when Manual Control is enabled, so that a dangerous condition is not created. If you are not qualified to prevent dangerous conditions (such as volatile combinations of gases), DO NOT attempt to operate the analyzer using Manual Control.**

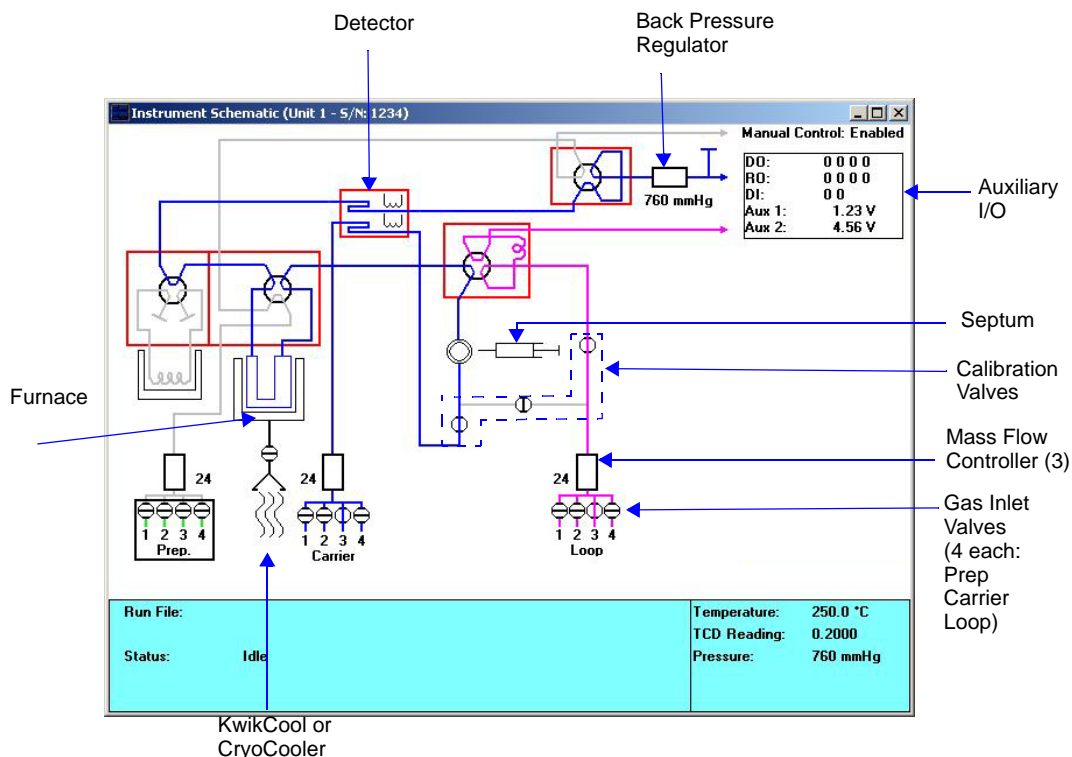


**Manual Control cannot be selected during an automatic operation unless the operation is suspended.**



You can select system components using the mouse or the tab key. A component is selected when it is surrounded by a ruled box. The information window on the right of the schematic indicates the name of the selected component in the upper pane and related statistics in the lower pane.

Most components on the instrument schematic can be manually controlled. These components each have a shortcut menu displaying the operations available for that component. The shortcut menu is accessed by right-clicking on the component, or by using the shortcut keys, **Shift F9**.

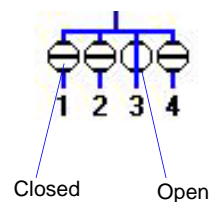


### Gas Pathways

The colored lines that lead from the various gas inlets through the analyzer and to the exhaust represent the current flow path. Each gas source is represented by a different color:

- Pink** - Loop gas
- Blue** - Carrier gas
- Green** - Preparation gas
- Black** - Blended gas

### Gas Valves (Inlet)



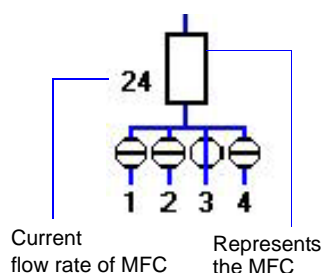
Click a gas valve icon to display (in the yellow window on the right) the name of the gas connected to each of that valve's four ports. The port's state (Open or Closed) is also displayed.

From left to right, the valves control the flow of Preparation gas, the Carrier gas, and the Loop gas.

**Flow Gas [n]**  
**Stop Flow**

Select the gas to flow.  
 Closes the open valve.

## Mass Flow Controllers



The Mass Flow Controllers control the flow of gas into the analyzer. Clicking on a MFC icon displays its current flow rate and the target rate for the current experiment. The current flow rate is also shown next to the MFC symbol. If the flow has been turned off (all inlet valves are closed), the target rate shown is the last non-zero target.

From left to right, the MFC's control the flow rate of the Preparation gas, the Carrier gas, and the Loop gas.

### Set Flow Rate

Displays a dialog so that you can enter a flow rate.

### Stop Flow

Stops the flow of gas through the selected Mass Flow controller.

## Rotary Valves



The Rotary Valves control the flow of gas through the cold trap, the sample tube, and the loop. Each valve has two states. Clicking the valve icon displays its state. Right-clicking the valve icon enables you to change the state of the valve. Click on the state.

From left to right, the valves and their states are as follows:

<b>Cold Trap</b>	Bypass, Trap
<b>Analysis</b>	Analyze, Prepare
<b>Loop</b>	Fill, Inject
<b>Back Pressure</b>	Analyze, Prepare

## Heat Zones



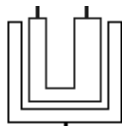
Heat zones are enclosed with a red rectangle. This example shows the loop valve heat zone.

Heat Zones control the temperature of various parts of the analyzer. Component heat zones are enclosed with a red rectangle. Click a Heat Zone icon to display its current flow temperature and the target temperature for the current experiment step.

Right-click, then choose **Set Temperature**, to change the heat zone's target temperature. We recommend using one temperature (100 °C, for example) for all heat zones to ensure a cleaner system.

From left to right on the display, the Heat Zones control the Cold Trap, the Analysis Valve, Detector, Loop Valve, and Back Pressure Valve.



**Sample Furnace**

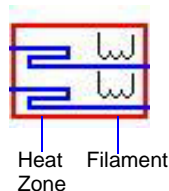
The Sample Furnace controls the temperature of the sample. Click its icon to display the Sample Temperature, the Target Temperature, the Furnace Temperature, and the Ramp Rate.

Right-click, then choose **Set Temperature** to change the Set Point and/or the Ramp Rate.

*Set point range:* -120 to 1100 °C with optional CryoCooler  
ambient to 1100 °C without CryoCooler)



**Do not use stainless steel sample tubes with temperatures above 850 °C. Doing so could weaken, corrode or break the sample tube.**

**Detector**

There are actually two areas of the detector icon that can be controlled. Click the filaments (right side) to specify their temperature. Click the other side of the rectangle to control the detector heat zone's temperature. In either case, right-click, then click on **Set Temperature** to change the temperature. The Filament Temperature must be set at least 20 °C above that of the detector heat zone.



**A carrier gas must be flowing through the detector whenever the detector filaments are turned on; otherwise, the filaments will deteriorate and lose sensitivity. If the gases are shut off, the filaments are automatically turned off after five minutes.**

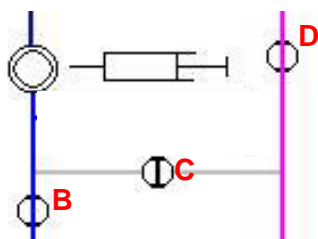
The flowing gas also continuously cleans the plumbing of any water vapor which can enter when the sample tube is removed. Ten cm<sup>3</sup>/min is sufficient to perform this function.

**Auxiliary I/O**

DO:	0 0 0 0
RO:	0 0 0 0
DI:	0 0
Aux 1:	1.23 V
Aux 2:	4.56 V

The Auxiliary I/O display provides a list of the current auxiliary settings for Digital and Relay Output (DO and RO) and for Digital Input (DI). The current Auxiliary 1 and Auxiliary 2 signals are also displayed, if they were recorded. A complete discussion of the auxiliary inputs and outputs is contained in Appendix E, page [E-1](#).

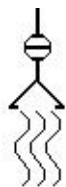
### Calibration Valves



Click a Calibration Valve icon to display its state (Open or Closed). There are three Calibration Valves, lettered, from left to right, B, C, and D. These valves are used by the software during automatic calibration processes.

Generally, it is not necessary for the operator to change the state of the calibration valves.

### KwikCool



The KwikCool (if installed) icon appears on the schematic below the sample tube. The two states of the KwikCool valve are open or closed.

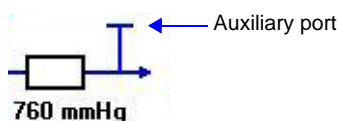
### CryoCooler



The CryoCooler (if installed) icon appears on the schematic below the sample tube. Right-click, then click **Set Speed** to turn on the CryoCooler and select the speed. Click **Stop** to turn off the CryoCooler.

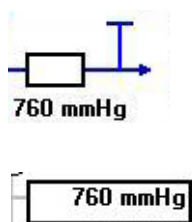
The following components on the instrument schematic are for informational purposes only; they cannot be controlled using the manual control function.

### Auxiliary Port



The Auxiliary Port enables you to connect an external detector, such as a mass spectrometer, to the analyzer. It is located on the left side panel as you face the unit. On the instrument schematic, it is located downstream of the back pressure controller.

### Pressure Transducers



The back pressure regulator symbol displays the system pressure.

The pressure transducer symbol displays the current pressure in the carrier gas path, upstream from the septum, but downstream from the Mass Flow Controller if it is different from the back pressure regulator reading.

During analysis, this symbol displays the difference between the manifold pressure and the back pressure regulator.

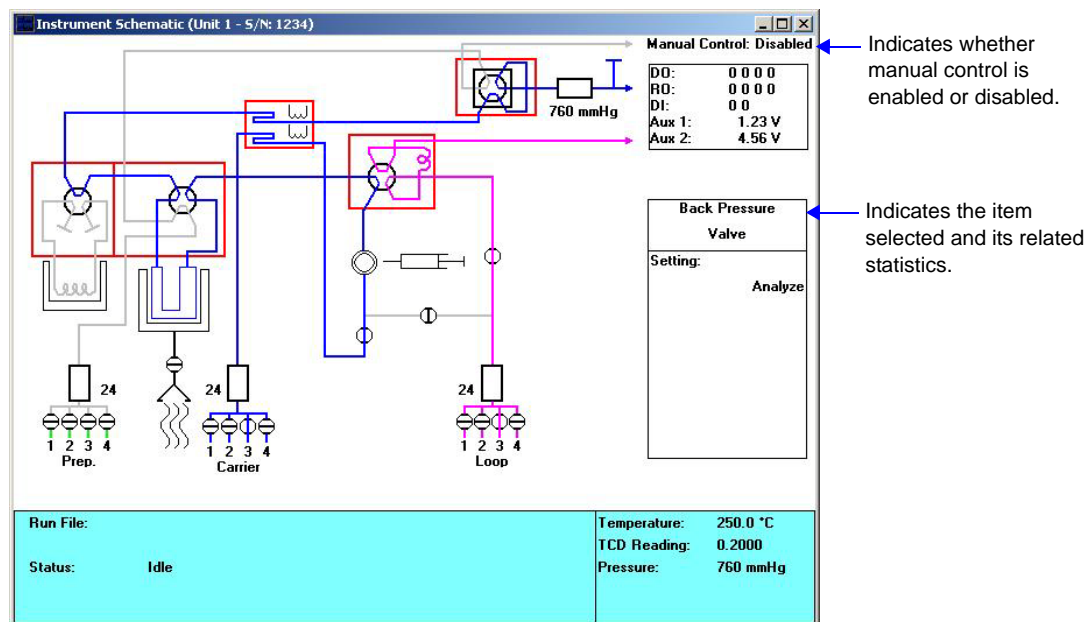
**Septum**

The septum is used for making manual injections of gas into the gas stream. Refer to **“Using the Septum” on page 3-33**.



## Show Instrument Schematic

Select this option to display the instrument schematic. The components of the instrument schematic are described in the previous section, **Enable Manual Control**.



The instrument schematic is a visual representation of the internal components of the analyzer. Remember that the schematic does not show the components' actual appearance or position. Instead, it provides a simplified graphic illustration.

In automatic mode, the schematic displays information about the instrument's status, as well as providing updated information during an analysis. In Manual Mode, the schematic makes it easy to change the status of various instrument components.

## Gas Pathways

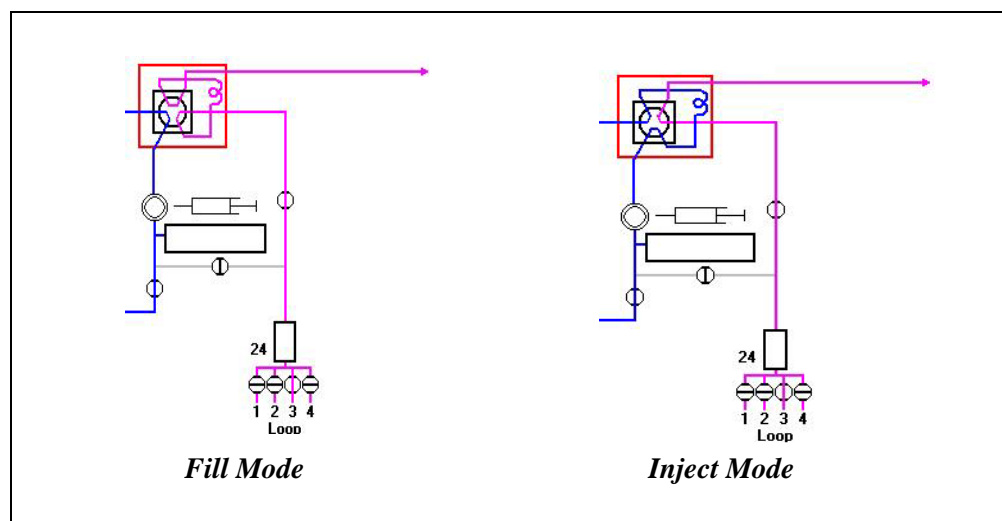
The colored lines that lead from the various gas inlets through the analyzer and to the exhaust represent the current flow path. Each gas source is represented by a different color:

<b>Pink</b>	Loop gas
<b>Blue</b>	Carrier gas
<b>Green</b>	Preparation gas
<b>Black</b>	Blended gas



**When a gas valve is opened or the flow path is changed, it takes several minutes for the new gas to move through the entire system.**

The behavior of the Loop valve is one example. When an analysis includes a loop injection, the instrument schematic shows the path that is available in Fill mode, then in Inject mode. In Fill mode, the analysis gas is shown entering the loop, and excess gas is vented out the exhaust. In Inject mode, the analysis gas is shown bypassing the loop and exiting the analyzer through the exhaust. At that time, the carrier gas path is available from the inlet all the way through the loop, through the sample tube, and past the detector.



When the valve switches to Inject mode, the quantity of analysis gas that is contained in the loop — one exact unit — is pushed out of the loop and through the sample tube by the carrier gas. When the software is recording the TCD signal, the loop injection shows as a peak on the Results view of the Analysis window.

The analysis gas may, in fact, be dissipated greatly as it travels out of the loop and through the sample tube, particularly if the carrier gas flow rate is low. Higher carrier gas flow rates minimize the dispersion of the loop gas.

## Show Status

The bottom portion of the Analysis dialogs displays the instrument's Status Bar. The Show Status option allows you to display the Status Bar only.

Run File:	C:\DEMO\2950\DATA\000-064.SMP	Temperature:	250.0 °C
Sample:	Silver oxide TPR	TCD Reading:	0.2000
Step:	1.01 Experiment		
Duration:	0.0 min	Repeat Count:	1



**If more than one instrument is attached to your computer, each instrument's Status Bar is displayed in a different color to help you differentiate the instruments' displays.**

The Status Bar displays:

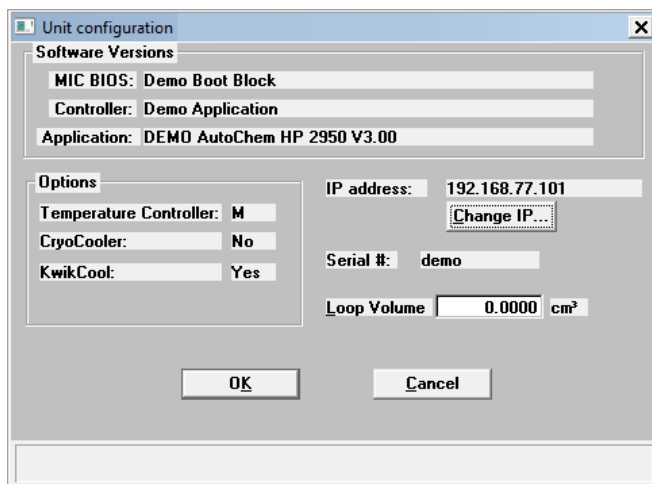
<b>Run File</b>	Gives the name of the sample information file currently being used for analysis. If no file is currently selected, this field remains blank.
<b>Temperature</b>	Gives the sample's temperature, as measured by the sample thermocouple.
<b>TCD Reading</b>	Displays the current detector reading (0.0000 if disabled).

During analysis, the Status Bar also displays:

<b>Sample (Identifier)</b>	Gives the identifier of the sample information file currently being used for analysis. If no file is currently selected, this field remains blank.
<b>Time</b>	Displays the number of seconds that have elapsed since data recording began. This field is displayed <i>only</i> when data is being recorded.
<b>Step</b>	Shows the step currently being performed. When no analysis is in progress, Status: Idle is displayed instead.
<b>Duration</b>	Shows the amount of time that has elapsed since the current step was started. If the analysis is waiting for a fixed period of time, the time left to wait is shown in parentheses next to the time.
<b>Repeat Count</b>	Shows how many times the current Repeat loop has been entered.

## Unit Configuration

Select this option to display the configuration of the instrument's software, IP address, and options.



**Software Versions**  
**MIC BIOS**

Gives the name and version of the instrument's internal software.

**Controller**

Gives the name and version of the instrument's controller software.

**Application**

Gives the name and version of the application currently being run on the computer.

**Options**

Displays the options available for the AutoChem HP and whether they are installed or not.

For the Temperature Controller: M = Micromeritics,  
E = Eurotherm

**IP Address**

Displays the IP address of the computer.

**Serial #**

Displays the instrument's serial number, as it was entered during installation.

## Loop Volume

Allows you to enter the volume of the gas injection loop that is attached to the instrument. The loop volume at 0 °C should be entered.

Three standard-volume loops are provided with your instrument. The loop volume is determined by performing a loop calibration. Loop calibration is performed by a specialized experiment step (refer to Appendix J, page [J-38](#)).

## Gas Selections

Select this option to specify which gases are connected to the ports for the preparation, carrier, and loop gases.

Preparation Gas		Carrier/Reference Gas		Loop Gas	
	MFC Constant		MFC Constant		MFC Constant
1: Argon	1.386	1: Helium	1.376	1: Argon	1.386
2: Helium	1.376	2: None	1.000	2: None	1.000
3: None	1.000	3: None	1.000	3: None	1.000
4: None	1.000	4: None	1.000	4: None	1.000

Enter the MFC gas flow conversion constant (0.010 - 2.000) for the gas.

Close

Run File:      Temperature: 250.0 °C  
Status: Idle      TCD Reading: 0.2000



**Gas Selections cannot be changed during an automatic operation.**

This dialog contains a drop-down list of the available gases for each of the 12 available ports. Each gas also has its MFC constant listed.

Identify what gas is attached to each port by selecting that gas name from the drop-down list. If no gas is attached to a given port, select **None**.

The gases in the drop-down list are those specified in the Gas Defaults table (on the Options menu).

When an analysis is started, the software checks the gases specified in the sample information file against the gases identified in this dialog. If there is a discrepancy between the gas indicated in the sample information file and the gas indicated in the Gas Selections dialog, a message is displayed; analysis cannot begin until the discrepancy is resolved.



The software is unable to determine if the wrong gas is physically connected to the port. It can only check that the gas selected agrees with the gas identified for each port in the Gas Selections dialog. It is important to be sure that the Gas Selections dialog accurately reflects which gas is physically connected to each port.

## Gas Flow Constant Calibration



The Mass Flow Controllers (MFCs) of your AutoChem 2950 HP were calibrated before shipping. Unless you suspect that one of the MFCs is operating improperly, you should use the calculated (factory) values.

Select this option to determine a constant that is used by the analyzer to ensure accurate gas flows through each Mass Flow Controller.

The screenshot shows a dialog box titled "Gas Flow Constant Calibration (Unit 1 - S/N: 25...)". It contains four numbered instructions: 1. Use the Instrument Schematic (in Manual Mode) to flow the gas to be calibrated through the MFC to be used for the calibration. 2. Connect a flow meter to the exhaust and measure the actual flow rate. 3. Enter the measured flow below and select the ACCEPT button to compute the conversion constant. 4. Update the Gas Selections: enter the new constant associated with the gas on the MFC used in step 1. Below the instructions, there is a section titled "Mass Flow Controller used for Calibration" with three radio buttons: "Preparation" (selected), "Loop", and "Carrier". At the bottom, there is a "Measured flow" field with the value "10.00" and the unit "cm³", an "Accept" button, a "Conversion constant" field, and a "Close" button.

A table of the constants for the most commonly used gases is provided in Appendix C. A set of conversion constants is shipped with the analyzer, and is displayed in the Gas Defaults table accessed from the Options menu. In most cases, the defaults provided in the table work well.

If you are using a gas that is not listed in the Gas Defaults table, or if you suspect that one of the MFCs is not functioning properly, calculate a new constant using the instructions and the worksheet provided in Appendix F.



## TCD Calibration

---

Analyses with the AutoChem 2950 HP yield data on signal reading, peak area, temperature, and time (plus auxiliary signals, if they were recorded). These data are sufficient for many applications, but in some cases, volume data are needed as well.



**In applications where volume data are not needed, it is not necessary to perform a calibration. If volume data are needed, calibration may be performed either before or after the analysis.**

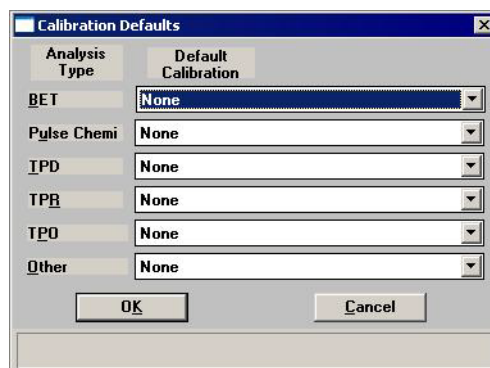
A group of automatic calibration routines is provided in the form of specialized experiment steps. A calibration run is an analysis using one of these experiment steps. The calibration run can be performed before or after the sample analysis. A calibration experiment can be included as a step within your sample analysis, or it can be performed as a separate analysis.

After the sample data and calibration data are collected, the calibration file is associated with the sample file, and the sample data are converted to volume. A single calibration run can be associated with an unlimited number of sample data files. For example, TPR yields peak area and the temperature(s) at which maximum reduction occurs. To obtain the volume of gas uptake, however, you must select a calibration file to associate with the analysis file. Then you can create reports in which the area data are converted to volume data.

You can associate a calibration file with a sample file in three different ways:

- Select **Unit > TCD Calibration > Defaults** and specify a default calibration file for each experiment type.
- Use the default file specified or choose a different file from the second view of the Start Analysis dialog.
- Click **Set Calibration** on the Peak Editor dialog (described in Chapter 7). This choice allows you to select a calibration file that was created after the sample file was used in an analysis.

## Defaults



Allows you to specify default calibration files for each type of analysis. An additional default can be specified for analyses you have customized, using the Other drop-down list. Setting default calibrations can save you time when you frequently perform analyses using the same gases.

The calibration files that appear in the lists are those that are available for the instrument.

You will have two opportunities to select a different calibration file before you print a report: during the series of dialogs used to start an analysis (**Start Analysis** from the Unit menu), and using **Set Calibration** on the Peak Editor dialog.

## New

Allows you to create a new TCD calibration file; the New TCD Calibration dialog is displayed. The TCD Calibration dialog is displayed enabling you to create calibration files. Create a file for each gas concentration in the calibration. For example, if you have gas concentrations of 10%, 30%, and 20%, you should mark your calibration peaks and create calibration files for all three concentrations; a total of three files.



**Calibrations are specific to each gas bottle. If a gas supply is changed, you should update the calibration.**

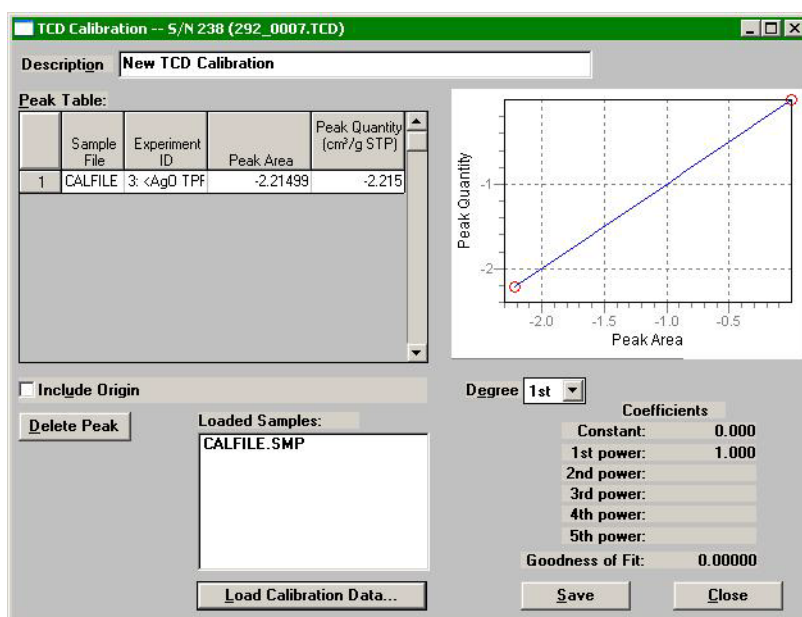
**Description**

Enter an intuitive description in the **Description** field. It is important that you use a description that you will easily recognize.

**Load Calibration Data**

Enables you to select a calibration file. The Load Calibration Sample dialog is displayed.

Select the file to use, then click **OK**.

**Loaded Samples**

Lists the calibration files that are currently loaded.

**Peak Table**

The peak table in the selected file is displayed. You can edit the table or use the **Delete Peak** button.

The values in the table are graphically depicted in a plot adjacent to the table.

**Include Origin**

Check to include the origin (Constant 0.00000) of the data.

**Degree**

Allows you to choose a different degree of polynomial; usually the 2nd degree is adequate.

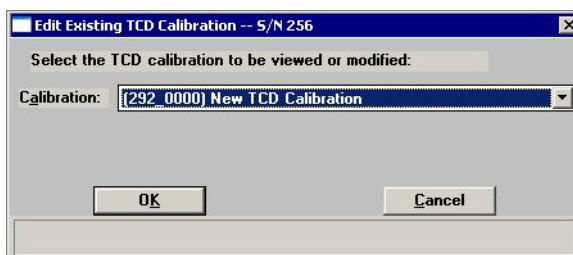
*Range: 1st through 5th*

**Coefficients**

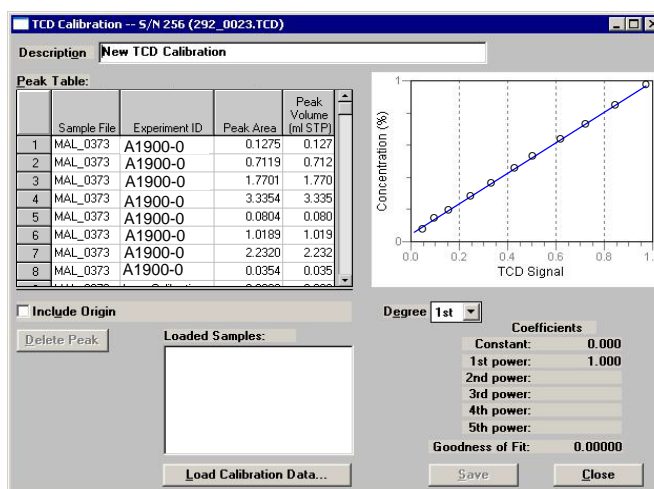
Shows the fit of data to the polynomial.

## Open

Allows you to open an existing calibration file for editing; the Edit Existing TCD Calibration dialog is displayed.



This dialog contains a drop-down list of existing TCD calibration files. After choosing the file, click **OK**; the TCD Calibration dialog is displayed.



### Description

Displays the name of the current calibration file. This field can also be used to enter a new name when changes have been made.

### Peak Table

This table displays the sample file number, the experiment identification, the peak area and peak volume.

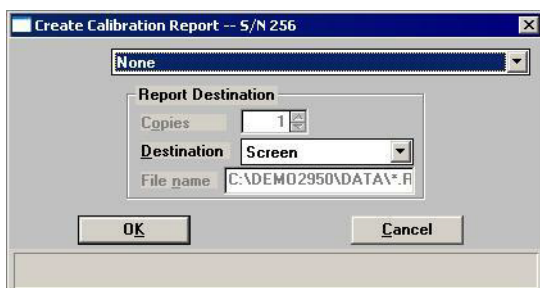
The values in the table are graphically depicted in a plot adjacent to the table.

For a description of the remaining fields on this dialog refer to **“New” on page 6-24**.

## Report

---

Select this option to print a report of a calibration file.

**Calibration**

From the drop-down list, select the file for the report.

**Report Destination  
Copies**

Enabled if you choose Printer or Printer as the report destination. You can print up to four copies.

**Destination**

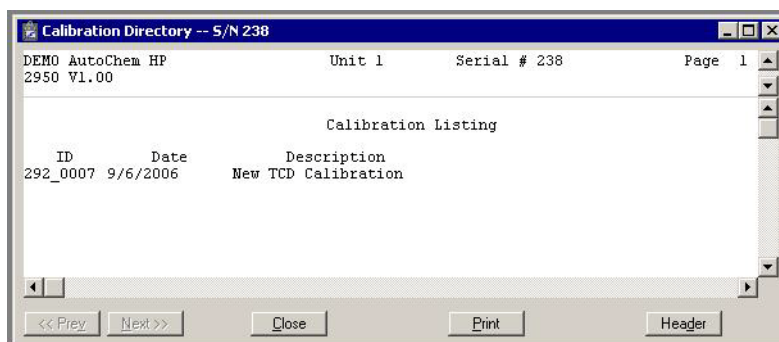
Allows you to choose a destination for the report.

**File name**

Enabled if **File** is selected as the report destination, allowing you to enter a file name (or accept the default).

## List

Select this option to display a list of the files in the Calibration Directory, including details such as the calibration file's identifier (as entered in the sample information file) and the date the file was created (or last edited).

**Next  
Prev**

Allow you to view the next or previous page of the list (if they exist).

**Close**

Closes the window.

**Print**

Prints the list.

**Header**

This toggle button hides/shows the header of this list. If the header is selected, it appears on every page of the list when it is displayed and/or printed.

## Instrument Calibration

A calibration of the analyzer is performed before the analyzer is shipped; the correct calibration values for each instrument are shipped with the analyzer. The contents of this calibration file are loaded into the analyzer software during installation.

This option displays the Instrument Calibration dialog.

	Detector Heat Zone	Cold Trap Heat Zone	Analyze Heat Zone	Loop Heat Zone	Back Press. Heat Zone
Reading	103	103	103	103	103
Low Cal.	20	0	0	0	-15
High Cal.	200	200	200	200	15
Calibrated	1/12/2006				
Raw Value	12345	12345	12345	12345	12345

	Sample Temperature *	Sample Pressure *	Auxiliary Input1	Auxiliary Input2	Furnace Temperature *
Reading	251	760	1.23	4.56	251
Low Cal.	0	-8274	14.7	-15.0	0
High Cal.	200	51715	1000.0	15.0	1100
Calibrated					
Raw Value	12345	123	1234	1234	12345

\* Be sure not to exceed recommended temperatures and pressures for the type of sample tube in use.

Run File:   
 Status: Idle   
 Temperature: 250.0 °C   
 TCD Reading: 0.2000

This dialog shows calibration data for the AutoChem 2950 HP components. It also enables you to calibrate the sample thermocouple. Recalibration should be performed only when you have replaced the sample thermocouple or when the analyzer's calibration file (which was installed during initial installation of the software) has been corrupted or lost. Refer [“Recalibrating the Sample Thermocouple” on page 9-24](#) for instructions on using this dialog to calibrate the sample thermocouple.

You should not attempt to calibrate other components without contacting your Micromeritics service representative.



**Do not attempt to use the Instrument Calibration dialog to calibrate the analyzer unless you are specifically instructed to do so by a Micromeritics service representative.**

### Load from File

Allows you to load a different calibration file.

### Save to File

Allows you to save the current calibration settings to a file. This function is useful in protecting your calibration data. It is wise to save the current settings before attempting a recalibration. Be sure to use the next sequenced file name generated by the system.



<b>Accept</b>	Replaces the existing calibration values with those you have entered in this session. After a confirmation, the software gives you the opportunity to save the existing values. The Instrument Calibration dialog is closed after you accept the new values.
<b>Cancel</b>	Cancels any changes made to the current calibration settings. A warning message is displayed before the dialog closes.
<b>Reset</b>	Enabled when a change has been made to any field on this dialog. Click to reset any changed value(s) to its last saved state.



**Do not edit parameters of the following options unless instructed to do so by a Micromeritics service representative.**

<b>Furnace PID</b>	Displays the control parameters for the temperature controller. The default values provide linear temperature ramps from ambient to 1100 °C.
<b>Sample PID</b>	Displays the control parameters for the sample temperature. The proportional and derivative are used. The default values are recommended.
<b>MFC Full Scale</b>	Displays the full scale flow rate for the mass flow controllers. The full scale was determined using H <sub>2</sub> as the calibration gas.



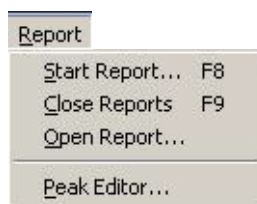
## 7. REPORT MENU

The Report menu contains options which allow you to start, open, and close reports. This menu also allows you to access the Peak Editor, a flexible and powerful tool for processing results.

This chapter describes the options contained on the Report menu, as well as a few examples of generated reports.

### Description

---

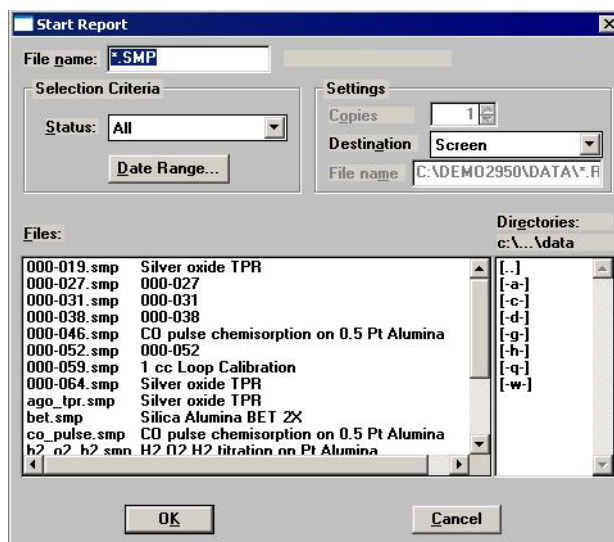


Listed below are brief descriptions of the options contained on the Report menu. Detailed descriptions follow this section.

<b>Start Report</b>	Allows you to generate a report on a sample which has been analyzed or on one which is being analyzed. Page <a href="#">7-2</a> .
<b>Close Reports</b>	Allows you to close all open report windows. Page <a href="#">7-4</a> .
<b>Open Report</b>	Allows you to open a saved report. Page <a href="#">7-4</a>
<b>Peak Editor</b>	Allows you to view peak data, combine and overlay results from multiple analyses, change the calibration files selected, and edit the data. Page <a href="#">7-16</a> .

## Start Report

Select this option to generate a report on a sample analysis. The Start Report dialog is displayed.



### File name

The name you select from the Files list box is copied to this field. If you select multiple files, the last one selected is displayed. If you have a file open, its name is displayed.

### Status

This drop-down list determines the type of sample files displayed in the Files list box.

**All** displays all of the sample files in the specified directory, and within the specified range of dates, that contain a status of Analyzing or Complete.

**Analyzing** displays sample files that are currently being used in an analysis.

**Complete** displays sample files that have been used in an analysis (within the specified range of dates).

### Date Range

Click this button to limit the list to those sample files created within a specified time period.

### Copies

Enabled when the **Printer** destination is chosen, allowing you to print up to four copies of the selected report(s).

**Destination**

Displays a drop-down list of output destinations.

If you select **Printer**, requested reports are sent to the selected printer.

If you select **Screen**, many options are available for manipulating and customizing reports. Refer to **Onscreen Reports** on page 7-6 for manipulation methods of onscreen reports.

If you select **File**, the tabular reports of the requested file are converted to a text file which can be viewed with a text editor or other text file manipulation tools. Graphical reports cannot be generated to a file.

**File name**

Enabled when you select **File** as the destination. Allows you to enter a name, or you may accept the default.

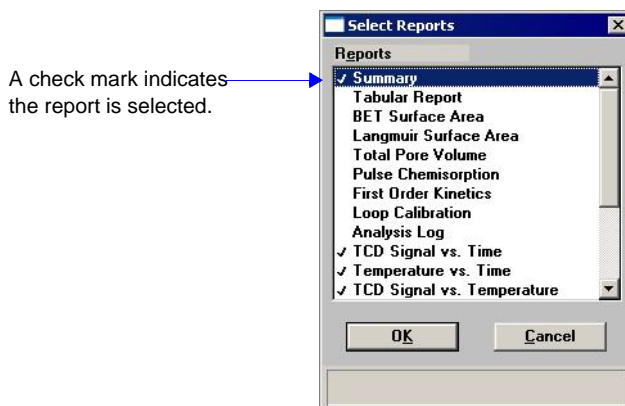
**Files list box**

Displays a list of the available sample files for the choice shown in the **Status** field.

**Directories**

Displays a list of available drives and directories. The drive and directory last accessed is displayed immediately above the **Directories** list box.

If a single sample file is selected, the Select Reports dialog is displayed. The reports selected are the ones specified in the sample file. Deselect reports or select additional ones. If more than one file is selected, this dialog is not displayed; the reports selected in each file are generated.



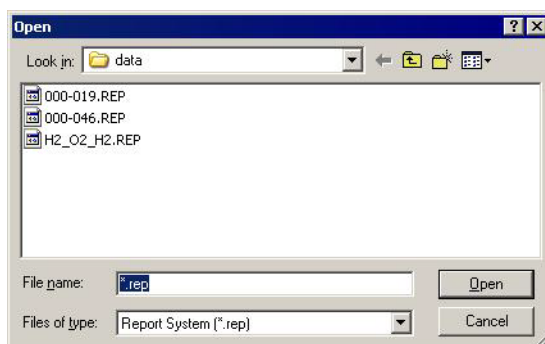
Double-click on the report(s) to select (or deselect). A check mark is placed before each selected report. Pressing the **Spacebar** (when the report is highlighted) performs the same function. After you make your report selection and click **OK**, the requested reports are sent to the specified destination.

## Close Reports

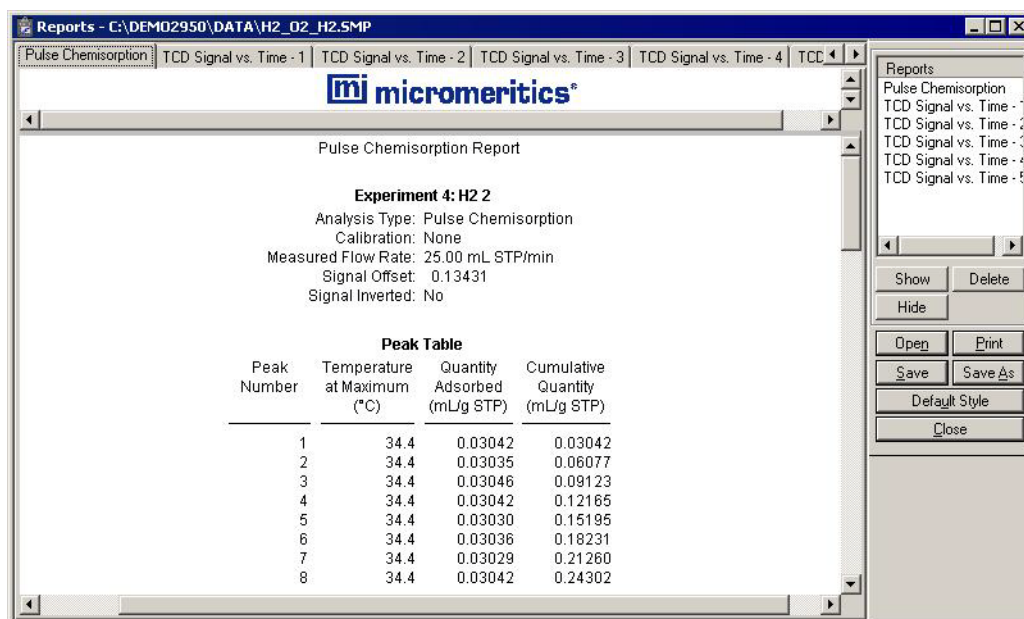
This option enables you to close all open report windows at one time. This avoids having to select **Close** on each report window. This option is unavailable if reports are being generated.

## Open Report

Select this option to open a saved report. The Open dialog is displayed.




Select the report, then click **Open**. The report is displayed on the screen.



Refer to **“Onscreen Reports” on page 7-6** for an explanation of the commands available for onscreen reports.

## Printed Reports

### Header

			
Micromeritics Instrument Corporation -- AutoChem HP 2950			
DEMO AutoChem HP 2950	Unit: 1	Serial # 256	Page 1
Sample: 1 cc Loop Calibration			
Operator:			
Submitter:			
File: C:\DEMO2950\DATA\000-059.SMP			
Started: 1/5/2006 10:51:06AM		Sample Mass: 1.0000 g	
Completed: 1/5/2006 10:51:17AM		Report Time: 1/11/2006 8:41:46AM	
Comments: Loop calibration using 0.8 ml user injections and 1 ml loop.			

All printed reports (either to the screen or to a printer) contain a header displaying file statistics.

- Tabular and graphical reports contain sample and instrument statistics such as date and time of analysis, analysis conditions, and so forth.

The headers for these reports also contain notes of any changes to the sample file which occur after analysis.

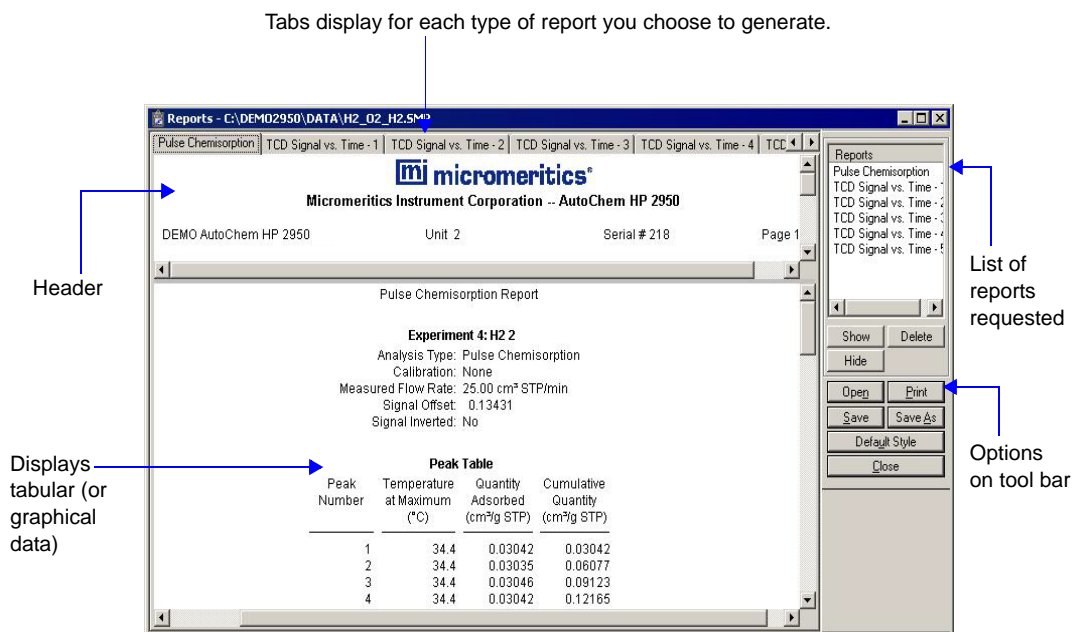
- Summary report headers contain the same type of information displayed in tabular and graphical reports with the exception of notes.

## Onscreen Reports

The report window containing onscreen reports provides many options for customizing and manipulating reports:

- a tool bar
- shortcut menu options
- zoom feature
- axis cross hairs

When reports are printed to the screen, they are printed in a window like the one shown below. Each requested report is listed in the Reports window on the tool bar; reports are also indicated by selectable tabs across the top of the report header. To view a specific report, select its tab or select the report in the Reports window and click **Show**.





## Tool Bar

---

**Reports**

Contains a list of all requested reports.

**Show**

Shows the selected report in the report window. If the report has been hidden, it and its associated tab will become visible.

**Delete**

Deletes the selected report. A deletion confirmation dialog is displayed since this function cannot be undone. The deleted report(s) will have to be regenerated if deleted in error.

**Hide**

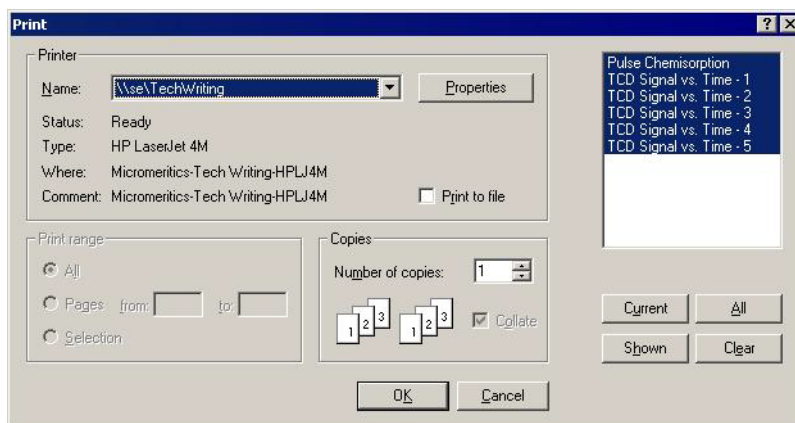
Hides (removes) the selected report from the report window. The report's associated tab is also removed.

**Open**

Allows you to open a previously saved report file.

**Print**

Displays a print dialog so that you can choose an appropriate printer for report output. A list of available reports is displayed in the window on the right side of the dialog.



For convenience in selecting which reports to print, buttons are provided beneath the report window. Or, you can make your selection by clicking on the reports.

**Current** selects the report displayed in the report window.

**Shown** selects only the shown reports; any non-highlighted reports indicate they are hidden. You can still select hidden reports from this window to print.

**All** selects all reports, including those that may have been hidden.

**Clear** clears all selections.

## Save

Saves all reports of the currently open file in a report format using the same name as the sample file, only with an rep extension. To specify a name and/or specific reports to save, use the **Save As** button.

## Save As

Allows you to save all or specified reports from the currently open file. The buttons displayed on this dialog perform in the same manner as the print dialog (explained above).

Reports can be saved in three different formats:

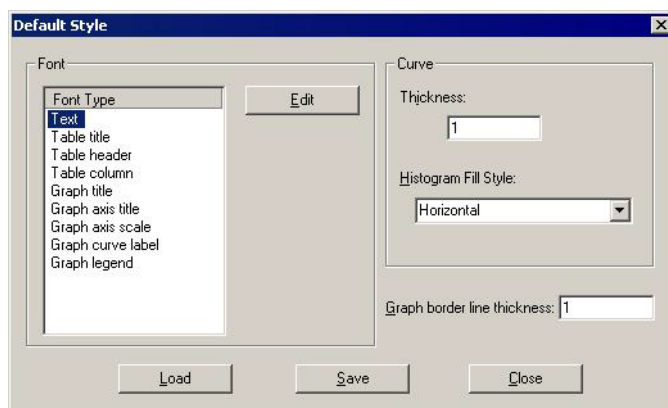
Report system (\*.rep): Saved in a format which allows you to reopen the file using the **Open** command on the Reports menu or the Report window tool bar.

Spreadsheet (\*.xls): Saved in a format which can be imported into most spreadsheet programs.

Ascii Text (\*.txt): Saved in ASCII text which can be imported into programs accepting this type of file.

## Default Style

Displays the Default Style dialog so that you can edit report fonts, curves, and borders.



### Font

The window on the left lists the items for which the font can be edited.

Click **Edit** to edit the font of a selected item; a font dialog is displayed so that you can specify the font and attributes.

**Default Style**  
(continued)**Curves**

Enter the thickness of curves displayed in graphs.

Select the histogram fill style.

**Graph Border**

Enter the thickness of the graph border.

Click **Save** to save the changes as the default. If you do not click **Save**, the changes will apply to the current report set only. The next reports will revert to the default font.

Click **Load** to load the default font of a selected item.

Click **Close** to close the dialog.

**Close**

Closes the Report window.

If you have multiple reports open, use the **Close Reports** option on the **Report** menu. This option allows you to close all open Report windows at one time, without having to click **Close** on each Report window.

## Shortcut Menus

---

Shortcut menus are accessed when you right-click on the tabular or graphical portion of a report.

### For Tabular Reports

---

**Resize column**

Displays a dialog so that you can specify the width of the selected column (in inches).

**Rename column**

Displays a dialog so that you can edit the name of the selected column. Use **Ctrl + Enter** to insert line feeds.

**Move column**

Allows you to move the location of the selected column to the left or to the right.

**Align column**

Enables you to right-align, left-align, or center the data in the selected column.

**Hide column**

Displays a list of all columns that can be hidden.

**Show column**

Displays a list of all hidden columns.

**Column font**

Displays a Font dialog, allowing you to change font attributes for the tabular data in the current report.

**Header font**

Displays a Font dialog, allowing you to change font attributes for column headers in the current report.

**Edit title**

Allows you to edit the table title and font.

**Copy table as text**

Enables you to copy the entire table (column headers and data) and then insert it into another program. Columns are tab-delimited, allowing easy alignment.

Use this option to copy data easily and quickly from the AutoChem program to other applications such as spreadsheet programs.

**For Graphs**

---

**Autoscale**

Sets the axis boundaries to autoscale, presenting the signal in full scale.

**Redraw**

Sets axis boundaries to its original view, allowing easier editing of peaks.



**In many cases, the *Autoscale* and *Redraw* options will show very little difference. Both options are also used to return the view to its original presentation after zooming a report or to remove cross-hairs.**

**Show curve**

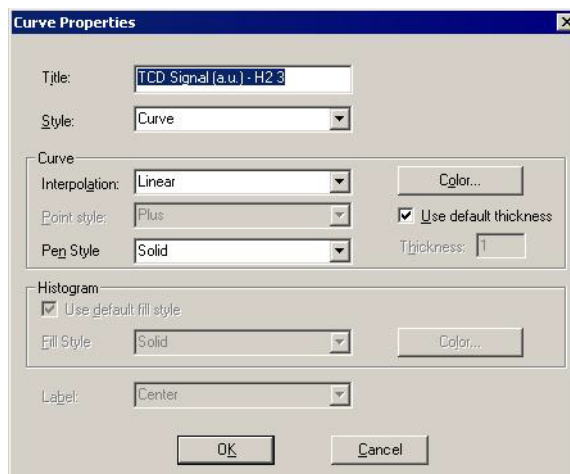
Allows you to show curves that have been hidden. This option is disabled if no curves have been hidden.

**Hide curve**

Allows you to hide unwanted curves.

**Edit curve**

Displays the Curve Properties dialog, allowing you to edit curve properties.

**Title**

Displays the title of the curve you are editing.

**Style**

Drop-down list containing styles in which collected data can be displayed.

**Curve group box**

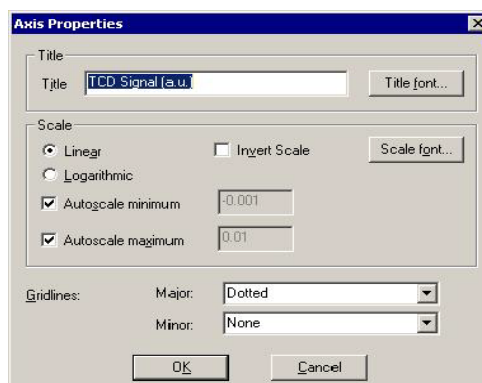
Contains options for curves and points. You can edit the curve interpolation, the style of curve and/or points, as well as the pen color. The options in this group box are disabled if Histogram is chosen in the **Style** drop-down list.

**Histogram group box**

Allows you to specify the type of fill as well as the color if Histogram is chosen as the style for collected data.

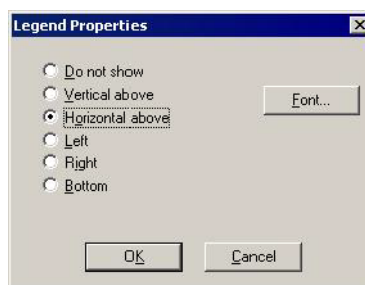
**Edit axis**

Displays the Axis Properties dialog allowing you to edit axis properties.

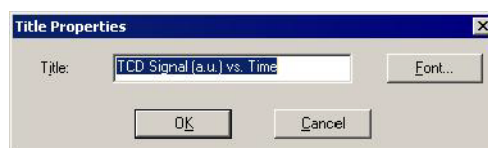


**Edit legend**

Displays the Legend Properties dialog, allowing you to edit the placement of the legend and the font.

**Edit title**

Displays the Title Properties dialog, allowing you to edit the current graph's title and font.

**Copy as metafile**

Copies the graph and places it on the clipboard, allowing you to paste it into other applications accepting Windows metafiles.

Use this option to capture graphics for use in reports and presentations.

**Copy as text**

Copies the data used to generate the graph as a series of tab-delimited columns of text.

---

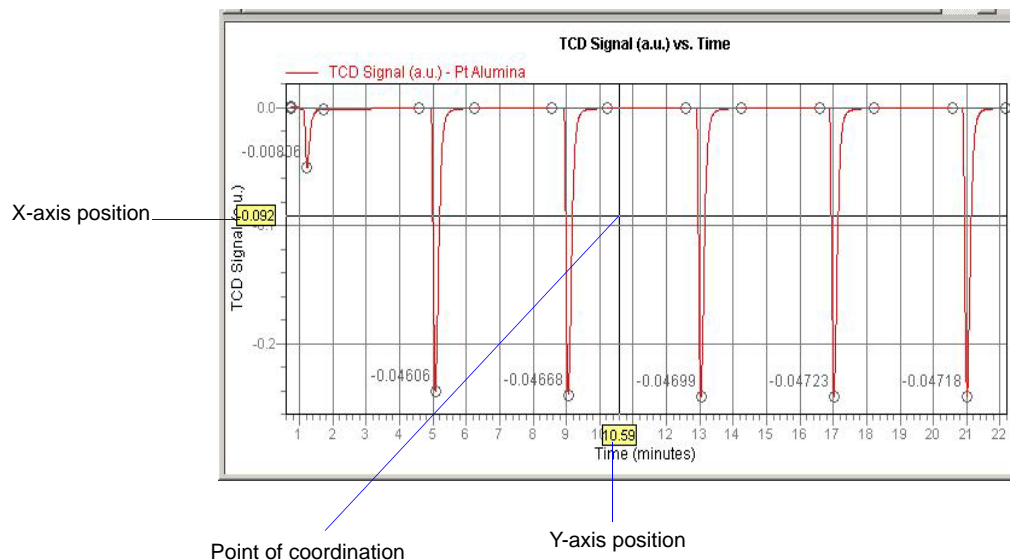
**Zoom Feature**

A zoom feature is included with the report system so that you can zoom in to examine fine detail of the distribution. To use this feature, simply hold down the left mouse button and drag the mouse cursor (drawing a box) across the area to view; then release the button. The enlarged area immediately fills the graph area. You can return to the normal view by right-clicking on the graph and selecting Autoscale.



## Axis Cross-Hair

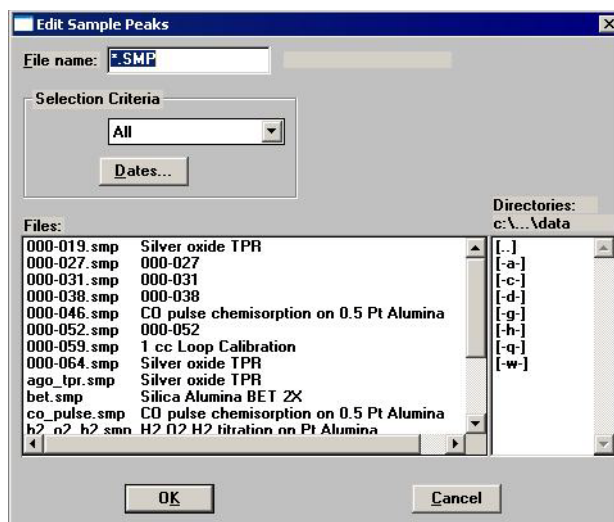
A cross-hair function is available so that you can view axis coordinates. To use this feature, simply left-click in the area of the graph.



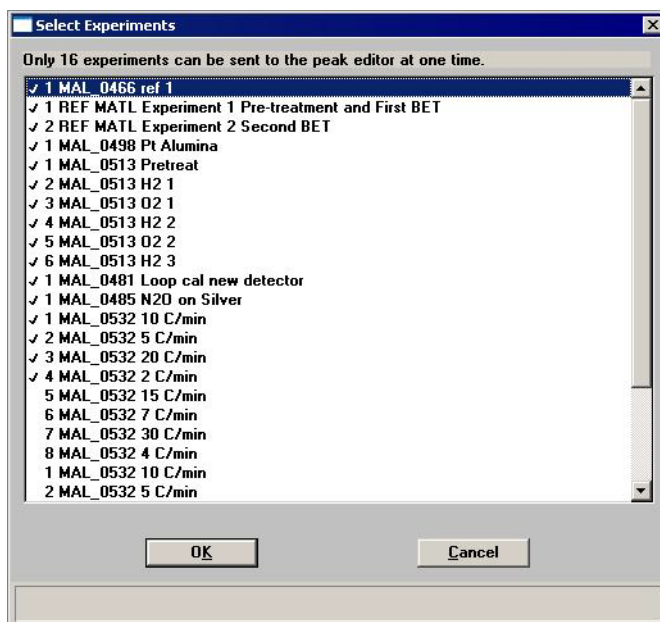
You can remove cross-hair lines and return to the normal view by right-clicking on the graph and selecting **Autoscale** or **Redraw**. You can also click out of the graph area to remove cross-hair lines.

## Peak Editor

The Peak Editor allows you to view and edit data from sample files. You can view the peaks in a stacked or overlaid manner. You can also edit the peaks and delete undesirable ones.

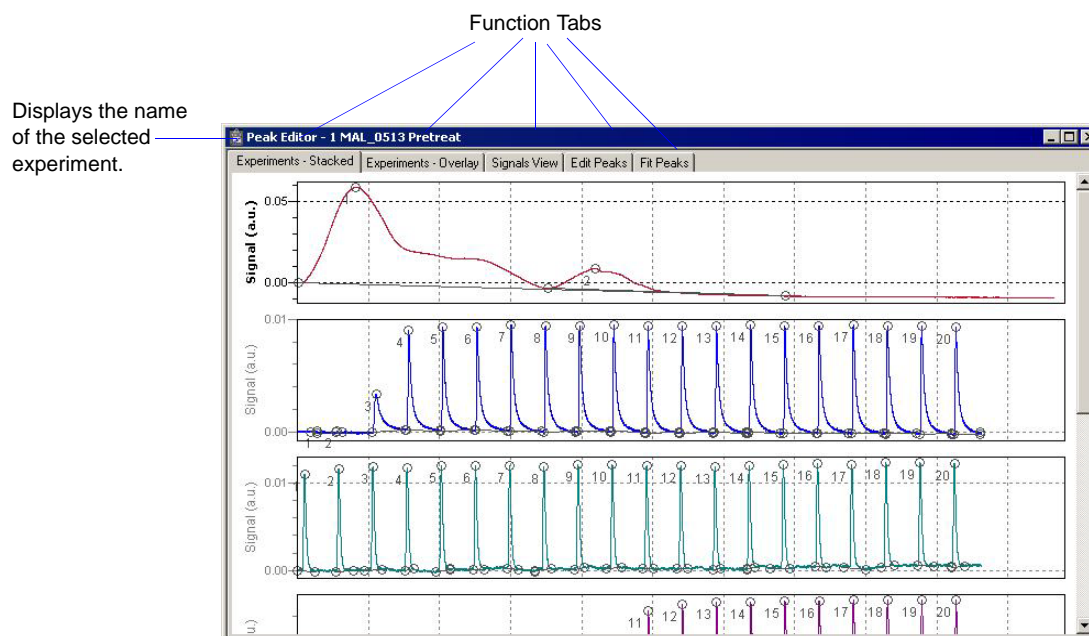


The fields on this dialog function in the same manner as other dialogs allowing you to select files. The Peak Editor allows you to view up to 16 experiments at one time. If you choose files containing more than 16 experiments, a dialog displaying the experiments available in the chosen files is displayed.



Choose the 16 experiments to view and defaults with the first 16 experiments selected by double-clicking on the file, or by highlighting the file and pressing the **Space Bar**.

Click **OK**, the Peak Editor dialog is displayed.



This window contains five tabs. the first three tabs — **Experiments Stacked**, **Experiments Overlay**, and **Signals View** — provide different views of the data; the last two tabs — **Edit Peaks** and **Fit Peaks** — provide the TCD vs. time (or temperature) and tabular information for the integrated peaks.

## Features of the Peak Editor Window

---

The Peak Editor window provides the following tools for viewing and editing peaks:

- Shortcut menus
- Shortcut keys
- Zoom in on the TCD signal
- Cross-hairs

### Shortcut Menu

---

A shortcut menu is available for each view of the Peak Editor window. This shortcut menu contains the available functions and is accessed by right-clicking in the graph area. For example, when you right-click in the Experiments view, the following menu is displayed.



In many cases, the *Autoscale* and *Redraw* options will show very little difference. Both options are also used to return the view to its original presentation after zooming a report or to remove cross-hairs.

The options on the menu displayed for each view are explained in subsequent sections.

### Shortcut Keys

---

Shortcut keys are provided for many of the functions in the Peak Editor. These shortcut keys have been assigned function-relevant characters enabling you to remember them more quickly. For example, **Ctrl+A** is assigned to Autoscale. Refer to the table on page 7-19 for a complete list of the shortcut keys you can use while operating the Peak Editor.

*Shortcut Keys for the Peak Editor*

Key(s)	Function
<b>Ctrl + A</b>	Sets the axes to autoscale (full scale view)
<b>Ctrl + D</b>	Returns the Peak Editor window to its original view
<b>Ctrl + M</b>	Shows or Hides Peak Marks
<b>Ctrl + T</b>	Displays the X-axis by Time or Temperature
<b>Ctrl + F</b>	Copies graph(s) as a Metafile, allowing you to paste it into another application
<b>Ctrl + C</b>	Copies data to the clipboard, allowing you to paste it into another application
<b>Ctrl + N</b>	Advances you to the next peak
<b>Ctrl + P</b>	Returns you to the previous peak
<b>Ctrl + L</b>	Marks the left edge of the peak
<b>Ctrl + R</b>	Marks the right edge of the peak
<b>Ctrl + S</b>	Saves the changes made to the current file

**Zoom Feature**

A zoom feature is included with the Peak Editor allowing you to zoom in on peaks. This feature functions in the same manner as the zoom for reports; hold down the left mouse button and drag the mouse cursor (drawing a box) across the area to view; then release the button. If you have multiple graphs displayed, the area you define on the selected graph is enlarged for each graph. You can return to the normal view by right-clicking on the graph and selecting Autoscale, or you can use the shortcut keys **Ctrl + D**.

**Cross-Hairs**

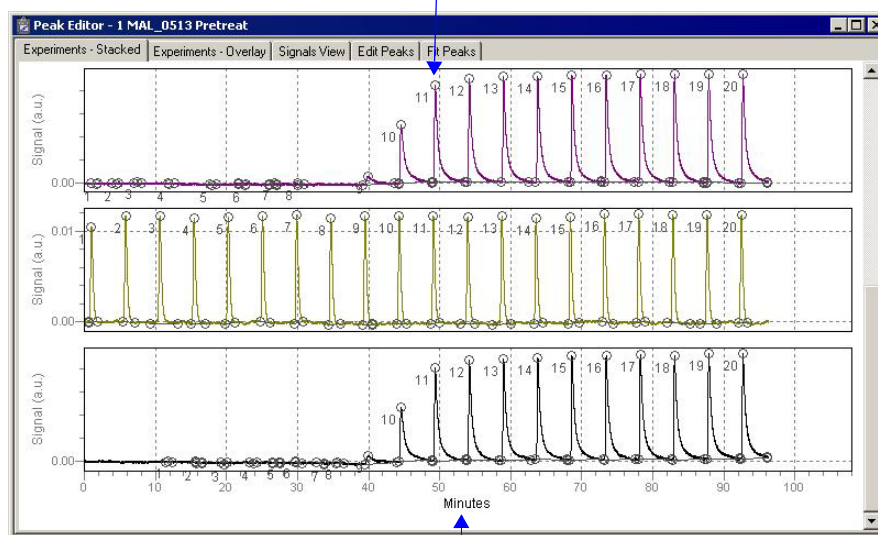
When you click the mouse in the graph area, locator cross-hairs are displayed. The exact values associated with the intersection of the cross-hairs are displayed along the axes. This makes it easy to determine exact values for points on the second Y-axis, or for the point on the time/temperature trace that corresponds with the signal/temperature trace. Refer to **Axis Cross-Hair** on page 7-15 for additional information on cross-hairs.

## Experiments

The Experiments View shows curves for all experiments within the selected files. You can display the curves in a stacked fashion or in an overlaid fashion. You can display up to 16 experiments; each experiment is displayed in a different color.

### Stacked

Peak marks can be shown or hidden.



X-axis can be displayed as Time or Temperature.

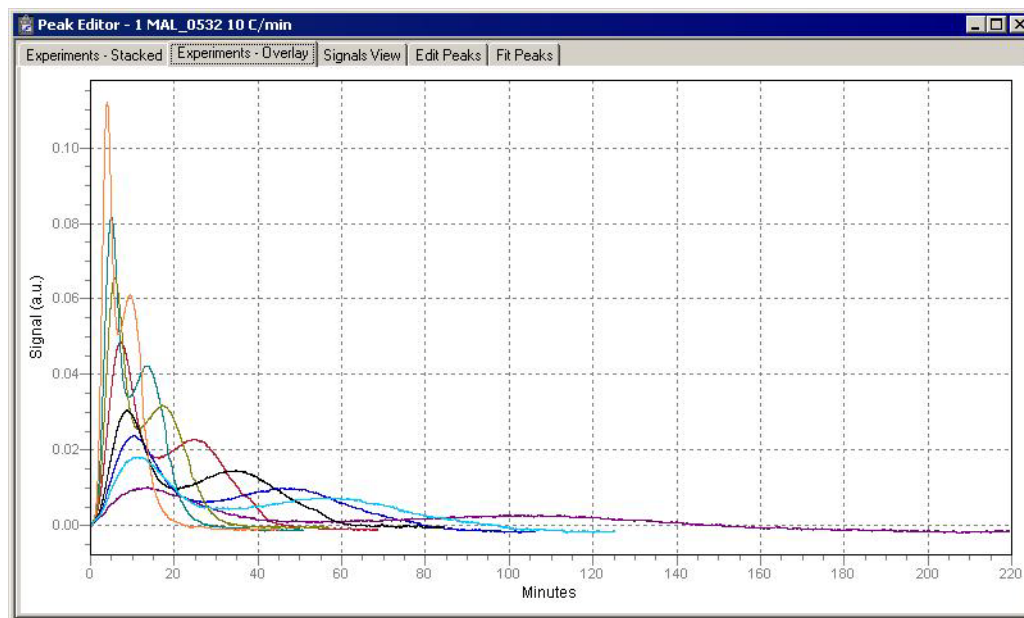
The Stacked view displays up to 16 experiments, stacked one after the other. In this view, you can select the individual experiments to obtain information. When you select an experiment, its name is displayed in the title bar of the window. Cross-hairs can be displayed to show exact values in the selected area.

When you right-click in the graph area, the following shortcut menu is displayed:



<b>Autoscale</b>	Sets the axis boundaries to autoscale, presenting the signal in full scale.
<b>Redraw</b>	Sets axis boundaries to its original view, allowing easier editing of peaks.
<b>View</b>	<p>Displays a cascading menu allowing you to:</p> <ul style="list-style-type: none"><li>• Show or hide the peak table</li><li>• Show or hide the peak marks</li><li>• Specify X-axis in Time or Temperature</li><li>• Copy the contents of the window to the clipboard as a metafile or as text, which you can paste into another application</li></ul>
<b>Insert</b>	Enables you to insert experiments from another sample file.
<b>Remove</b>	Removes the selected experiment from the Peak editor window; the data are not deleted from the sample file.
<b>Save As</b>	Allows you to save the experiments in the window as a new sample file. For example, if you have a file opened containing five experiments but are only interested in two of them, you can remove the three of no interest and save the file with the remaining two.
<b>Close</b>	Closes the Peak Editor window.

## Overlay



The Overlay view displays up to 16 experiments, overlaid on each other. This view does not allow you to select an experiment; you must switch to the Stacked view to select experiments.

When you right-click in the graph area, the following shortcut menu is displayed:

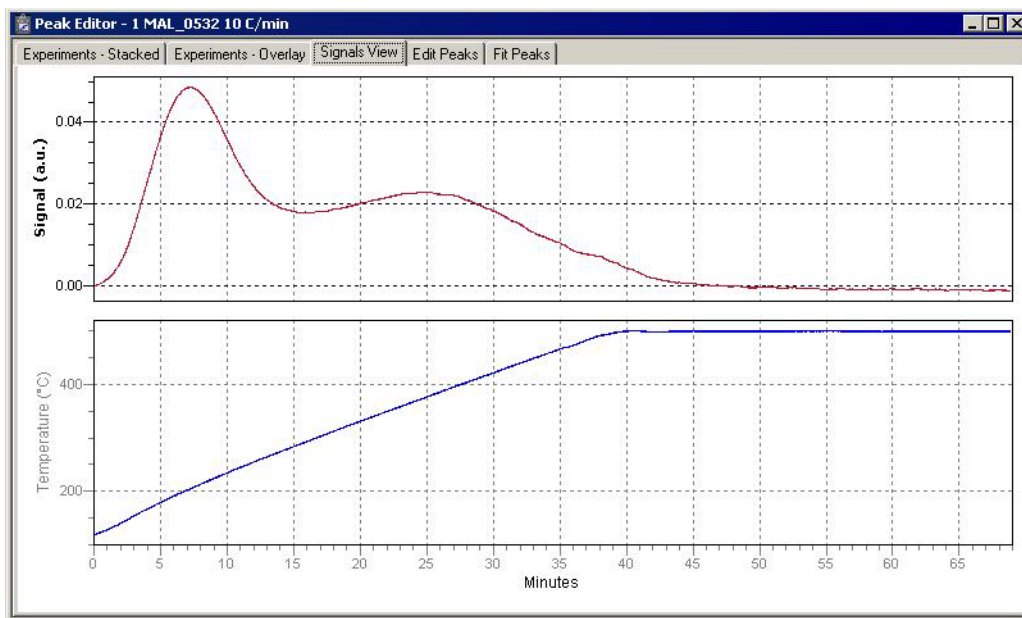


The options on this menu are identical to the ones available in the Stacked view (page 7-20). **Remove** is disabled on this menu because individual experiments cannot be selected in the Overlay view.



## Signals View

The Signals view displays the signals for the selected experiment stacked one after the other. This allows you to view the relationship of peaks to temperature and auxiliary signal conditions that occurred during data collection.



When you right-click in the graph area of the Signals view, the following shortcut menu is displayed:

Autoscale	Ctrl+A
Redraw	Ctrl+D
✓ X Axis by Time	Ctrl+T
X Axis by Temperature	Ctrl+T
Copy as Metafile	Ctrl+F
Copy as Text	Ctrl+C
Close	Ctrl+F4

### Autoscale

Sets the axis boundaries to autoscale, presenting the signal in full scale.

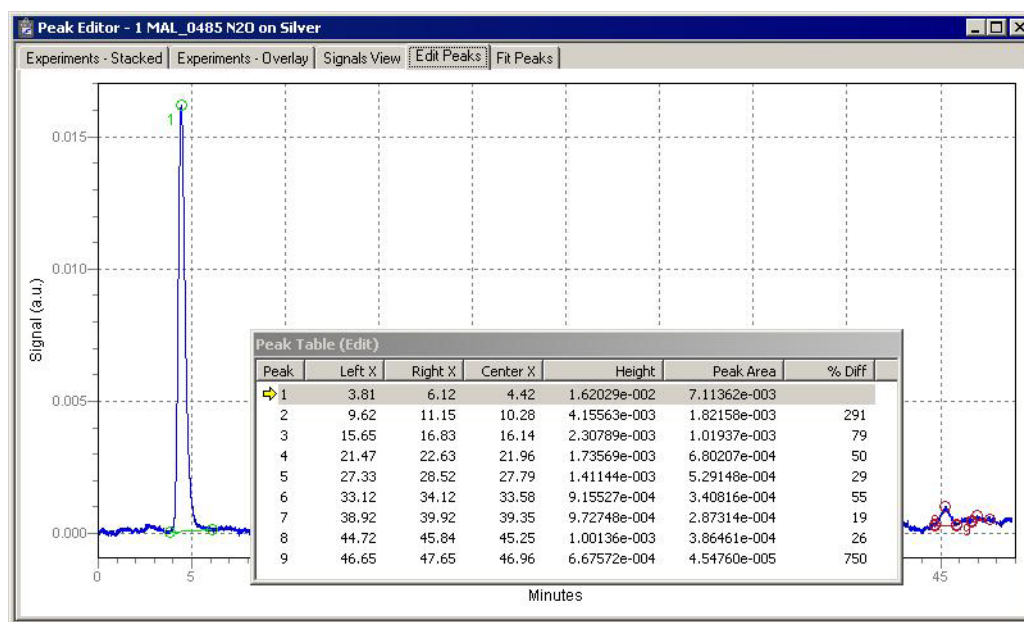
### Redraw

Sets axis boundaries to its original view, allowing easier editing of peaks.

<b>X-Axis by Time</b>	Enables you to display the X-axis by Time or Temperature.
<b>X-Axis by Temperature</b>	
<b>Copy as Metafile</b>	Enables you to copy the graph area to the clipboard as a metafile or as text so that it can be pasted it into another application.
<b>Copy as Text</b>	
<b>Close</b>	Closes the Peak Editor window.

## Edit Peaks

Edit Peaks allows you to edit the peaks of the selected experiment; the Peak Table dialog listing peak information for all peaks of the selected experiment is displayed.



Use one of the following methods to select a peak:

- Highlight the peak in the Peak Table
- Access the shortcut menu and select **Next Peak** or **Previous Peak** until you navigate to the peak
- Use **Ctrl + N** (advance to the next peak) or **Ctrl + P** (return to the previous peak) to navigate to the peak

When a peak is selected, its peak marks display in a light green color; the peak marks for unselected peaks display in red.

Peaks are edited by accessing functions from the shortcut menu which is displayed when you right-click in the graph area.

Autoscale	Ctrl+A
Redraw	Ctrl+D
Next Peak	Ctrl+N
Previous Peak	Ctrl+P
Insert New Peak	Ins
Mark Left Edge	Ctrl+L
Mark Right Edge	Ctrl+R
Find All Peaks	
Delete Peak	Del
Delete All Peaks	
View	
Save	Ctrl+S
Set Calibration...	
Peak Integration Options...	
Close	Ctrl+F4

**Autoscale**

Sets the axis boundaries to autoscale, presenting the signal in full scale.

**Redraw**

Sets axis boundaries to its original view, allowing easier editing of peaks.

**Next Peak**

Advances you to the next peak.

**Previous Peak**

Returns you to the previous peak.

**Insert New Peak**

Inserts a new peak in a graph.

Click in the graph where you want to insert a peak, then select **Insert New Peak**.

**Mark Left Edge**

A peak is automatically marked by the program; this option allows you to redefine the left edge. Place the mouse cursor to the left edge marked, right-click, and select this option.

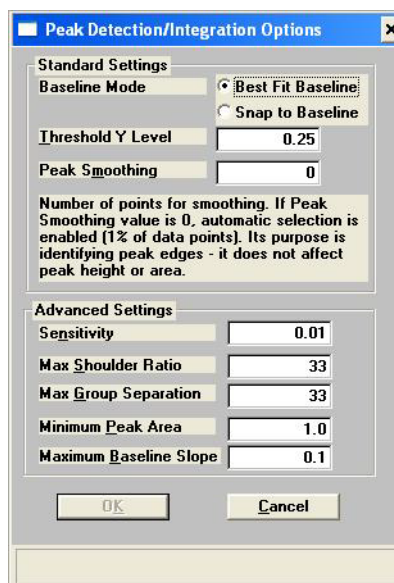
**Mark Right Edge**

A peak is automatically marked by the program; this option allows you to redefine the right edge. Use the same method used for marking the left edge.

<b>Find All Peaks</b>	This option is used to find all peaks automatically in the Peak Editor window. This is a rapid technique for integrating pulse chemisorption data. This option is most effective for narrow peaks with good baseline separation.
<b>Delete Peak</b>	Removes the selected peak from the peak table. Deletes all peak information for a single peak.
<b>Delete All Peaks</b>	Clears the Peak table of all data and removes all markings from the peaks.
<b>View</b>	Displays a cascading menu allowing you to: <ul style="list-style-type: none"><li>• Show or hide the peak table</li><li>• Show or hide the peak marks</li><li>• Specify X-axis in Time or Temperature</li><li>• Enable or disable smoothing, and invert or zero the signal (peak)</li><li>• Copy the contents of the window to the clipboard as a metafile or as text, then paste it into another application</li></ul>
<b>Save</b>	Saves the peak table to the sample file.
<b>Set Calibration</b>	Allows you to choose a TCD calibration that is appropriate for the current experiment.

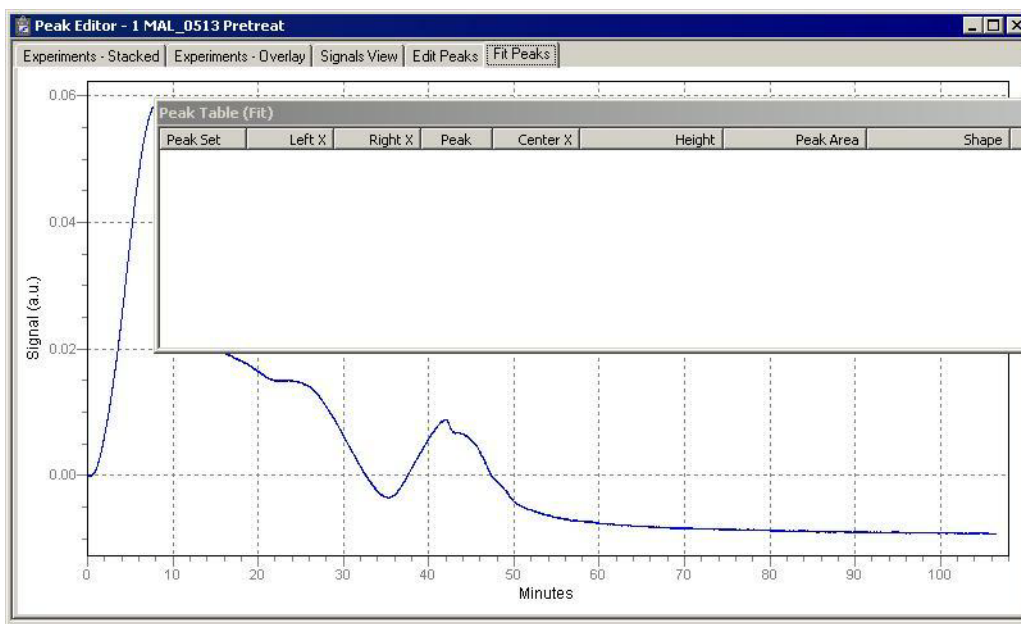
## Peak Integration Options

Displays the Peak Integration Options dialog, which allows you to adjust parameters to optimize the results you obtain using the **Find All Peaks** option. (Refer to Appendix H **PEAK DETECTION / INTEGRATION OPTIONS**.)



## Fit Peaks

**Fit Peaks** allows you to separate overlapping peaks by fitting curves to the data, and save them as a new peak table.



Right-click in the graph area to access the shortcut menu for creating the table.

Autoscale	Ctrl+A
Redraw	Ctrl+D
Insert New Peak Set	
Insert New Peak	Ins
Mark (Peak Set) Left Edge	Ctrl+L
Mark (Peak Set) Right Edge	Ctrl+R
Change Peak Shape	▶
View	▶
Delete Peak	Del
Delete Peak Set	
Delete All Peaks	
Import Standard Peaks	
Optimize Peaks	
Auto Fit	
Display...	
Save	Ctrl+S
Close	Ctrl+F4

### Autoscale

Sets the axis boundaries to autoscale, presenting the signal in full scale.

### Redraw

Sets axis boundaries to its original view, allowing easier editing of peaks.

### Insert New Peak Set

Enables you to define a region of the TCD signal that contains multiple peaks. The peak fitting and optimization are constrained to this region of the TCD signal.

Click in the graph where you want to insert a new peak set, then select **Insert New Peak Set**.

After you add the peak set, you can use the commands on the graph shortcut menu to define the peak set (mark edges, for example).

### Insert New Peak

Inserts a new peak in a graph.

Click in the graph where you want to insert a peak, the select **Insert New Peak**

### Mark (Peak Set) Left Edge

Changes the left edge of the peak set.

### Mark (Peak Set) Right Edge

Changes the right edge of the peak set.

<b>Change Peak Shape</b>	Allows you to choose the type of peak shape.
<b>Log Normal Skewed</b>	This is a 4-parameter, log-normal shape that allows for skewed peaks, and is the default peak.
<b>Gaussian</b>	This is the traditional symmetrical shape.
<b>View</b>	Displays a cascading menu enabling you to copy the contents of the window to the clipboard as a metafile or as text, then paste it into another application.
<b>Delete Peak</b>	Removes the selected peak from the peak set table.
<b>Delete Peak Set</b>	Deletes the selected Peak set from the table.
<b>Delete All Peaks</b>	Removes all peaks and peak-set information from the table.
<b>Import Standard Peaks</b>	Imports the saved peak parameters from the Peak Edit screen and uses them for the initial estimates for the peak set.
<b>Optimize Peaks</b>	Locates the best peak height, width, and skewness after initial peak estimates have been given.
<b>Auto Fit</b>	Locates the best number of peaks and parameters from the Peak Set without user estimates.
<b>Display</b>	Enables you to smooth the signal for display purposes only, and to select or deselect zeroing and signal inversion.
<b>Save</b>	Save the current peak set.
<b>Close</b>	Closes the Peak Editor window.





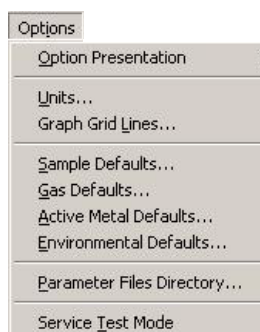
## 8. OPTIONS MENU

The Options menu enables you to configure the system to suit your laboratory's requirements. With the options on this menu, you can:

- Select a default for the format of the sample information file, as well as for its values
- Specify or edit program defaults
- Enter environmental conditions
- Specify graph grid lines
- Specify a directory for predefined parameter files

### Description

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Listed below are brief descriptions of the Options menu contents. Detailed descriptions are found in this chapter.

<b>Option Presentation</b>	Allows you to set the default for the sample information file presentation format. Choose between Basic, Advanced, or Restricted format. Page <a href="#">8-3</a> .
<b>Units</b>	Allows you to select the units of measurement for quantity and pressure data presented on the screen and in printed reports. Page <a href="#">8-5</a> .
<b>Graph Grid Lines</b>	Allows you to specify the way in which graph lines are shown on the screen and in printed reports. Page <a href="#">8-6</a> .
<b>Sample Defaults</b>	Allows you to specify defaults for the parameters contained in the sample information file. Page <a href="#">8-7</a> .
<b>Gas Defaults</b>	Provides a table defining characteristics of gases used for analysis, to be used in calculations. Page <a href="#">8-13</a> .

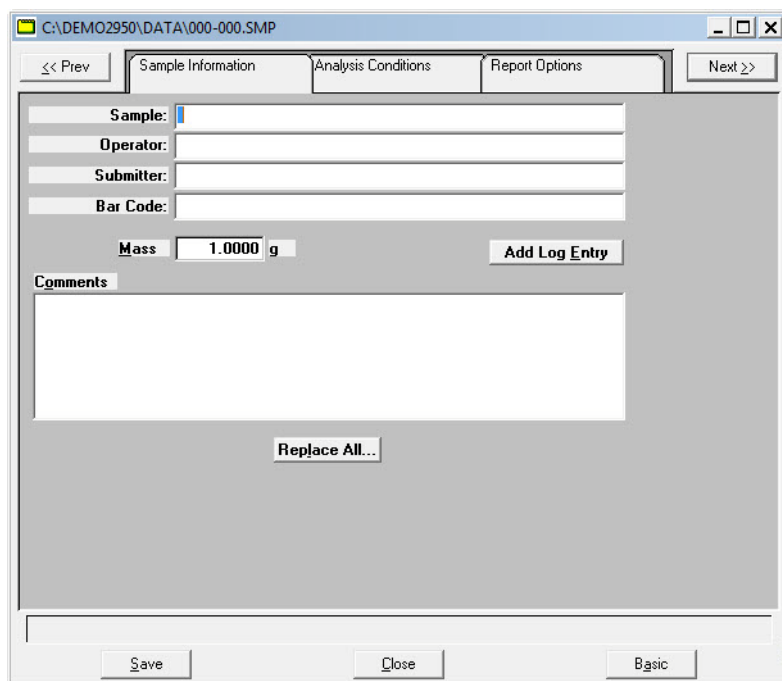
<b>Active Metal Defaults</b>	Provides a table defining characteristics of metals contained in samples, to be used in calculations. Page <a href="#">8-14</a> .
<b>Environmental Defaults</b>	Allows you to enter the current environmental conditions, so that their effect on analyses can be accounted for in calculations. Page <a href="#">8-16</a> .
<b>Parameter File Directory</b>	Allows you to specify a directory for predefined parameter files displayed in the drop-down lists of the Basic sample information dialog. Page <a href="#">8-17</a> .
<b>Service Test Mode</b>	Provides password-protected access to certain functions of the analyzer to Micromeritics-trained service personnel only. Do not attempt to access Service Test Mode unless you are specifically directed to do so by a Micromeritics service representative. Page <a href="#">8-17</a> .

## Option Presentation

Sample information file dialogs may be presented in three formats: Basic, Advanced, and Restricted.

### Advanced Format

Advanced format presents all parts of the sample information file as a group of dialog boxes with index card-type tabs (the Collected data tab does not appear until after analysis). Clicking on each tab opens an associated dialog.



The screenshot shows a software window titled "C:\DEMO2950\DATA\000-000.SMP". It features a tabbed interface with three tabs: "Sample Information", "Analysis Conditions", and "Report Options". The "Sample Information" tab is active, displaying fields for "Sample:", "Operator:", "Submitter:", and "Bar Code:". Below these is a "Mass" field set to "1.0000 g" and an "Add Log Entry" button. A "Comments" section with a large text area and a "Replace All..." button is also visible. At the bottom of the window are "Save", "Close", and "Basic" buttons. Navigation buttons "<< Prev" and "Next >>" are located at the top of the dialog area.

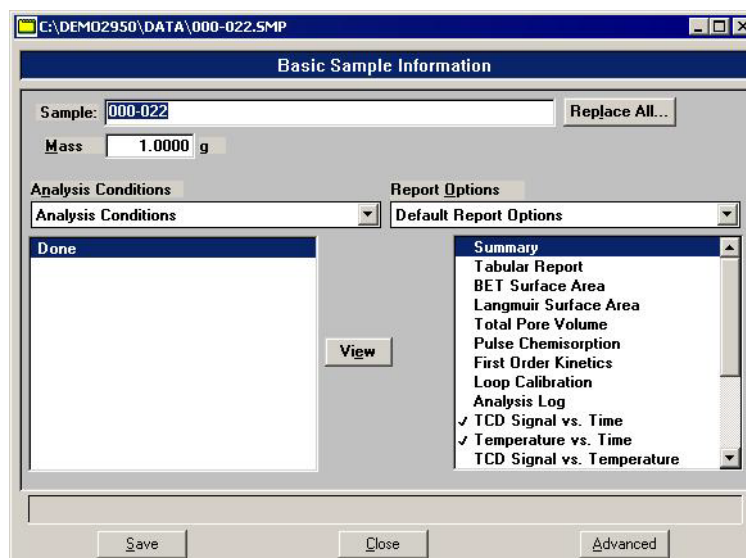


**Basic and Advanced format files provide a toggle button for conveniently switching back and forth between them, regardless of which format is specified as the default**

Use this format to edit the parameter files and create customized Sample Information files, rather than using standard parameter files. Click on the tab of the parameter to edit, or use **Next** and **Prev** to navigate among the dialogs.

## Basic Format

Basic format presents the entire sample information file in a single dialog box.



Basic format makes it easy to select previously defined parameter files from convenient drop-down lists. If editing is required, you can easily switch to the **Advanced** format by clicking **Advanced**.

## Restricted Format

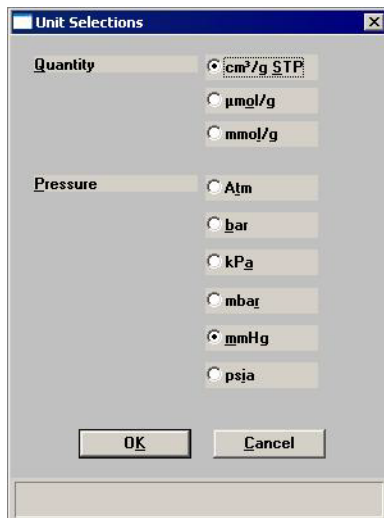
Restricted format is identical to the Basic format, except that certain menu options are disabled. You also cannot switch to the Advanced format. This format is password-protected and typically is used in laboratories where analysis conditions must remain constant, for example, in the pharmaceutical industry.



You can enter any password (up to 31 characters) to enable the Restricted format. The same password must be used to exit the Restricted format. For example, if you enter "password" to enable the Restricted format, then you must enter "password" to exit. If you forget the password, open the system INI file and navigate to the Private section. The password is shown immediately following "OptionPresentationPassword." Make a note of the password, exit the INI file, and enter the password where requested. Deleting the password from the INI file will not disable the Restricted mode; you must enter the password using the Password dialog to exit the Restricted format.

## Units

This option enables you to select the units of measurement for the quantities and pressures displayed on the screen or in printed reports.



### Quantity

Choose one of the following for quantity data presentation:

cm<sup>3</sup>/g STP  
μmol/g  
mmol/g

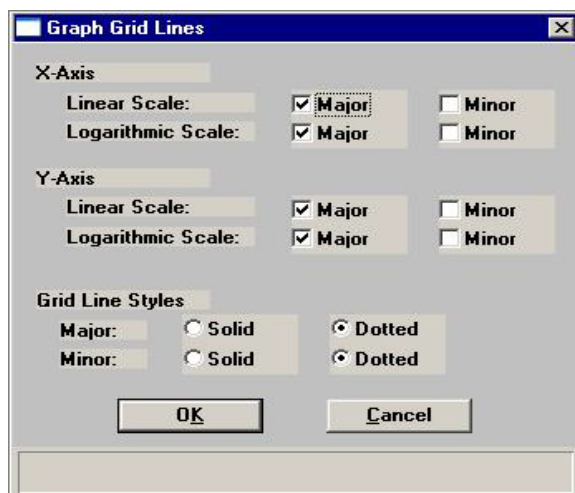
### Pressure

Choose one of the following for pressure data presentation:

Atm  
bar  
kPa  
mbar  
mmHg  
psia

## Graph Grid Lines

Use **Graph Grid Lines** to specify how graph lines are to be displayed.



### X-Axis

### Y-Axis

Enables you to choose **Major** and/or **Minor** lines to display in printed reports for the Logarithmic and Linear scales.

If you deselect these items (remove the check marks), your report will not display grid lines.

### Grid Line Styles

Allows you to choose the type of grid line to display if grid lines are being shown.

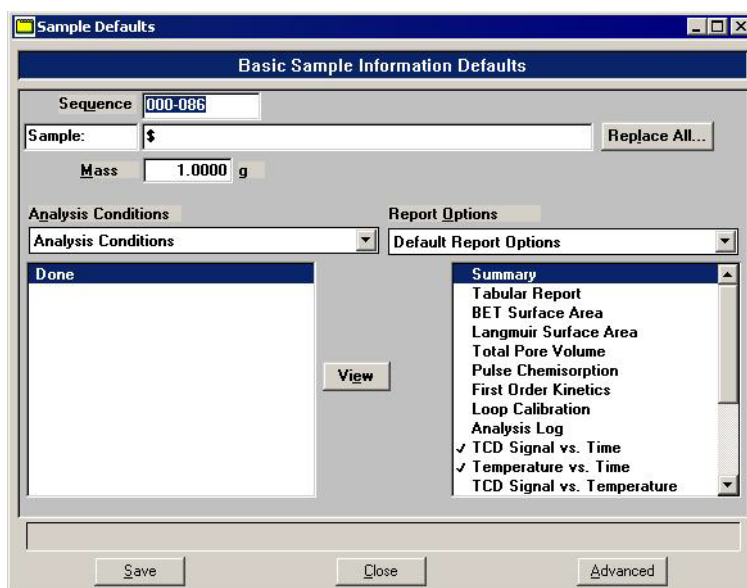
## Sample Defaults

This option allows you to specify default parameters for the dialogs used to create sample information files.

Sample defaults are the values you see in the sample information files when you create a new sample file. For efficiency, it is best to specify defaults for materials you most commonly analyze. You can always edit the values in the sample file when it is created.

The dialog used for specifying default parameters is presented in the same format you have selected as your default for sample information files. Sample defaults cannot be specified using the Restricted format.

### Basic



#### Sequence

Allows you to specify a default sequence for the file name. The number you specify is incrementally sequenced each time you create a file. It is the number that appears in the File name field when you select **File > Open > Sample Information**.

- Use numbers, letters, or other printable characters, such as dashes.
- Include at least three numbers
- Use up to eight characters.
- Do not use special characters such as \* or ?. Refer to your operating system manual for more information.

**Sample**

Allows you to enter an additional identification that provides more information than the sequence alone.

In the field on the left, you can edit the *prompt* for the sample identification. For example, you may prefer to use **Test** or **Material**.

In the field on the right, you can specify a format for the sample identification.

- Use numbers, letters, or other printable characters, such as dashes.
- Include the automatically generated file name as part of the identification by using the \$ symbol where you want the sequence number to appear.

For example, if the sequence number is 000-001, enter the sample identification as follows:

**Lab #25 - \$**

The resulting sample identification for the first Sample Information file would be:

**Lab #25 - 000-001**

and for the second one:

**Lab #25 - 000-002**, and so on.

**Replace All**

Click this button to copy the values from an existing sample information file into the default file you are creating. A dialog is displayed to choose the file to copy. Click **OK**; the values are copied into the defaults automatically. You may then edit the values in the default file without altering the sample information file from which they were copied.

**Mass**

Enter a value for the sample mass. This value can be edited in the sample file if needed.



<b>Analysis Conditions</b>	From the drop-down list, choose a default analysis conditions file. The list contains the analysis conditions files included with the software, as well as any you have created and stored in the parameter file directory (selected on the Options menu). Each step in the analysis is listed in the Synopsis and its parameters can be viewed by clicking <b>View</b> .
<b>Report Options</b>	From the drop-down list, choose a default report options file. The list contains the report options files included with the software, as well as any you have created.
<b>Report Options List Box</b>	Contains the reports available for the report options file you selected as the default. Select the report(s) to include as a default(s).
<b>Save</b>	Saves the defaults you have selected.
<b>Close</b>	Closes the dialog. If there are any unsaved changes, you are given the option to save them.
<b>Advanced</b>	Changes the presentation of the Sample Defaults dialog to the Advanced format.

## Advanced

When you select Sample Defaults while your system is defaulted to Advanced format, the Sample Defaults dialog is displayed in the Advanced format.

The screenshot shows the 'Sample Defaults' dialog box with the 'Sample Information' tab selected. The 'Sequence' field is set to '000-000'. Below it are fields for 'Sample:', 'Operator:', 'Submitter:', and 'Bar Code:', each with a corresponding 'Omit' checkbox. The 'Mass' field is set to '1.0000 g'. There is a large 'Comments' text area and a 'Replace All...' button. At the bottom are 'Save', 'Close', and 'Basic' buttons. Navigation buttons '<< Prev' and 'Next >>' are at the top.

### Sequence

Allows you to specify a default sequence for the file name. The number you specify is incrementally sequenced each time you create a file. It is the number that appears in the File name field when you select **File > Open > Sample Information**.

- Use numbers, letters, or other printable characters, such as dashes.
- Include at least three numbers.
- Use up to eight characters.
- Do not use special characters such as \* or ?. Refer to your operating system manual for more information.

### Sample

Allows you to enter an additional identification that provides more information than the sequence alone.

In the field on the left, you can edit the *prompt* for the sample identification. For example, you may prefer to use **Test** or **Material**.

**Sample**  
(continued)

In the field on the right, you can specify a format for the sample identification.

- Use numbers, letters, or other printable characters, such as dashes.
- Include the automatically generated file name as part of the identification by using the \$ symbol where you want the sequence number to appear.

For example, if the sequence number is 000-001, enter the sample identification as follows:

**Lab #25 - \$**

The resulting sample identification for the first Sample Information file would be:

**Lab #25 - 000-001**

and for the second one:

**Lab #25 - 000-002**, and so on.

**Operator**  
**Submitter**  
**Bar Code**

These fields enable you to enter defaults for the operator, submitter, and bar code information.

The fields on the left can be edited to display a different label.

The fields on the right allow you to specify default names or titles, and bar code information.

- Maximum number of characters for the fields on the left is 20.
- Maximum number of characters for the fields on the right is 42.
- Select **Omit** adjacent to any field to omit from displaying on the sample information dialog

**Mass**

Enter a value for the default sample mass. You can edit this value in the sample file if needed.

<b>Comments</b>	Enables you to enter comments concerning the sample or its conditions. The information you enter in this window will display in the report header
<b>Replace All</b>	Click this button to copy the values from an existing sample information file into the default file you are creating. Choose the file to copy. Click <b>OK</b> ; the values are copied into the defaults automatically. You may then edit the values in the default file without altering the sample information file from which they were copied.

## Analysis Conditions Defaults

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The default settings for analysis conditions files are specified using the Advanced format sample information file defaults, regardless of whether you plan to create independent analysis conditions files or complete analysis conditions dialogs as part of the sample information file.

Select **Options > Sample Defaults**; the Advanced format of the 2950 Defaults dialog is displayed; click the Analysis Conditions tab. The fields on the sample defaults dialog are identical to the ones on the Advanced format sample file, which is described on page [5-9](#).



**If you repeatedly use the same sample preparation techniques, then vary the analysis parameters to make the default analysis conditions the preparation steps alone. Then the preparation steps will always be in place when you open a new sample information file, ready for you to add analysis steps as needed.**



**When the first experiment is inserted into an analysis conditions file, its initial values are set by the first experiment step in the default analysis conditions. Including a single “default” experiment step in your default sample file will ensure that new experiments are properly initialized.**

## Report Options Defaults

---

The default settings for report options files are set using the Advanced format sample information file defaults, regardless of whether you plan to create independent report options files or complete report options tabs as part of the sample information file.

Select **Options > Sample Defaults**; the 2950 Defaults dialog is displayed in Advanced format; click the Report Options tab. The fields on the sample defaults dialog are identical to the ones on the Advanced format sample file, which is described on page [5-34](#).

## Gas Defaults

In order to characterize the gases used in an analysis, a symbol and a Mass Flow Controller (MFC) Constant must be defined in the Gas Defaults table.

	Gas	Symbol	MFC Constant	% Active Concentration
1	10 H2-Ar	10H2-AR	1.335	10.000
2	2 O2-He	2O2-HE	1.365	2.000
3	30 N2-He	30N2-HE	1.234	30.000
4	Ammonia	NH3	0.782	100.000
5	Argon	AR	1.386	0.000
6	Carbon Monoxide	CO	0.980	100.000
7	Helium	HE	1.376	0.000
8	Hydrogen	H2	1.000	100.000
9	Nitrogen	N2	0.995	0.000
10	Oxygen	O2	0.980	100.000

'Ctrl-Down Arrow' to append

### Symbol

Lists the standard symbol (mnemonic) for each gas.

### MFC Constant

Contains the MFC Conversion Constant for each gas listed. This constant allows the MFC to control the gas flow precisely. If this number is incorrect, the actual flow rate for that gas differs from the rate reported.

**This value defaults to 1 when a new gas is listed.** You must enter the correct gas constant. A table of MFC conversion constants is provided in Appendix C. If the gas you are using is not listed in the table, or if you are using a mixture of gases, you can determine the correct MFC Constant using the instructions in Appendix F.



In general, the MFC conversion constants provided with the analyzer are accurate and should be used. If you suspect that a MFC is not functioning properly, calibrate the MFC.

### % Active Concentration

Displays the percentage of the gas that is active.

You can change any one of the values in the table by highlighting it and entering the new value. You can also edit the gases listed using the following buttons:

- Insert** Inserts a row in the table in which you may enter a new gas and its values. The new row is inserted above the row in which the cursor is located.
- Delete** Deletes the selected row.
- Clear** Clears all but one entry of the table, after confirmation. One entry is required.

## Active Metals Defaults

In order to characterize the active metals in a sample, certain properties of the metals must be defined.



**The values defined in this table are the defaults used when a new entry is inserted into the metal table of the Pulse Chemisorption report options. Changing this table does not affect the tables within the report options files.**

A table containing default values for the following properties is provided in Appendix G:

Active Metals Defaults					
	Element	Atomic Weight	Atomic Area (nm <sup>2</sup> )	Density (g/cm <sup>3</sup> )	Stoichiometry Factor
1	Mercury	1.000	0.0000	1.000	1.000
2	chromium	51.996	0.0635	7.200	1.000
3	cobalt	58.933	0.0662	8.900	1.000
4	copper	63.540	0.0680	8.920	1.000
5	molybdenum	95.940	0.0730	10.200	1.000
6	nickel	58.710	0.0649	8.900	1.000
7	palladium	106.400	0.0787	12.020	1.000
8	platinum	195.090	0.0800	21.450	1.000
9	rhenium	186.200	0.0649	20.530	1.000
10	rhodium	102.905	0.0752	12.400	1.000
11	silver	107.868	0.0869	10.500	1.000

'Ctrl-Down Arrow' to append

### Atomic Weight

The atomic weight of the element.

*Range: 1.0000 to 500.0000*

**Atomic Area**

The atomic cross-sectional area of the element.

*Range: 0.0000 to 9.9999 nm<sup>2</sup>*

**Density**

The density of the element.

*Range: 0.0000 to 99.9999 g/cm<sup>3</sup>*

**Stoichiometry Factor**

A factor which expresses the ratio between the number of active metal atoms and the number of adsorbate molecules. This number varies, based on the adsorbate used. The most common value should be entered into the defaults table. Within the Pulse Chemisorption report options, the value for a specific analysis can be set.

The following are values for some common gases:

Hydrogen: 2

Oxygen: 2

Carbon Dioxide: 1

2 is commonly assumed for a dissociative chemisorption.

1 is commonly assumed for a non-dissociative chemisorption.

You can change any of these values by highlighting it, then entering the value you prefer. You can also edit the elements listed using the following buttons:

**Insert**

Inserts a row in the table in which you may enter a new gas and its values. The new row is inserted before the row in which the cursor is located.

**Delete**

Deletes the selected row.

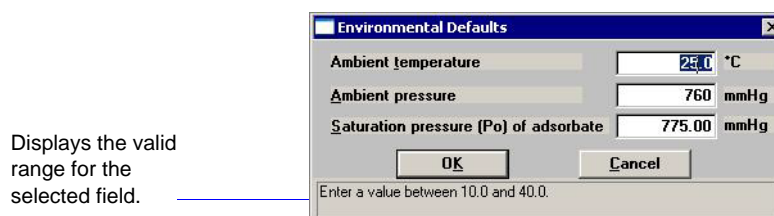
**Clear**

Clears all but one entry of the table, after confirmation. One entry is required.

## Environmental Defaults

To ensure accurate loop calibration and BET surface area data, you should enter current environmental conditions every day, or whenever there is a significant change in one or more of the environmental conditions. These values are used in some calculations to account for the effect of environmental conditions on the analysis. When you select this option, the Environmental Defaults dialog opens.

As an analysis is started, these parameters are saved in the Collected Data portion of the sample information file.



Valid ranges for the selected field are shown in the information bar on the dialog

For the highest quality of results, these defaults should be updated on a regular basis.

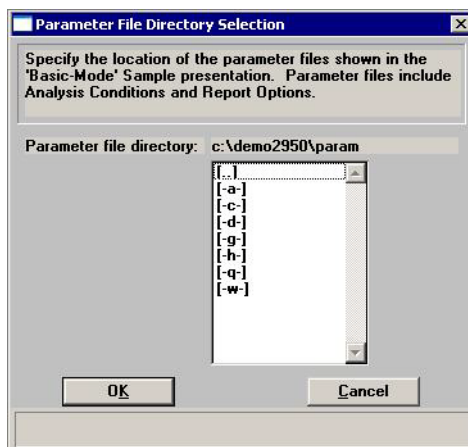
<b>Ambient temperature</b>	Enter the temperature of the room in which the analyzer is located.
<b>Ambient pressure</b>	Enter the atmospheric pressure of the room in which the analyzer is located.
<b>Saturation pressure (Po) of adsorbate</b>	Enter the saturation pressure of the adsorbate gas in the room in which the analyzer is located. Disregard this field if the saturation pressure of the adsorbate gas is not pertinent to your analysis.



## Parameter Files Directory

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When you select this option, the Parameter File Directory Selection dialog is displayed.



This option allows you to choose which analysis conditions and report options files (parameter files) display in the drop-down lists on the Basic format sample information dialog. You do so by selecting a directory. Parameter files located in the specified directory appear in the drop-down lists; files located in other directories do not.

For this reason, it is important that whenever you create an independent parameter file, you save it in the correct directory. The destination directory is specified on the Open Analysis Conditions or Open Report Options dialog when you first begin to create the parameter file. To be sure that the parameter files you create are included in the drop-down list, make sure that the directory they are saved to matches the directory specified using this option.

## Service Test Mode

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Various service tests are included in the AutoChem 2950 HP operating program. These tests can be performed only with the assistance of a trained Micromeritics service representative. When you select **Service Test Mode** from the Options menu, a dialog box prompting you to enter a password is displayed. This password is coded to change on a regular basis and is known only by your service representative. You will not be able to perform these tests without his guidance.



## 9. MAINTENANCE AND TROUBLESHOOTING

This chapter describes potential operational problems and their solutions, and maintenance procedures. If further assistance is needed after following the procedures in this chapter, contact a Micromeritics service representative.

### Troubleshooting

Operating problems encountered with the analyzer are usually easily corrected. Typical problems and corrective actions are described in the following table.

*Table 9-1. Potential Operational Problems*

What Happened	Why	What To Do
Unit does not work when power switch is turned on.	Power cord not fully inserted at one of the ends.	Insert power plug firmly into outlet socket; insert unit connector into power connector opening.
	No power at outlet.	Plug in lamp or small appliance to test outlet. If there is no power, contact electrician.
	Plug prongs bent so that contact not made at outlet.	Gently move power plug at outlet while watching the status LEDs on the front panel. If at least one LED becomes illuminated, have an electrician replace the outlet or the plug.
	Power cord damaged.	Have electrician check cord using a test meter. Replace cord if defective.
	Loose internal connection or broken wire.	Call a Micromeritics Service Representative for repair or replacement.
Liquid nitrogen boils away too quickly to complete an analysis.	Dewar damaged.	Replace Dewar.
	Lengthy analysis.	Refill Dewar during analysis.
Specified temperature not reached or not maintained.	Thermocouple or other internal component is damaged or disconnected.	Call the appropriate service personnel.

**Table 9-1. Potential Operational Problems (continued)**

What Happened	Why	What To Do
No gas flow to the analyzer.	Leaks in the gas line connection.	Replace the gas tank; then pressurize the system. Close, then open the cylinder valve. If the needle on the pressure gauge on the gas cylinder jumps abruptly, a leak in the gas line connection may be indicated. Check all gas line connections.
Too much heat supplied to analyzer.	Thermocouple or other internal component is damaged or disconnected.	Call the appropriate service personnel.
A stable TCD baseline cannot be maintained.	Faulty RTD in one of the temperature zones.	Access manual mode and check for temperature stability in the temperature zones. Refer to <b>“Enable Manual Control” on page 6-12.</b>
	Possible leak in carrier path.	Perform leak test in the carrier path. Refer to <b>“Performing a Leak Test” on page 9-26.</b>
	TCD filaments are contaminated or need to be replaced.	Call the appropriate service personnel.
Data collection results in very high or very low peaks that are inconsistent with previous experience.	A different (lower) TCD temperature is being used.	Repeat the analysis using a higher temperature.
	TCD filaments are contaminated or need to be replaced.	Call the appropriate service personnel.
During data collection, a TCD signal of zero is recorded, or no peaks are seen.	TCD temperature was not reset after a run automatically suspended.	Be sure to re-enable the TCD after all run suspensions.
	TCD filaments are contaminated or need to be replaced.	Call the appropriate service personnel.
Furnace will not heat.	Furnace not closed and latched.	Close and latch the furnace.
	The furnace is not plugged in.	Plug in the furnace connector.
	Thermocouple damaged.	If unplugged, plug in the thermocouple. Replace the thermocouple if necessary.
Pressure will not rise or readings very unstable.	Furnace not closed and latched.	Close and latch the furnace.
	The furnace is not plugged in.	Plug in the furnace connector.

## Preventive Maintenance Schedule

The table below lists the preventive maintenance procedures you should complete to keep your analyzer operating at peak performance. Instructions for each procedure follow the table. Micromeritics also recommends that you have preventive maintenance procedures and calibration performed by one of our service representatives every 12 months.

Maintenance Required	Frequency
Clean Dewar, <b>“Cleaning the Dewar” on page 9-4</b>	As required or once a month
Clean or change the cold trap or delay path, <b>“Cleaning or Changing the Cold Trap or Delay Path” on page 9-4</b>	Check one a month Clean or change as required or every 3 months
Clean foam air filter, <b>“Changing or Cleaning the Foam Air Filter” on page 9-7</b>	As required or every 3 months
Replace sample tube filter and O-ring, <b>“Changing the Sample Tube Filter” on page 9-10</b>	As required or every 3 months
Replace cold trap O-ring, <b>“Replacing the Cold Trap O-Ring” on page 9-12</b>	As required or every 6 months
Change septum, <b>“Changing the Septum” on page 9-13</b>	As required or every 6 months
Reposition or replace the sample thermocouple, <b>“Recalibrating the Sample Thermocouple” on page 9-24</b>	Reposition as required Replace as required or every 6 months
Recalibrating the sample thermocouple, <b>“Recalibrating the Sample Thermocouple” on page 9-24</b>	When the sample thermocouple is replaced
Clean the outside of the analyzer, <b>“Cleaning the Analyzer” on page 9-26</b>	As required or every 6 months
Test analyzer for leaks, <b>“Performing a Leak Test” on page 9-26</b>	As required or every 12 months

Perform Reference Material analysis, <b>“Performing a Reference Material Analysis”</b> on page 9-30	As required
Perform Mass Flow Controller calibration, <b>“Performing a Mass Flow Controller Calibration”</b> on page 9-30	When using a gas or gas mixture not contained in the Gas Defaults table

## Preventive Maintenance Procedures

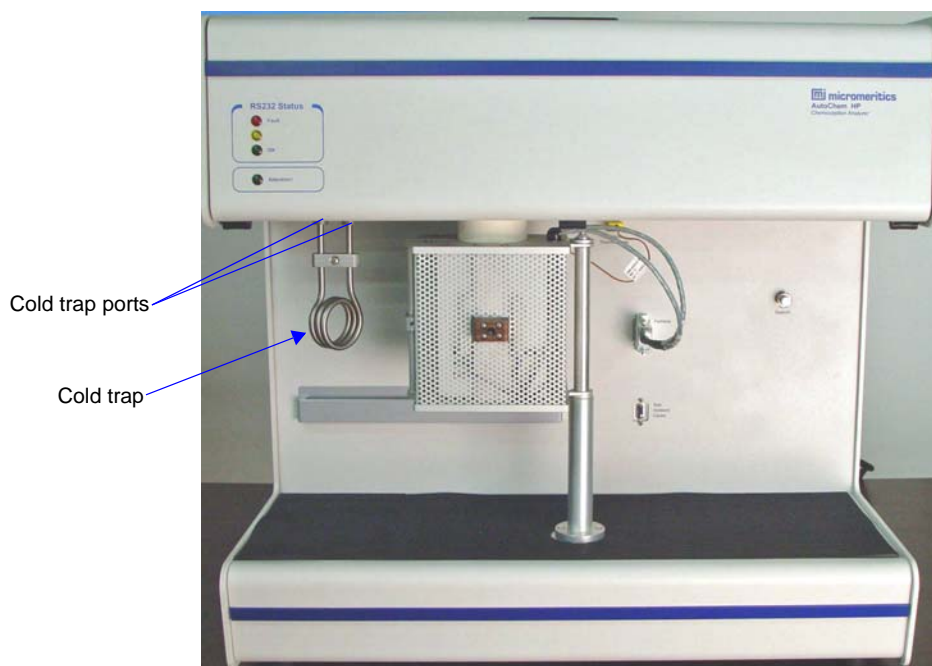
### Cleaning the Dewar

To clean the Dewar, empty it, then rinse it with water. Allow the Dewar to dry thoroughly before using again.

Be sure to observe Dewar precautions when cleaning the Dewar. Refer to **“Precautions”** on page 3-18.

### Cleaning or Changing the Cold Trap or Delay Path

Because the cold trap removes condensibles from the gas stream, it will require occasional cleaning. The delay path (used for BET analyses) also attaches to the cold trap port fittings. If you need to change from one attachment to the other, follow the steps below, but substitute the appropriate attachment during reassembly.



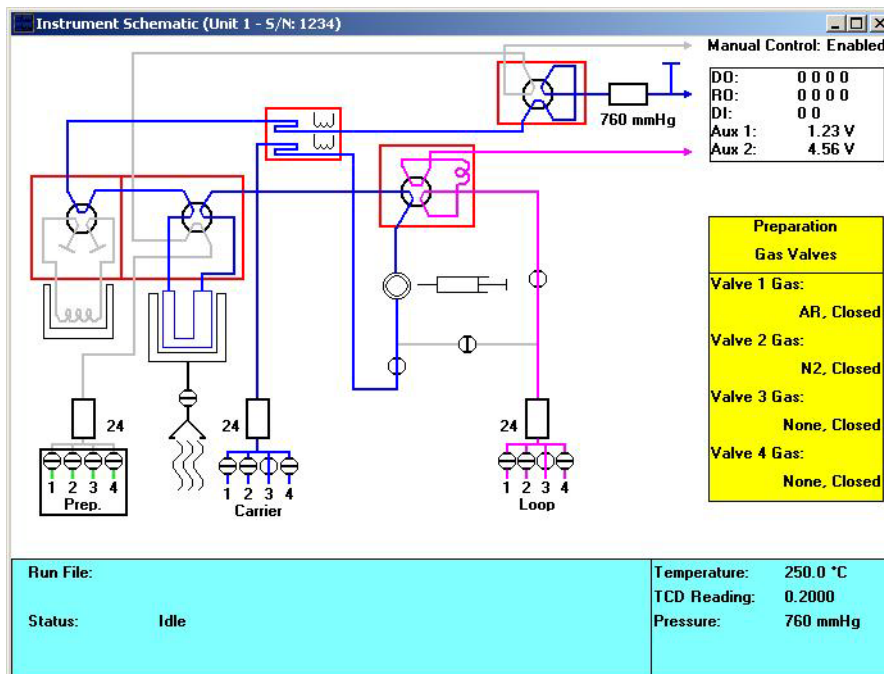
To clean or change the cold trap or delay path, follow the steps below.

1. If a Dewar is around the cold trap, raise the Dewar slightly, slide the stand out of the way or remove it, and remove the Dewar. Allow the cold trap to warm to room temperature.

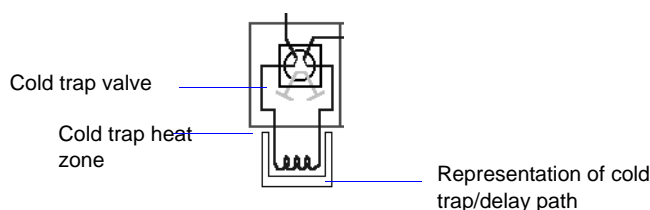


**Depending upon the state of the analyzer, the cold trap fittings may be hot. Use caution. If necessary, wear cotton gloves to protect your hands.**

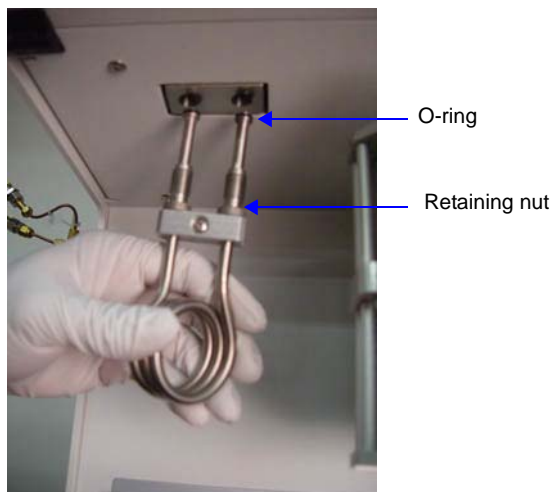
2. Select **Unit > Show instrument schematic**, then select **Unit > Enable manual control**.



3. Right-click on the cold trap valve (the detail from the schematic is shown below) and choose **Bypass**.



4. Loosen the retaining nuts on the cold trap port fittings. (These can be unscrewed using your fingers.)



5. Remove the cold trap (or delay path) and O-rings.
6. Clean the cold trap (or delay path) by flushing it with isopropyl alcohol (IPA). Dry the trap in an oven or by blowing compressed air or nitrogen through it before replacing it.



**Use only Kalrez™ O-rings. Kalrez is rated for both chemical and temperature suitability in this application. O-rings of other materials could burn, melt, or decompose.**

7. Inspect the O-rings for cracks or other damage that might cause leaks. If the O-rings are cracked or damaged, replace them before reinstalling the cold trap (or delay path). Otherwise, clean the O-rings you removed, dry them, and reinstall them.
8. Place the O-rings on the cold trap (or delay path).



**Do not overtighten the retaining nuts. Doing so may damage the O-rings. Finger-tight is sufficient.**

9. Hold the cold trap (or delay path) in place and tighten the retaining nuts. Tighten the nuts only enough to hold the cold trap securely in place and prevent leaks.



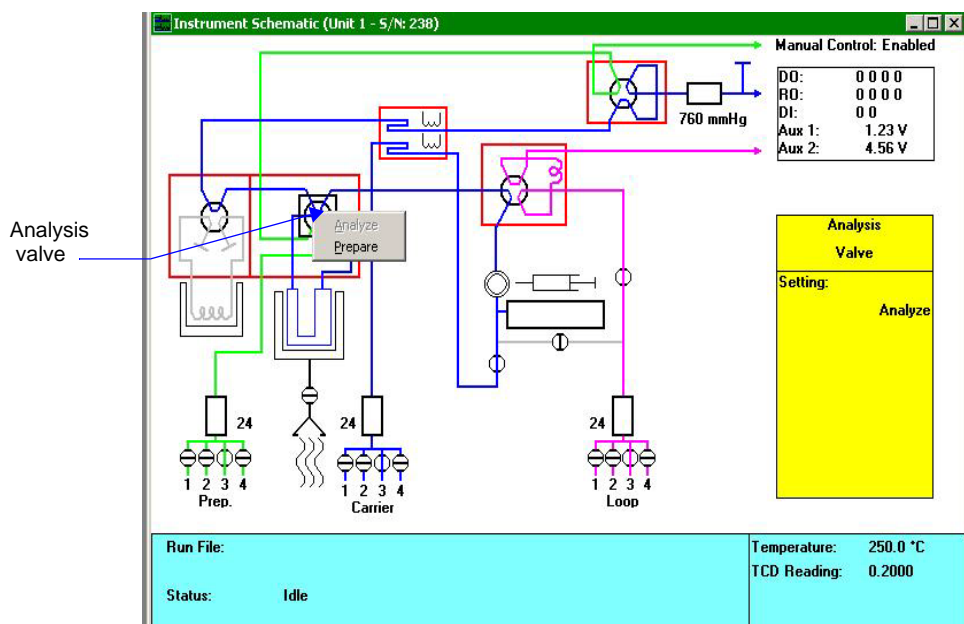
## Changing or Cleaning the Foam Air Filter

The air filter in the AutoChem II analyzer should be checked every 30 days and cleaned when necessary, at least once every three months. If, after cleaning, you determine that the filter is degrading, you should replace it.

1. Click **Unit > Show instrument schematic** to display the instrument schematic.
2. Right-click the Analysis valve and ensure that it is in the **Prepare** position.

If it is not in the Prepare position:

- a. Click **Unit > Enable Manual Control**.
- b. Right-click the Analysis valve and click **Prepare**.



3. Remove the furnace and any accessories from the front of the analyzer.

4. Remove the black mat from the lower panel.



5. Remove the screws that hold the stainless steel support plate in place.



6. Hold the furnace support post and lift the support plate up and away from the analyzer.



7. Pull the filter up and out of the analyzer.



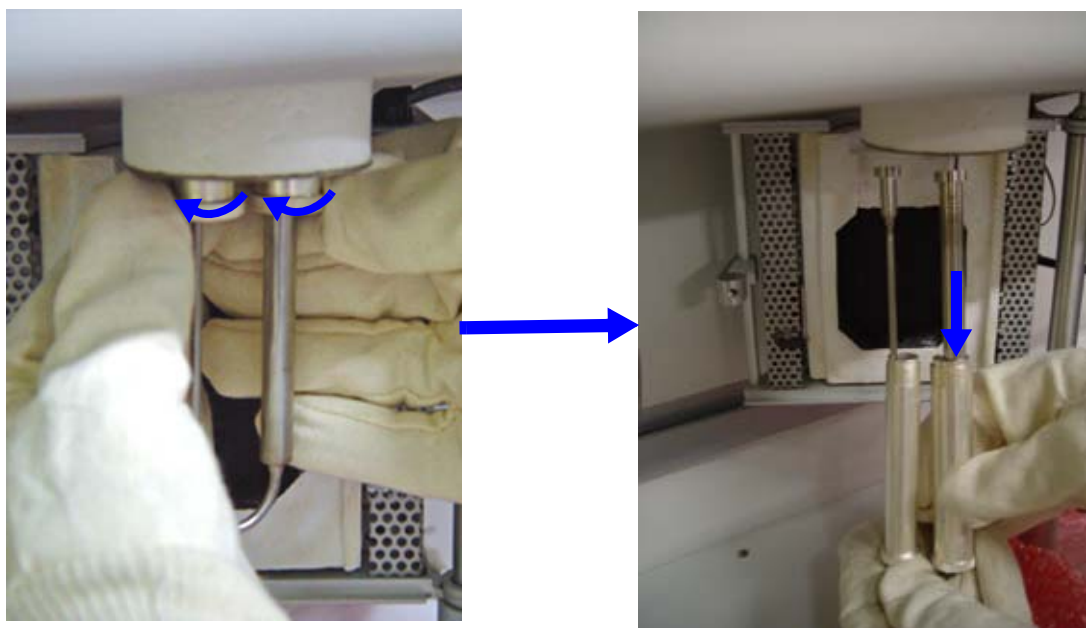
8. Check the filter and, if dusty, rinse it with clean water and allow it to dry completely.
9. If the filter is in good condition, place it back inside the instrument. If the filter needs to be replaced, obtain a new filter and place it inside the instrument.
10. Place the support plate back into position and reinstall its retaining screws.
11. Reinstall the furnace and accessories.

## Changing the Sample Tube Filter

A frit filter is used to protect the analyzer internal components from contaminants that may be forced out of the sample tube during an analysis. However, as the analyzer is used, the filter may gradually become clogged with trapped particles. If the filter does become blocked, it must be removed and cleaned or replaced.

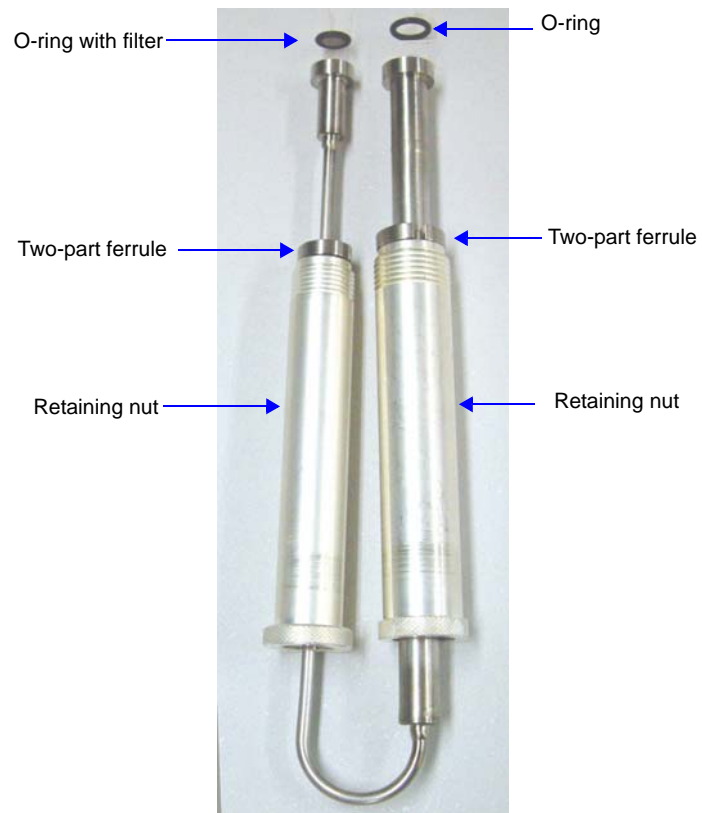
To change the analysis port filter, follow the steps below:

1. Loosen the analysis port fittings while holding the sample tube.
2. Carefully remove the sample tube by unscrewing the retaining nuts. Be careful not to allow the nuts to slide down onto the curved portion of the sample tube or a quartz tube may break.



**The sample tube and nearby components may be hot. Make sure you allow the sample tube to cool before removing it. Use the cotton gloves provided with the analyzer to protect your hands.**

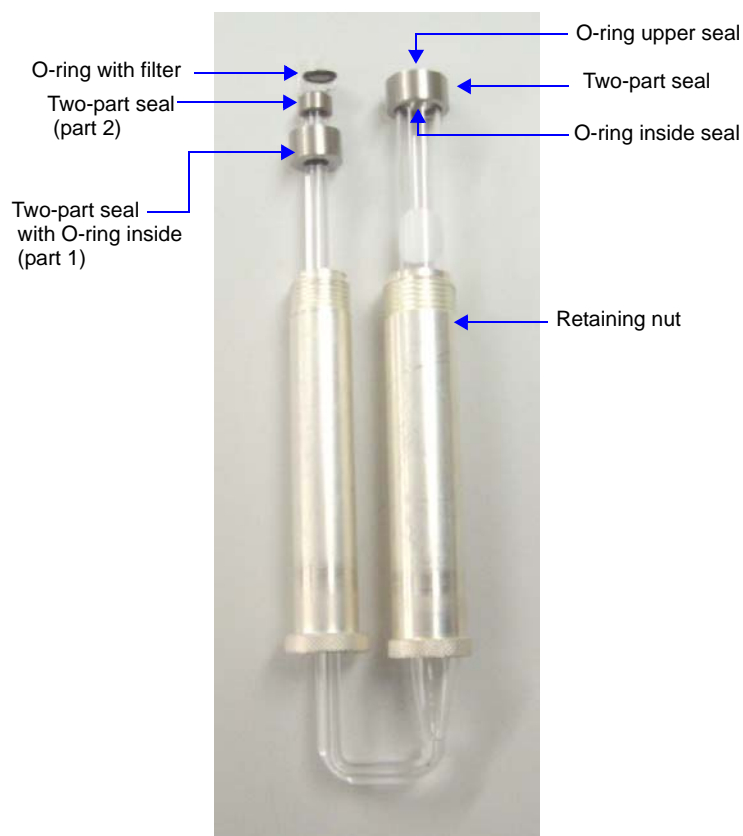
3. Inspect the O-ring for cracks or cuts. If the O-ring is damaged, replace it. Otherwise, clean and dry the O-ring you removed.
4. Using a new filter, reassemble the sample tube components as shown in either *Assembly A* or *Assembly B* on the following pages.



*Assembly A: Metal Tube*



**Use only Kalrez™ (or equivalent) O-rings. Kalrez is rated for both chemical and temperature suitability in this application. O-rings of other materials could burn, melt, or decompose.**



*Assembly B: Quartz Tube*

## Replacing the Cold Trap O-Ring

When using the cold trap, if the baseline begins to drift, that may be an indication that the O-rings in the cold trap fitting are worn and need to be replaced.

To replace the cold trap O-rings, follow the instructions for **“Cleaning or Changing the Cold Trap or Delay Path” on page 9-4**. In step 8, insert a new O-ring.

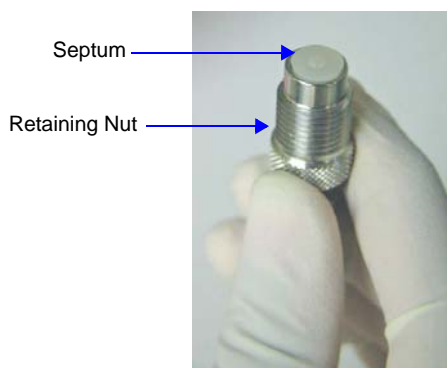
## Changing the Septum

Because the septum is pierced each time you perform an analysis requiring an injection, it will eventually leak. When the analyzer septum begins to leak, replace it with a new septum.

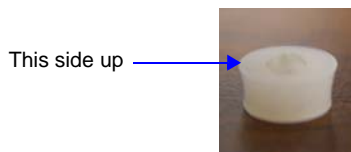
1. Turn the knurled septum retaining nut on the front panel counterclockwise to loosen it.



2. When the retaining nut is free, remove it with the old septum.
3. Remove the old septum from the retaining nut.



4. Install a new septum using the original retaining nut. Insert the septum with the larger end up (see below).



**Do not use a wrench to tighten the septum retaining nut. Do not overtighten the retaining nut. Doing so may damage the septum or the fitting.**

5. Use your fingers to turn the septum retaining nut clockwise until it is tight. You will feel resistance as the septum is squeezed, and then the nut will be difficult to tighten further.

## Repositioning, Replacing, or Adjusting the Sample Thermocouple

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The sample thermocouple is designed to pass either through the analysis port and into the sample tube or through an adjacent opening to rest along the side of the sample tube.

### Replacing the Thermocouple

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Because the sample thermocouple is heated to high temperatures, it will eventually fail and must be replaced. Two indications that the sample thermocouple may need to be replaced are unreasonably high sample temperature readings or erratic sample temperature readings.

To replace the thermocouple, follow the instructions for [Changing the Position of the Thermocouple](#) below. Instead of moving the thermocouple from one position to the other, disconnect the used thermocouple from the cable that attaches it to the analyzer. Attach the new thermocouple to the cable and place it in the appropriate opening.



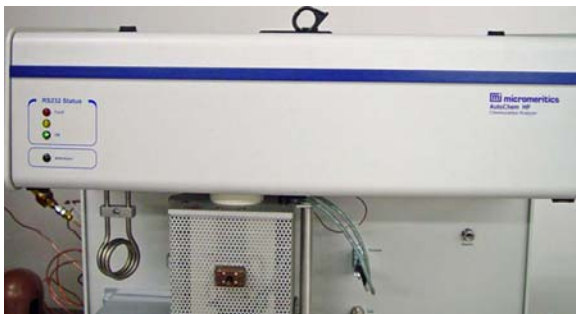
**After you replace the sample thermocouple, you must calibrate it. Refer to [“Recalibrating the Sample Thermocouple”](#) on page 9-24.**



## Changing the Position of the Thermocouple

To change the position of the thermocouple:

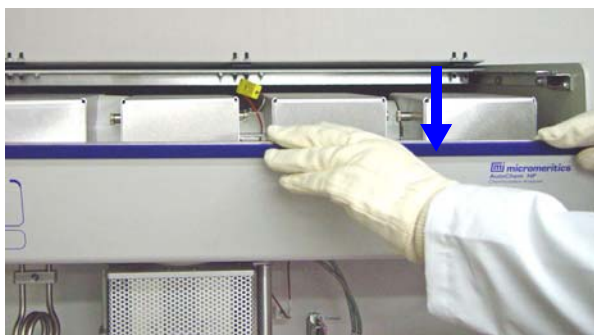
1. Turn the analyzer's power switch off, then disconnect the analyzer from its power source.
2. Press in on the right side of the retractable handle on the top of the analyzer to protrude the handle.



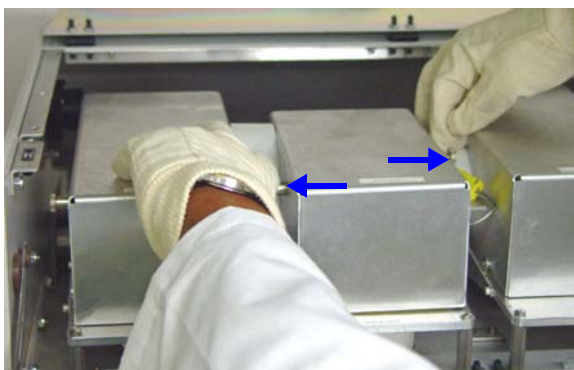
3. Using the handle, open the top panel. The top panel is hinged to allow easy access to its components; it is not necessary to remove the top panel.



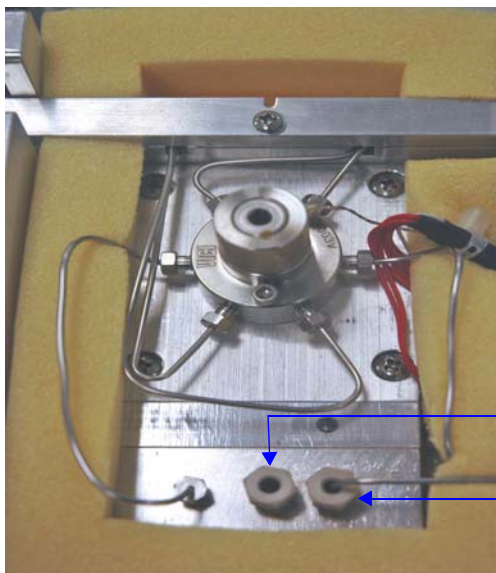
4. Pull down the upper front panel.



5. The Sample valves are in the second block from the left. The cover is labeled **Sample Valve**. The cover is held in place by two knobs or plungers. Remove the valve cover by pulling the plungers outward until the valve cover is released. Pull cover up and out.



6. Remove the insulation that surrounds the Sample valve ports. The thermocouple ports are located toward the front of the block.



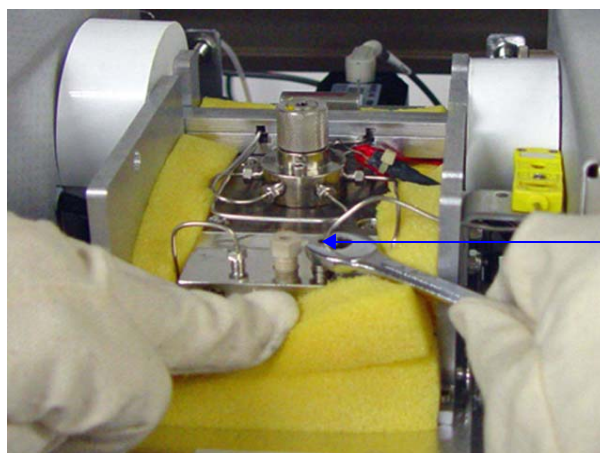
Use the left port to place the thermocouple outside the tube

Use the right port to place the thermocouple inside the tube



The sample thermocouple and nearby components may be hot. Make sure you allow the sample thermocouple to cool before removing it. Use the cotton gloves provided with the analyzer to protect your hands. If the zone is not near room temperature, loosening or tightening the fittings may cause damage to components of the instrument.

7. Use an open-ended wrench to loosen the sample thermocouple fitting.



Thermocouple fitting

8. Loosen the fitting for the plug in the alternate thermocouple position and remove the plug.
9. Remove the fitting that retains the sample thermocouple.
10. Move the sample thermocouple from its current position to the new position.

If you are using the quartz sheath, perform the following before proceeding to the next step. Remember, the sheath can only be used when the thermocouple is being placed inside the sample tube.

- a. Remove the ferrule from the thermocouple, straighten the thermocouple as much as possible.
- b. Insert the thermocouple into the sheath.
- c. Place a larger ferrule around the sheath.



**The sheath is very fragile and can be easily broken. Because the sheath is made of quartz, you must use the cotton gloves when handling it. (Oils from your fingers may lower the quartz' melting point.)**

11. Place the stainless steel plug in the unused thermocouple opening and tighten the fitting. Reinstall the sample thermocouple retaining screw.
12. Adjust the exposed portion of the sample thermocouple to the appropriate length. Refer to **“Determining the Length of the Thermocouple” on page 9-18.**
13. Tighten the retaining screw.
14. Place the insulation in its original position. Reinstall the Analyze/Prepare valve cover.
15. Return the upper front panel and the top panel to their original closed positions.

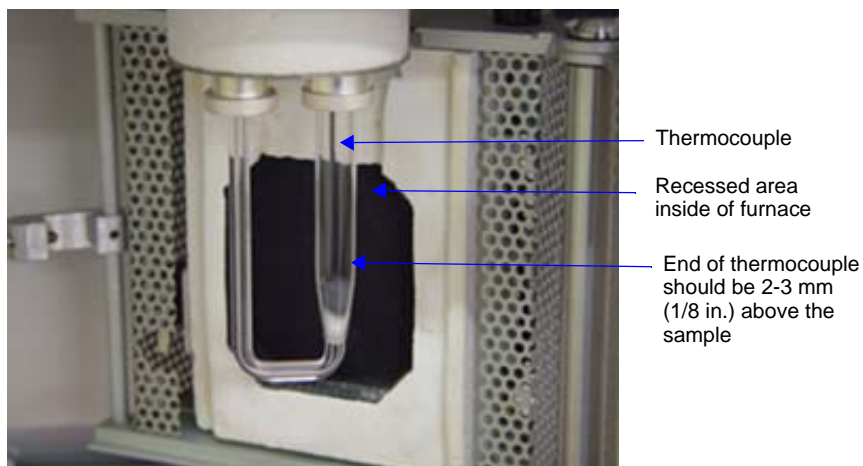
16. Turn the instrument's power switch on.
17. If you replaced the thermocouple, calibrate it as described in the next topic.

## **Determining the Length of the Thermocouple**

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### **Guidelines**

When you change the type or amount of sample being analyzed, you may need to adjust the length of the sample thermocouple to ensure that it is positioned just above the sample. Follow the guidelines below when adjusting the thermocouple length.



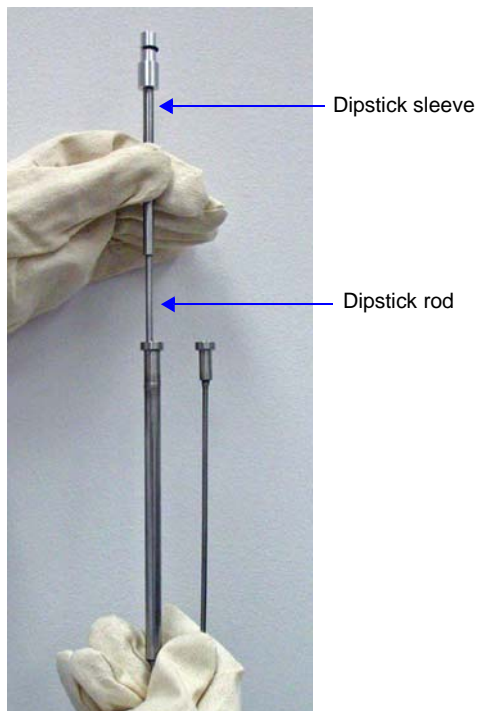
- The thermocouple's end should never be placed outside the painted (black), recessed area inside the furnace
- If the thermocouple is positioned inside the tube, its end should generally be located approximately 2-3 mm (1/8 in.) above the sample surface. In some cases, you may allow the thermocouple to extend into the sample.
- If the thermocouple is positioned outside the tube, its end should not extend below the point where the sample tube begins to taper.

### **Metal Tubes**

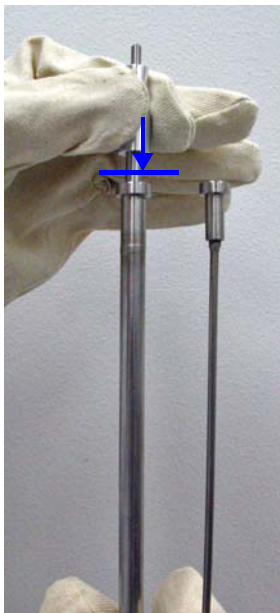
When using metal tubes, it may be helpful to use the dipstick included with the instrument accessories to determine the appropriate length of the sample thermocouple. At times, a small amount of sample may adhere to the dipstick. When using very small amounts of sample for an analysis, the amount that adheres to the dipstick may affect analysis results.

To use the dipstick:

1. After inserting the quartz wool and sample into the sample tube, insert the dipstick into tube as shown below.



2. When the dipstick reaches the sample, gently press down on the sleeve until it is flush with the top of the sample tube.

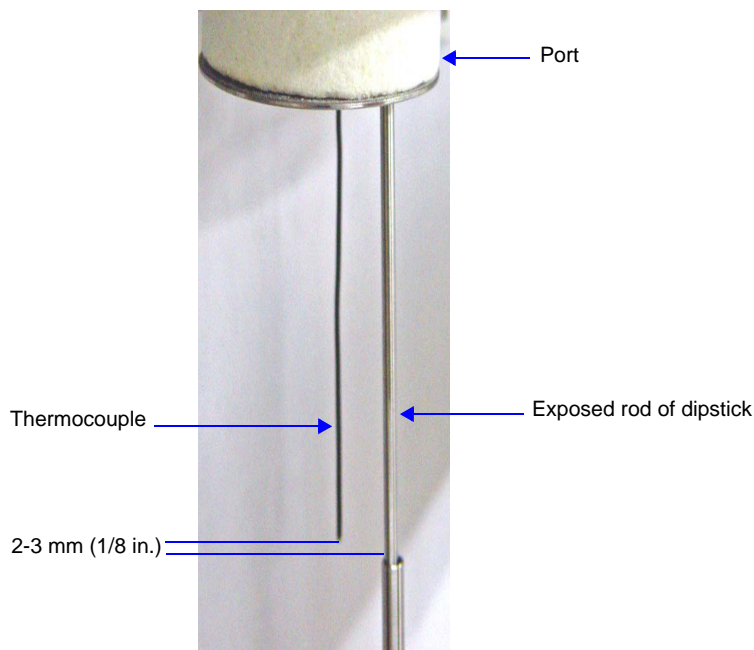


3. Remove and invert the dipstick.



The exposed rod indicates the approximate length for the external portion of the thermocouple

4. Place the inverted dipstick on the underside of the analysis port close to the thermocouple. Make sure you place it on a flat surface and not into one of the openings in the port.



The end of the thermocouple should be 2-3 mm (1/8 in.) above the exposed rod. If it is not, adjust the thermocouple length as needed. (Refer to **“Adjusting the Length of the Thermocouple” on page 9-21.**)

### Adjusting the Length of the Thermocouple

---

If the thermocouple is positioned inside the tube, its end should generally be located approximately 2-3 mm (1/8 in.) above the sample surface. In some cases, you may allow the thermocouple to extend into the sample.

If the thermocouple is positioned outside the tube, its end should not extend below the point where the sample tube begins to taper.

If you need to adjust the sample thermocouple to achieve the correct location, follow the instructions below.



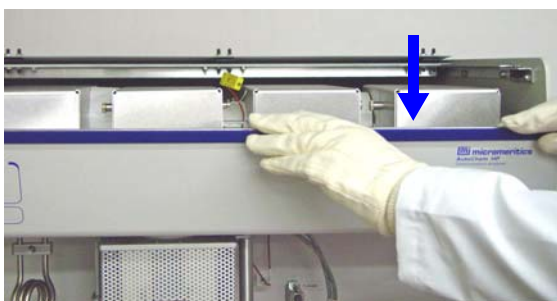
1. Press in on the right side of the retractable handle on the top of the analyzer to protrude the handle.



2. Using the handle, open the top panel. The top panel is hinged to allow easy access to its components; it is not necessary to remove the top panel.

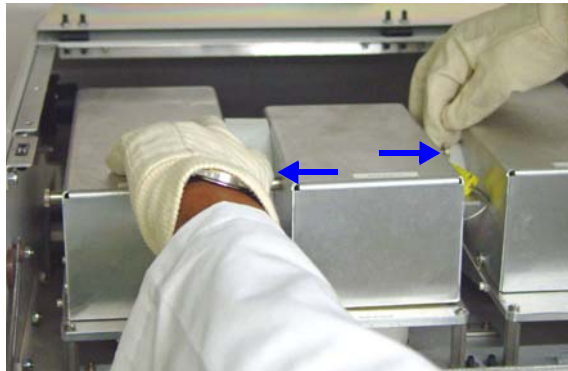


3. Pull down the upper front panel.





4. The thermocouple connector is in the second block from the left. The cover is labeled **Sample Valve**. The cover is held in place by two knobs or plungers. Remove the cover by pulling the plungers outward until the cover is released. Pull cover up and out.

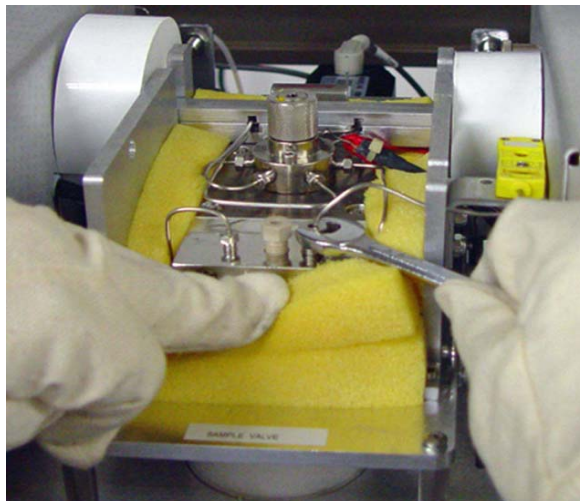


5. Remove the insulation that surrounds the Sample valve ports. The thermocouple ports are located toward the front of the block.



**The sample thermocouple and nearby components may be hot. Make sure you allow the sample thermocouple to cool before removing it. Use the cotton gloves provided with the analyzer to protect your hands. If the zone is not near room temperature, loosening or tightening the fittings may cause damage to components of the instrument.**

6. Use an open-ended wrench to loosen the sample thermocouple fitting.



7. Adjust the thermocouple wire up or down as needed.
8. Tighten the thermocouple fitting.
9. Place the insulation in its original position. Reinstall the cover.
10. Return the upper front panel and the top panel to their original closed positions.
11. Turn the instrument's power switch on.

## Recalibrating the Sample Thermocouple

A complete calibration file is created specifically for each analyzer before the analyzer is shipped. This calibration file is installed during initial software installation. You may need to recalibrate the sample thermocouple if you replace the thermocouple or if the original calibration file is corrupted or missing.



**Do not attempt to calibrate anything other than the sample thermocouple without prior approval from a qualified Micromeritics Service Representative.**

Use the following instructions to recalibrate the sample thermocouple.

Before beginning, make sure:

- The analyzer and software are turned on.
- You are familiar with manually controlling the analyzer.
- All gas flows through the analyzer are stopped.
- All heat zones are stabilized at ambient temperature.
- The furnace is open and the sample tube is removed (the thermocouple is bare and exposed).
- A traceable thermocouple meter and a water bath (ambient temperature) to immerse the sample thermocouple are available.

1. Select **Unit > Instrument Calibration**; the Instrument Calibration dialog opens.

	Detector Heat Zone	Cold Trap Heat Zone	Analyze Heat Zone	Loop Heat Zone	Back Press. Heat Zone
Reading	103	103	103	103	103
Low Cal.	20	0	0	0	-15
High Cal.	200	200	200	200	15
Calibrated	1/12/2006				
Raw Value	12345	12345	12345	12345	12345

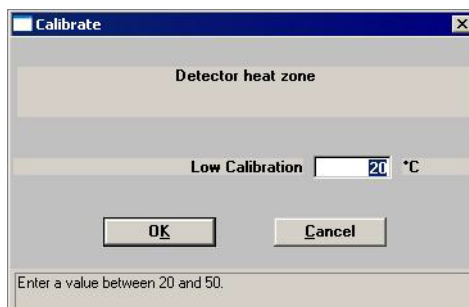
	Sample Temperature *	Sample Pressure *	Auxiliary Input1	Auxiliary Input2	Furnace Temperature *
Reading	251	760	1.23	4.56	251
Low Cal.	0	-8274	14.7	-15.0	0
High Cal.	200	51715	1000.0	15.0	1100
Calibrated					
Raw Value	12345	123	1234	1234	12345

Buttons: Load from file..., Save to file..., Accept, Cancel, Reset, Furnace PID..., Sample PID..., MEC Full Scale...

\* Be sure not to exceed recommended temperatures and pressures for the type of sample tube in use.

Run File:	Temperature: 250.0 °C
Status: Idle	TCD Reading: 0.2000

2. Place the water bath around the thermocouple so that the lowest 25 mm (1 inch) is immersed in the water. Place the thermocouple meter (reference thermocouple) parallel to the sample thermocouple so that both thermocouples are at the same temperature.
3. After the reference thermocouple temperature stabilizes, enter its reading on the Calibrate Hardware dialog in the **Low Cal** field. To do so, click the **Low Cal** field, then press the space bar. Enter the value in the Calibrate dialog, then click **OK**.



4. Remove the water bath. Move the tip of the reference thermocouple so that it touches the sample thermocouple tip, yet allows the furnace to be closed.
5. Use manual control of the sample furnace to set the furnace target temperature to 1000 °C. Use a ramp rate of 50 °C/min. (Refer to **Enable Manual Control** on page 6-12.)
6. Observe the instrument schematic to ensure that both the furnace temperature and sample temperature are increasing.
7. After the furnace and sample temperature have stabilized, read the value from the reference thermocouple. Enter its reading on the Instrument Calibration dialog in the **High Cal** field. To do so, click on the **High Cal** field, then press the space bar. Enter the value in the Calibrate dialog, then click **OK**.
8. Use manual control of the furnace to return its temperature to ambient.
9. On the Instrument Calibration dialog, click **Accept** to save the new calibration values. The software then displays dialogs that allow you to verify this change and make a backup copy of the values you are replacing. Make a backup copy by clicking **Yes** then specify a file name.

## Cleaning the Analyzer

The exterior casing of the analyzer may be cleaned using a clean cloth dampened with isopropyl alcohol (IPA), a mild detergent, or a 3% hydrogen peroxide solution.



**Do not immerse the analyzer or the power cord in any liquids. Doing so could result in electrical shock to personnel or damage to the unit.**

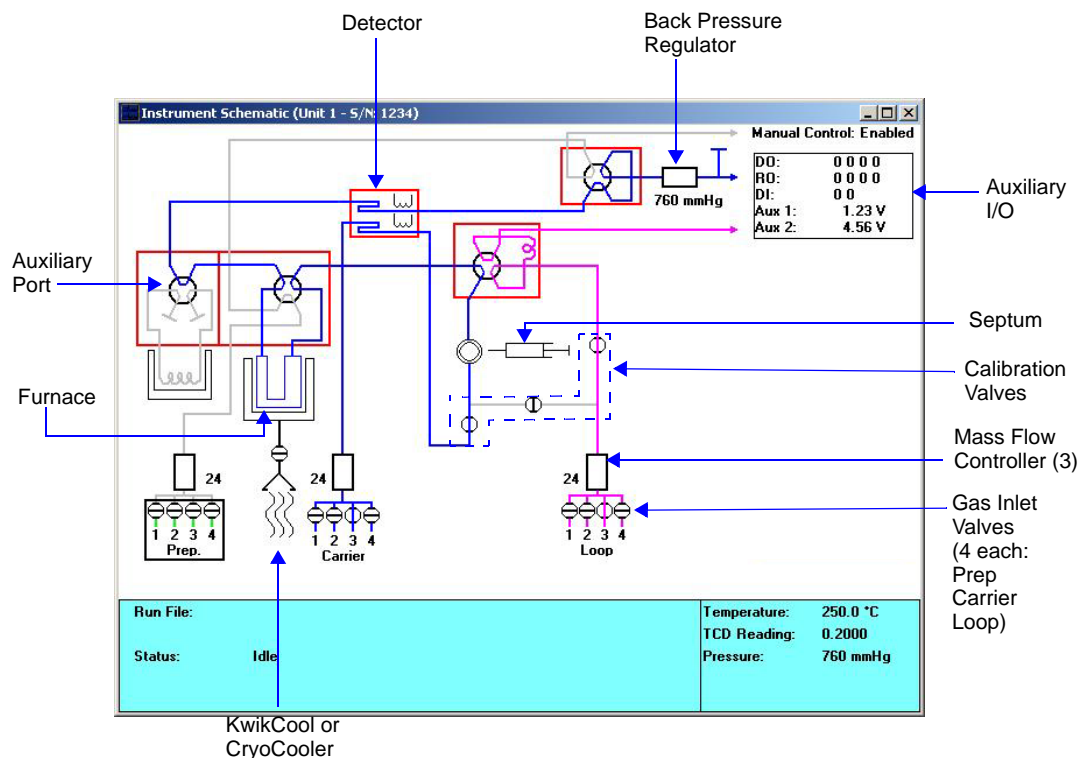


**Do not allow liquid to penetrate the casing of the analyzer. Doing so could result in damage to the unit.**

## Performing a Leak Test

### Set Up

1. Select **Unit > Enable Manual Mode**, then select **Unit > Show Instrument Schematic**.



2. Set each of the three Mass Flow Controllers to 50 cm<sup>3</sup>/min. by right-clicking, choosing **Set Flow Rate**, and entering 50 cm<sup>3</sup>/min.



**This test must be done in the order detailed in this document to test fully the AutoChem. Any flow reading other than zero indicates a leak in the gas flow path being tested.**

### Preparation Gas Path Tests

---

1. Right-click the analysis valve and choose **Analyze**.
2. Right-click the Prep. valves and select **Flow Gas [n] (He)**.
3. Plug the preparation gas exhaust outlet.
4. Observe the Preparation Gas Flow Controller reading as it slowly drops to zero.

A flow reading of zero indicates the flow path between the Preparation Gas Flow Controller and the preparation exhaust does not leak.

This tests all fitting connections in the gas flow path and part of the analysis valve.

5. Any flow reading other than zero indicates a leak in the gas flow path being tested.
6. To fix a leak that may be indicated on the Preparation Gas Flow Controller, tighten the fittings in the gas flow path and observe the reading for any decrease that would indicate that the leak has stopped.



**Any leak detected in the remaining steps of this procedure may be fixed by using the same process mentioned in Step 6.**

7. Right-click the Preparation Gas Flow Controller and choose **Stop Flow**. This will close the helium valve.
8. Remove the plug from the preparation gas exhaust outlet.

### Carrier Gas Path Tests

---

1. Right-click the Cold Trap Valve and select **Bypass**.
2. Right-click the Analysis Valve and select **Prepare**.
3. Right-click the Loop Valve and select **Fill**.
4. Turn the pressure control knob on the helium gas regulator to deliver less than 50 psi (345 kPag).
5. Right-click the Carrier Valves and choose **Flow Gas [n] (He)**.
6. Plug the analysis gas exhaust outlet.
7. Right-click on Calibration Valve B and select **Close Valve**, then repeat the step for Calibration Valve C.
8. Observe the Carrier Gas Flow Controller reading as it slowly drops to zero.

9. A flow reading of zero indicates the flow path between the Carrier Gas Flow Controller and Calibration Valve B does not leak.
10. Right-click Calibration Valve B and select **Open Valve**.
11. Observe the Carrier Gas Flow Controller reading as it slowly drops to zero or remains at zero from the previous test.

A flow reading of zero indicates the flow path between the carrier MFC and the carrier exhaust outlet does not leak.

This tests all fitting connections in the gas flow path and part of the analysis valve, part of the bypass valve, the transducer, the septum, part of the inject valve, and the analysis side of the thermal conductivity detector.

12. Right-click the Loop Valve and select **Inject**.
13. Observe the Carrier Gas Flow Controller reading as it slowly drops to zero or remains at zero from the previous test.

A flow reading of zero indicates the flow path between the Carrier Gas Flow Controller and the carrier gas exhaust outlet does not leak.

This tests all fitting connections in the gas flow path, the remaining parts of the Loop Valve, and the loop.

14. Install an empty sample tube on the sample port.
  15. Right-click the Analysis Valve and select **Analyze**.
  16. Observe the Carrier Gas Flow Controller reading as it slowly drops to zero or remains at zero from the previous test.
- A flow reading of zero indicates the flow path between the Carrier Gas Flow Controller and the carrier gas exhaust outlet does not leak.

This tests all fitting connections in the gas flow path, the sample tube, and the remaining parts of the Analysis Valve.



**The transducer, septum, inject valve, part of the analysis valve, part of the bypass valve, and thermal conductivity detector have already been proven to have no leaks.**

17. Install a cold trap (or a delay path) on the cold trap port.
18. Right-click the Cold Trap Valve and select **Trap**.
19. Observe the Carrier Gas Flow Controller reading as it slowly drops to zero or remains at zero from the previous test.

A flow reading of zero indicates the flow path between the Carrier Gas Flow Controller and the carrier gas exhaust outlet does not leak.

This tests all fitting connections in the gas flow path, the remaining parts of the Cold Trap Valve, and the cold trap.



**The transducer, septum, inject valve, analysis valve, part of the bypass valve, and thermal conductivity detector have already been proven to have no leaks.**

20. Right-click the Carrier Valves and select **Stop Flow**. This will close the helium valve.
21. Remove the plug from the analysis exhaust outlet.
22. Reset the pressure on the Helium gas regulator to its original pressure.

### Loop Gas Path Tests

1. Right-click the Loop Valve and select **Inject**.
2. Right-click Calibration Valve C and select **Close Valve**, then repeat the step for Calibration Valve D.
3. Plug the loop gas exhaust outlet.
4. Right-click the Loop Gas Valves and choose **Flow Gas [n] (He)** (or other appropriate gas) through the Loop Gas Flow Controller.
5. Observe the Loop Gas Flow Controller reading as it slowly drops to zero.

A flow reading of zero indicates the flow path between the Loop Gas Flow Controller and Calibration Valves C and D does not leak.

This tests all fitting connections in the small section of the gas flow path to Calibration Valves C and D.

6. Right-click Calibration Valve D and select **Open Valve**.
7. Observe the Loop Gas flow Controller reading as it slowly drops to zero or remains at zero from the previous test.

A flow reading of zero indicates the flow path between the Loop Gas Flow Controller and the loop gas exhaust outlet does not leak.

This tests all fitting connections in the gas flow path, Calibration Valves C and D, and the tubing to the Loop Valve.



**The flow path beyond the inject valve has already proven to have no leaks.**

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## Performing a Reference Material Analysis

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When you have doubts about the integrity of analysis data or when you would just like to ensure the highest quality data, perform a reference analysis. Reference Material Kits are available from Micromeritics (refer to Chapter 10, [ORDERING INFORMATION](#)). Each kit contains reference material and an instruction booklet. Follow the instructions in the booklet to perform the analysis and review the results.

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## Performing a Mass Flow Controller Calibration

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The AutoChem 2950 HP Analyzer uses Mass Flow Controllers (MFCs) to control the flow of gases. These MFCs require a conversion constant for each gas or gas mixture to compensate for variations in gas flows resulting from variations in the gas properties. Default MFC conversion constants are provided for a wide variety of gases in the software.

In most cases, using the default MFC conversion constant yields accurate data. A new conversion constant may be determined if:

- You are using a unique gas mixture
- The gas to use is not included in the Gas Defaults table
- You require a higher precision calibration of the MFCs for a given gas
- Unexpected analysis data lead you to believe that the default value is in error

Refer to Appendix F, [CALIBRATING THE MASS FLOW CONTROLLERS FOR EACH GAS](#), for instructions.



## Routine Maintenance

The procedures in this section are performed as needed.

### Changing Gases

#### CGA Guide for Common Gases

The tables below list the regulator CGA fittings for commonly-used gases. Make sure you use regulators with the correct CGA fittings for the gases to be attached to the analyzer. (Regulators are available from Micromeritics, refer to Chapter 10 - [ORDERING INFORMATION](#).)

##### *Pure Gases*

Gas	Formula	CGA	Notes
Ammonia	NH <sub>3</sub>	660	Stainless steel supply lines required
Argon	Ar	580	
Carbon Dioxide	CO <sub>2</sub>	320	
Carbon Monoxide	CO	350	
Helium	He	580	
Hydrogen	H <sub>2</sub>	350	
Nitric Oxide	NO	660	Stainless steel supply lines required
Nitrogen	N <sub>2</sub>	580	
Nitrous Oxide	N <sub>2</sub> O	326	Stainless steel supply lines required
Oxygen	O <sub>2</sub>	540	Verify regulator is compatible with oxygen

##### *Blended Gases*

Active Gas	Balance	CGA	Notes	Application
10% Ammonia	helium	705	Stainless steel supply lines required	TPD
10% Oxygen	helium	590		TPO
10% Oxygen	argon	590		Pulse Chemisorption

Active Gas	Balance	CGA	Notes	Application
10% Hydrogen	argon	350		TPR and Pulse Chemisorption
10% CO	helium	350		Pulse Chemisorption
10% Butadiene	helium	350		TPD and Pulse Chemisorption
30% Nitrogen	helium	580		BET Surface Area
10% Nitrous Oxide	helium	580	Stainless steel supply lines required	Pulse Chemisorption

### Guidelines for Connecting Gases to the Analyzer

Use these guidelines when installing regulators and gas lines:

- Ensure that all gases needed are available and near the instrument. If the gas bottles are not located near the instrument, you will have to use gas line extenders, which may degrade gas quality and reduce pressure.
- Carefully route the gas lines from the bottle to the analyzer, avoiding overlapping or entangling gas lines. This will ensure the correct gas pressure and make maintenance easier.
- Label both ends of the gas lines before connecting for ease in identification and maintenance.
- Ensure that all gas bottles are closed before connecting them to the analyzer.
- Make sure that no analysis is in progress (the software is idle).
- Copper tubing and brass reducer fittings are supplied with the analyzer. Some gases are incompatible with brass and copper, for example: ammonia, nitrous oxide, and nitric oxide, which require stainless steel supply lines and appropriate regulators. Stainless steel gas lines and appropriate regulators are available from Micromeritics.

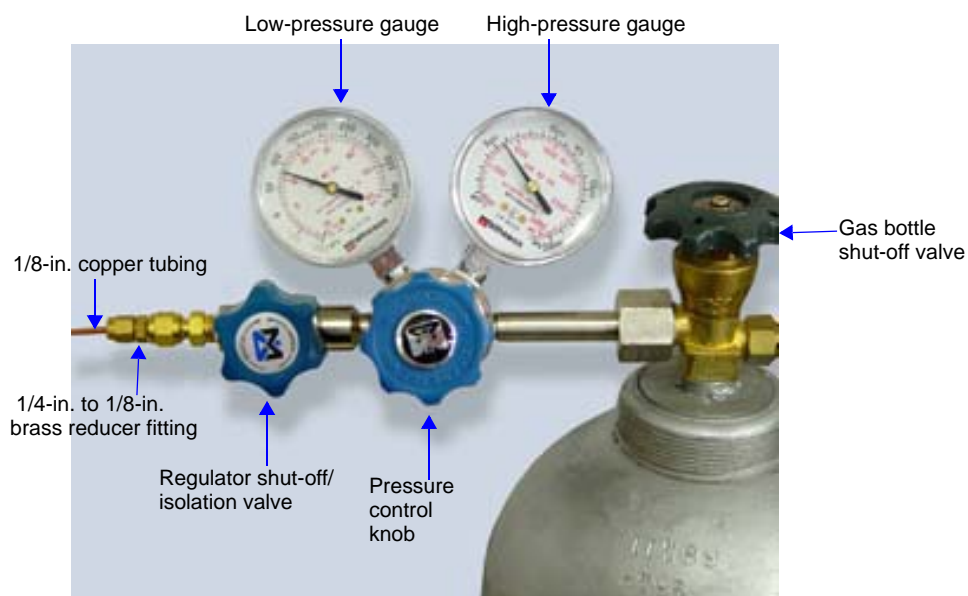
The instructions below describe a typical installation. Some configurations require additional components, such as regulator expansion kits, when one gas source will be used for several operations or when the gas bottle cannot be located close to the analyzer.

## Connecting a Gas Regulator to the Gas Supply

1. Attach an appropriate regulator to the gas supply bottle or gas line you want to connect and tighten with a wrench. Leave the gas bottle shut-off valve closed until instructed otherwise.



**If an expansion kit is required, attach it to the regulator before proceeding. (Refer to the instructions included in the expansion kit.)**



2. If the regulator has a 1/8-in. outlet, proceed to the next step. If the regulator has a 1/4-in. outlet, attach the reducer fitting to the outlet of the regulator shut-off/isolation valve and tighten the valve nut with a 9/16-in. wrench (as described in the step 3, a through d.).



**Do not overtighten the fittings. Doing so could collapse the brass fitting and cause a leak.**

3. Attach the gas inlet line to the regulator, reducer fitting, or regulator expansion a follows:
  - a. Insert the gas tubing into the fitting.
  - b. Make sure that the tubing is seated fully inside the fitting.
  - c. Tighten the nut finger-tight.
  - d. While holding the fitting body steady, tighten the nut with a wrench 3/4 turn.

4. Purge the regulator as follows:



**It is important to purge the regulator before proceeding. This will prevent contamination of the analysis gas.**

- a. Close the regulator shut-off valve by turning it fully clockwise.
- b. Turn the pressure regulator control knob fully counterclockwise.
- c. Slowly open the gas bottle valve by turning it counterclockwise, then quickly close the gas bottle valve.
- d. Observe the high pressure gauge.

If the pressure decreases, tighten the nut connecting the regulator to the gas bottle. If the pressure is stable, proceed to step e.



**When using compressed gases, make sure the gas supply equipment is adequately vented before performing the next step.**

- e. Turn the pressure regulator control knob clockwise until the low pressure gauge indicates at least 50 psig (345 kPag).
  - f. Open each regulator shut-off valve by turning it counterclockwise.
  - g. Open the gas bottle shut-off valve. Flow gas for 10 to 30 seconds, then close each valve. These actions purge the regulator and gas inlet tubing.
  - h. Ensure that the gas shut-off bottle valve is completely closed (turn clockwise).
5. Repeat steps 1 through 4 for each gas supply bottle to be attached to the analyzer.
  6. Proceed to the next section to connect the copper delivery tubing to the analyzer.

## Connecting the Gas Delivery Tubing to the Analyzer

The gas inlet ports are located on the right side panel of the analyzer. The ports are labeled **Preparation** 1 through 4, **Carrier** 1 through 4, and **Loop** 1 through 4.



To change the gas attached to one of the ports:

1. Make sure the port valve is closed (refer to **“Enable Manual Control” on page 6-24**).
2. Detach the gas line from the port.
3. Attach the new gas line to the port.
4. Set the regulator pressure using the following guidelines:
  - A pressure of 50 psig is appropriate for low pressure applications.
  - A pressure of 1050 psig is recommended for high pressure applications.
  - When high flow rates are required, you may have to increase the pressure to 30 psig.
  - If you are using a gas supply tubing that is longer than the tubing supplied with your analyzer, you may have to raise the pressure to achieve the flow rate.
5. Make sure any unused gas inlet ports are plugged.
6. Record the gas installed in the port in the analysis program as described in the next topic.

## Specifying Gas Ports

When you change the gas attached to a port on the analyzer, you must enter the change in the analysis program as follows.

1. Select **Unit > Gas Selections** to display the Gas Selections screen.

Gas Selections (Unit 1 - S/N: 256)

Preparation Gas		Carrier/Reference Gas		Loop Gas	
	MFC Constant		MFC Constant		MFC Constant
1: Argon	1.386	1: Helium	1.376	1: Argon	1.386
2: Helium	1.376	2: None	1.000	2: None	1.000
3: None	1.000	3: None	1.000	3: None	1.000
4: None	1.000	4: None	1.000	4: None	1.000

Enter the MFC gas flow conversion constant (0.010 - 2.000) for the gas.

Close

Run File:      Temperature: 250.0 °C  
Status: Idle      TCD Reading: 0.2000

This dialog contains a drop-down list of the available gases for each of the 12 available ports. Each gas also has its MFC constant listed.

Identify what gas is attached to each port by selecting that gas name from the drop-down list. If no gas is attached to a given port, select None.

The gases in the drop-down list are those specified in the Gas Defaults table (on the Options menu).

When an analysis is started, the software checks the gases specified in the sample information file against the gases identified in this dialog. If there is a discrepancy between the gas indicated in the sample information file and the gas indicated in the Gas Selections dialog, a message is displayed; analysis cannot begin until the discrepancy is resolved.



**The software is unable to determine if the wrong gas is physically connected to the port. It can only check that the gas selected agrees with the gas identified for each port in the Gas Selections dialog. It is important to be sure that the Gas Selections dialog accurately reflects which gas is physically connected to each port.**

## Venting the Gas Exhausts

Each of the exhaust ports should be safely vented. The appropriate method for venting each port depends upon the gases you are using and the experiments you are performing.



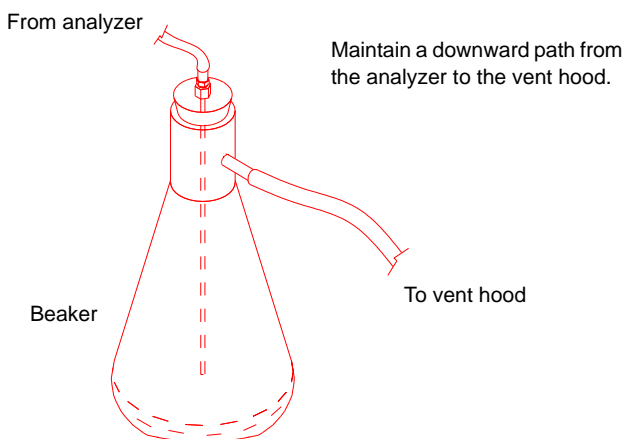
**Use extreme caution when establishing your exhaust method in order to prevent dangerous accumulations or combinations of gases, vapors, or condensibles.**

### Recommended Method

The ideal treatment for exhaust is to run the exhaust through heated extension lines to a fume hood, separate from any other exhausts. Additionally, a downward slope of the extension lines should be carefully maintained to prevent accumulation of liquids.

### Alternative Method

As an alternative to the method recommended above, vent the exhaust through a collection container to collect any condensibles before gases are vented.



Particular care should be taken if you plan to vent the exhaust from more than one port into a single collection container before diverting the gases. Make sure that you consider any hazards of combining the various exhausts in a single venting container, basing your decision on the materials being used in the experiment.

## Results of Improper Venting

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**If no venting method is provided, the vapor is discharged into the laboratory, potentially causing a hazard for laboratory personnel.**

Another possible effect of improper venting is condensation and the formation of liquid drops in the exhaust line, which could cause intermittent blockage of the line. As a result, detector signals may become erratic. This is visible in the data as smaller-than-expected peaks, missing peaks, or even larger peaks, depending upon the timing of the drop blocking the exhaust.

## Purging the System

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### Purging Air

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The instrument must be purged of air after you change the instrument hardware. For example, you must purge the instrument and gas supply lines after changing a gas bottle (that line only must be purged).

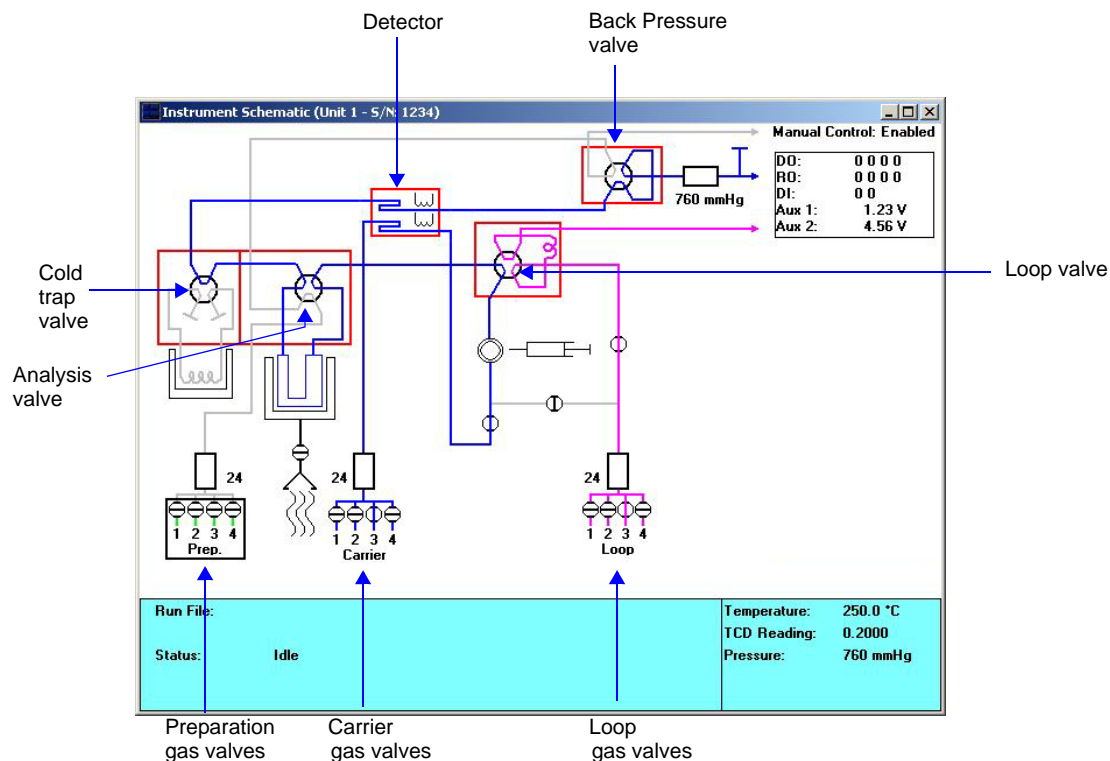


**Purge one line at a time. It is the operator's responsibility to make sure that dangerous combinations of gases are not created while using Manual Control. It may be necessary to flow an inert gas through the system between flows of incompatible gases.**

Purge the analyzer's internal gas lines of air as follows:

1. Select **Unit > Show Instrument Schematic**, then **Enable Manual Control**. (Refer to **Enable Manual Control** on page [6-12](#) for detailed information on manually controlling the system.) The following instructions are sufficient for you to purge the instrument.





2. Set the Cold Trap valve, Analysis valve, Loop valve, and Detector heat zone temperatures to 110 °C.
  - a. Position the cursor over the valve's heat zone (the cursor will change to a thermometer) and right-click.
  - b. Select **Set temperatures**, then enter 110.
  - c. Click **OK**.
3. Wait until the temperature reaches the 110 °C target. (Click on the heat zone to display both the target and current temperatures in the box on the right side of the schematic.)



**When you have clicked on the heat zone, the cursor appears as a thermometer. If you have accidentally clicked on the valve, move the cursor slightly and click again.**

4. First, purge all air from the preparation gas line.
  - a. Position the cursor over the preparation gas Mass Flow Controller (MFC) and right-click.
  - b. Select **Set flow rate** and enter 50.
  - c. Click **OK**.

- d. Position the cursor over the four preparation gas valves and right-click.
  - e. Open the valve that corresponds to the gas to flow by clicking the appropriate **Flow Gas [n]** menu choice.
  - f. Allow the gas to flow for approximately 20 minutes or until you are certain the lines have been purged of air.
5. Repeat step 4 for each valve to which a gas bottle is connected.
  6. Repeat the process (steps 4 and 5) for the carrier and loop gas flows.

### **Purging Gases**

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When you change the gas that is flowing through the instrument, or when you change the type of gas connected to a port, you should purge the instrument of the previous gas by flowing the new gas.

Use the instructions for **Purging Air from the System** to purge the previous gas. Be sure to change the Gas Selections dialog to reflect the change in gases. (See **Specifying Gases** on page [“Specifying Gas Ports” on page 9-36](#) for more information.)

### **Removing or Installing the Furnace**

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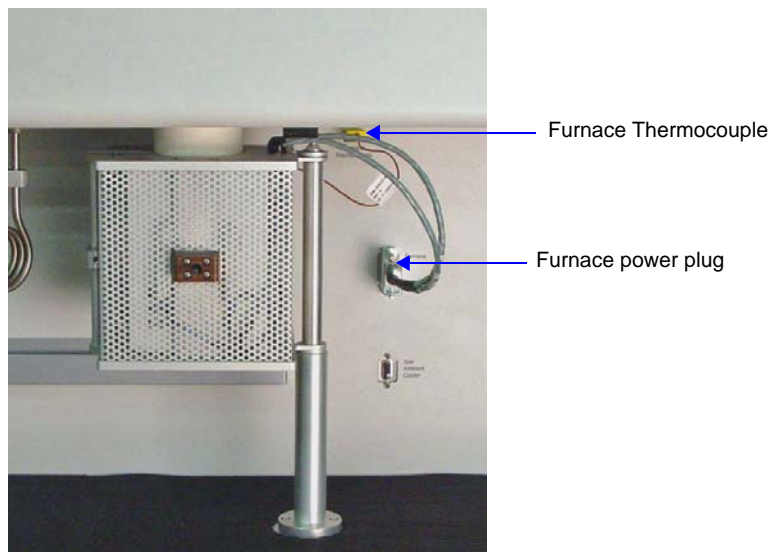
Initially, the furnace will be installed on the analyzer. However, it should be removed when performing B.E.T. analyses to allow placement of a cold bath Dewar around the sample. It can then be reinstalled to perform other types of analyses.

#### **Removing the Furnace**

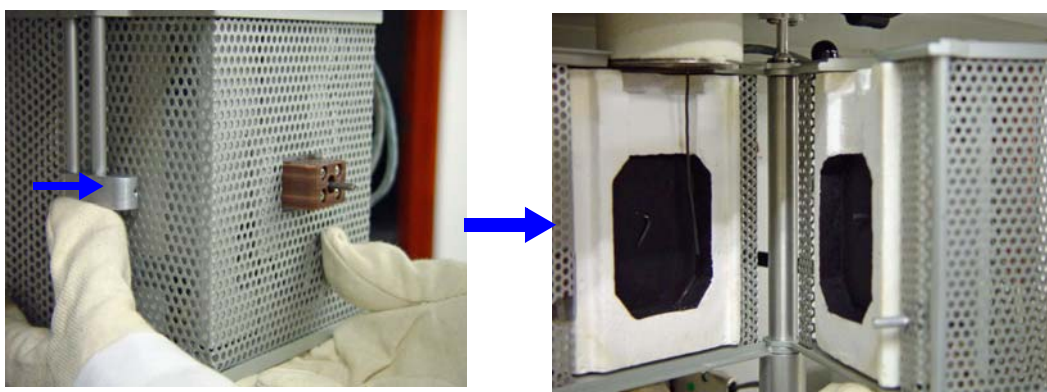


**If the analyzer has recently been used, allow the furnace to cool to room temperature before performing this procedure. We recommend that you wear the cotton gloves provided with the instrument when handling the furnace.**

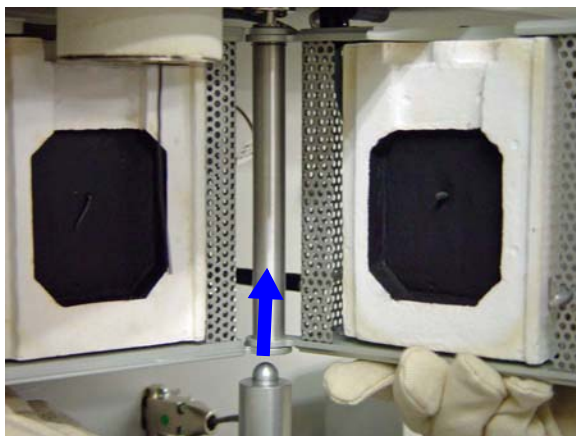
1. Unplug the furnace power plug and thermocouple from the front of the analyzer.



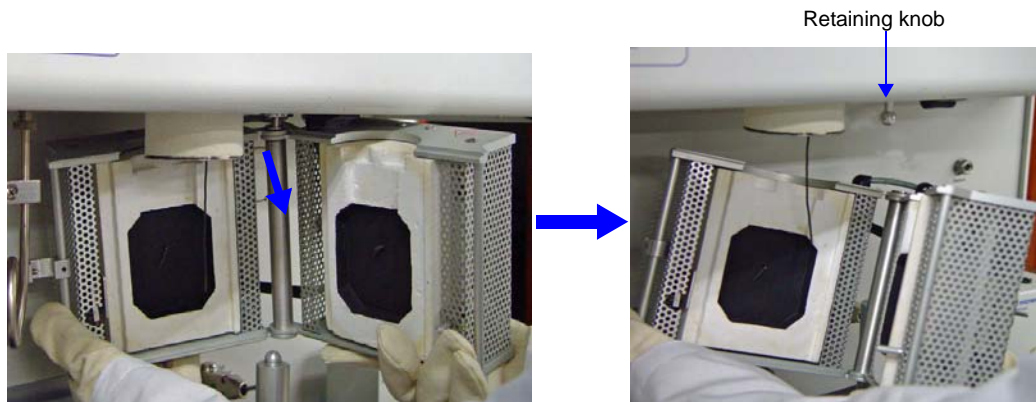
2. Open the latch, then open the furnace.



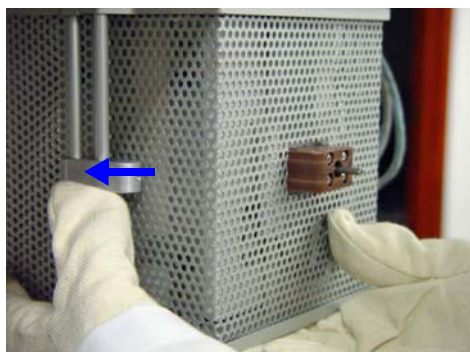
3. Gently lift the right side of the furnace until it is freed from the support post.



4. Lower the furnace, moving it away from the support post until it is free of the retaining knob.

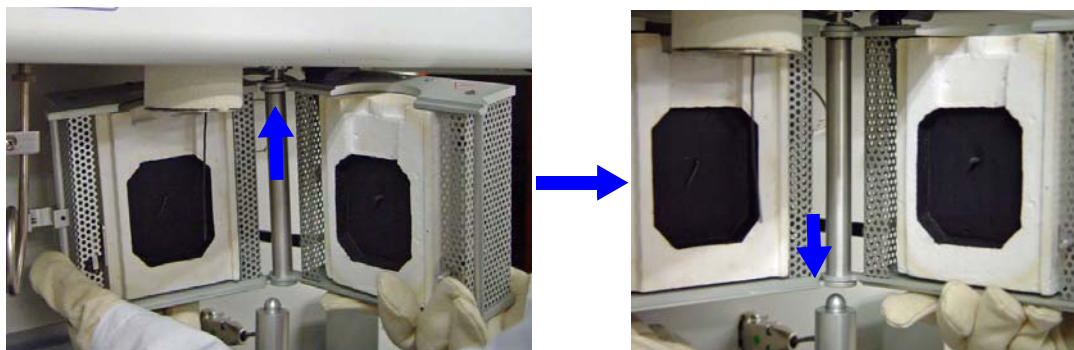


5. Close the furnace and secure the furnace latch. Place the furnace in a location where it will be protected from damage.

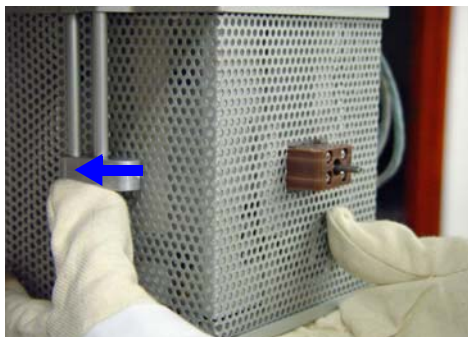


### **Installing the Furnace**

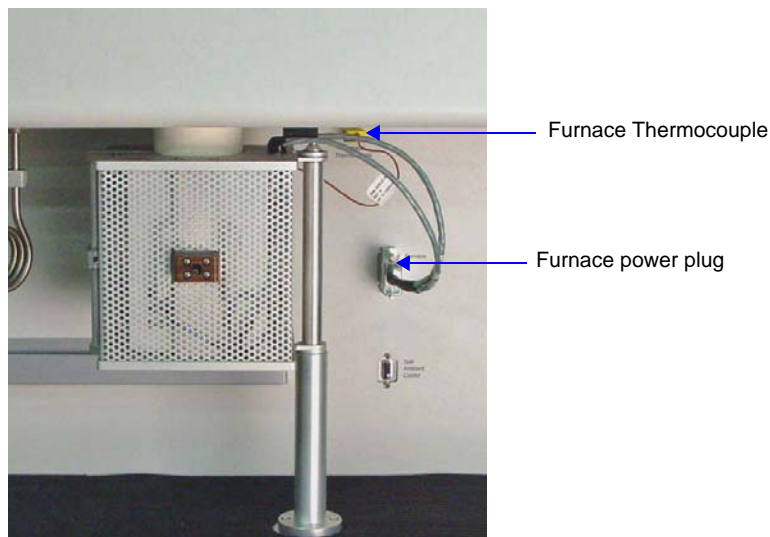
1. Hold the open furnace next to the support post. Raise the furnace, moving it above the support post until the retaining knob is inserted in the hole in the top of the furnace.



2. When the retaining socket on the bottom of the furnace is located above the support post, gently lower the furnace until it rests securely on the post.
3. When you are ready to begin the experiment, close the furnace around the sample tube and secure the latch.



4. Plug the thermocouple cable and the furnace power cable into the appropriate connectors on the analyzer.





## Changing the Injection Loop

The AutoChem 2950 HP is shipped with three loops of different sizes. If you need to change to a loop, follow the instructions below.

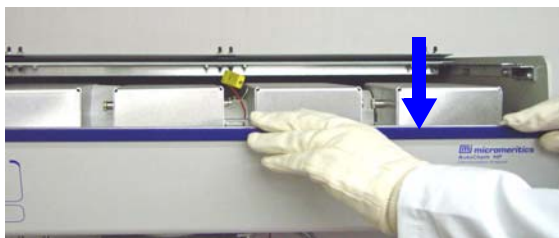
1. Turn the analyzer's power switch off, then disconnect the analyzer from its power source.
2. Press in on the right side of the retractable handle on the top of the analyzer to protrude the handle.



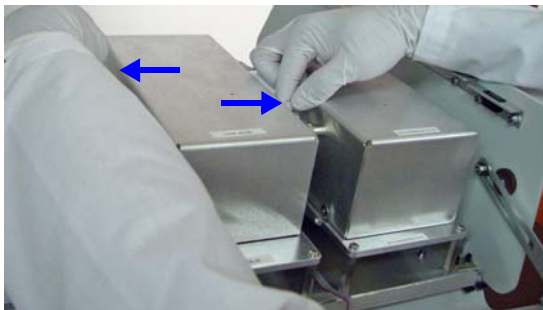
3. Using the handle, open the top panel. The top panel is hinged to allow easy access to its components; it is not necessary to remove the top panel.



4. Pull down the upper front panel.



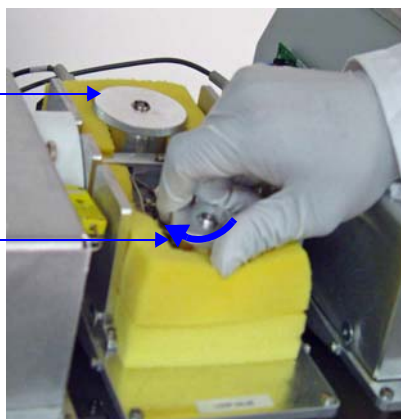
5. The Loop valve is located in the second block from the right. The cover is labeled **Loop Valve**. The cover is held in place by two retaining plungers. Remove the valve cover by pulling the plungers outward until the cover is released. Pull the cover up and out.



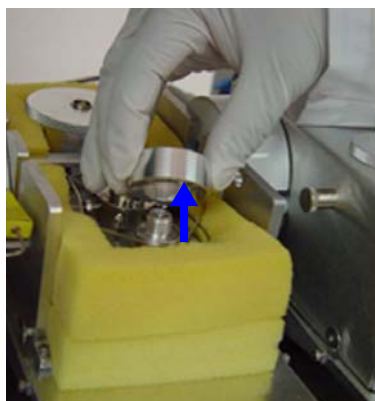
6. Remove the insulation from the Loop valve.
7. Unscrew the loop nut and lay it aside. It may be necessary to loosen the screw in the middle of the assembly in order to free the loop nut.

The 5.0-cm<sup>3</sup> loop is located in this position when installed

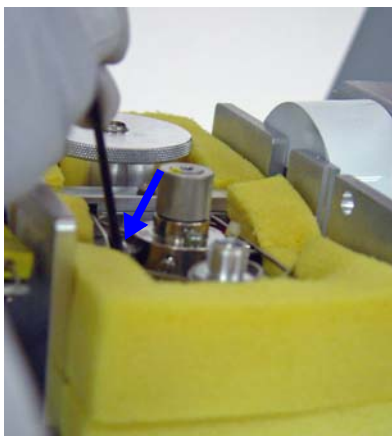
Both the 0.5-cm<sup>3</sup> and the 1.0-cm<sup>3</sup> loops are located in this position when installed



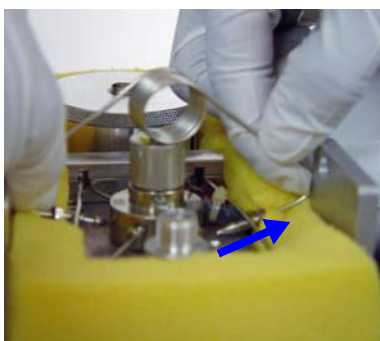
8. Remove the enclosure ring.



9. Use a 1/4-in. open-end wrench to loosen the nuts on the injection loop.



10. Pull up and remove the injection loop and enclosure ring from the mounting plate.

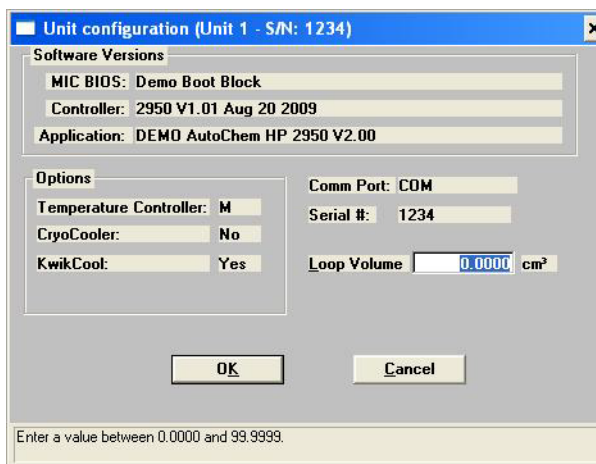


**Make sure the body of the loop is positioned in the temperature-controlled enclosure of the loop valve. This orientation keeps the temperature (and volume) of the loop constant. Failure to keep the full loop at a constant temperature may cause variations in the quantity of gas contained in the loop.**

11. Install the replacement injection loop by placing the ends of the loop into the sides of the valve and turning the nuts finger-tight.
12. Push the replacement loop down to contact the mounting plate and place the loop enclosure ring firmly into place.
13. Screw the loop nut down finger-tight. Tighten the screw in middle of the assembly if it was loosened earlier.
14. Tighten the loop nuts securely, using a 1/4-in. open-end wrench.
15. Check the injection loop for leaks. Refer to **“Performing a Leak Test” on page 9-26**. If you detect a leak, the injection loop is probably not installed properly. Repeat the installation and check for leaks again.
16. After you have checked for leaks, move the insulation back into position and reinstall the Fill/Inject valve cover.



17. Close the top panel and the lower front panel.
18. After installing a new injection loop, it is necessary to calibrate the loop. Follow the instructions for performing a loop calibration in Appendix J.
19. Select **Unit > Unit Configuration** in the 2950 software to display the Unit Configuration dialog.



The image shows a software dialog box titled "Unit configuration (Unit 1 - S/N: 1234)". It contains two main sections: "Software Versions" and "Options".

**Software Versions:**

- MIC BIOS: Demo Boot Block
- Controller: 2950 V1.01 Aug 20 2009
- Application: DEMO AutoChem HP 2950 V2.00

**Options:**

- Temperature Controller: M
- CryoCooler: No
- KwikCool: Yes
- Comm Port: COM
- Serial #: 1234
- Loop Volume: 0.0000 cm<sup>3</sup>

At the bottom are "OK" and "Cancel" buttons. A status bar at the very bottom reads: "Enter a value between 0.0000 and 99.9999."

20. Enter the loop volume in the **Loop Volume** field.

## Attaching the KwikCool or Optional CryoCooler

You can attach a KwikCool (shipped with the unit) or a CryoCooler (optional accessory) to the analyzer to cool the furnace more rapidly after analysis. After you install one of these units, you should exit, then restart the AutoChem 2950 software so that the software will recognize the added hardware and display it in the instrument schematic.



### Safe Operation Guidelines for Stainless Steel Tubes

- External cooling of the tube above 200 °C may be assisted only with air not to exceed 3 cfm @ 30 psi.
- External cooling of the tube below 200 °C may be assisted with the CryoCooler at a rate less than 90 °C/min.
- Do not use other cooling agents below 200 °C because they may embrittle the sample tube.
- Do not submerge the tube in water or oil, or spray the tube with water or oil.

Failure to follow these practices may cause bodily harm and/or damage to the instrument and sample tube. The AutoChem HP has been optimized to use these guidelines for safe operation.

## KwikCool

A KwikCool unit is shipped with every AutoChem II 2950. This unit allows you to cool the furnace quickly between analyses.

### Installation

1. Turn off the analyzer.
2. Attach the cable of the KwikCool unit to the connector labeled **Sub Ambient Cooler** on the front panel of the analyzer.
3. Attach the tubing provided to the inlet on the front of the furnace.
4. Attach a supply of dry house air (or nitrogen) to the gas inlet on the front of the KwikCool; the pressure is indicated next to the inlet.
5. Fill a Dewar with an ice water bath.
6. Place the KwikCool unit on top of the Dewar.



7. Turn on the analyzer and start the analysis program.

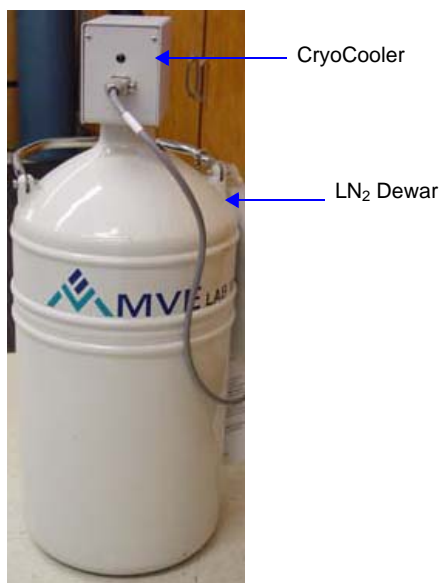
### Operation

When you create a sample file, a space is provided (on the Temperature Ramp dialog) for you to indicate that you are using the KwikCool. Refer to [“Creating Sample Information Files” on page 3-11](#).

## CryoCooler

The CryoCooler is an optional device that enables you to start an analysis at sub ambient temperatures. The CryoCooler cools the sample quickly and holds the temperature at a user-specified value between +20 and -100 °C.

The CryoCooler unit consists of a cryogenic pump mechanism, a storage Dewar for liquid nitrogen (LN<sub>2</sub>), and a delivery hose that attaches to the AutoChem furnace.



Stable subambient temperatures are achieved by pumping LN<sub>2</sub> into the analyzer's furnace cavity, which contains the sample.



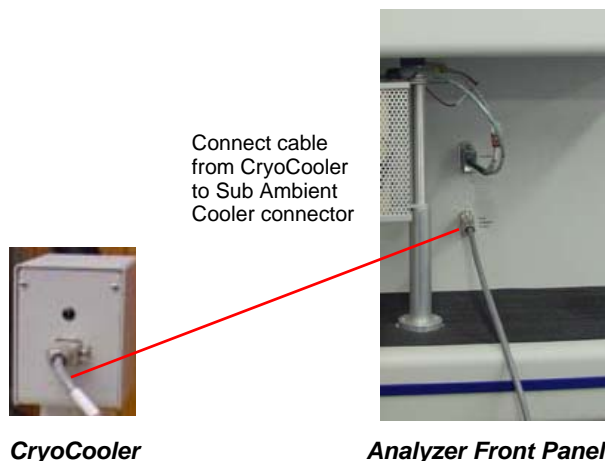
**Always wear a safety face shield or goggles and gloves when handling liquid nitrogen to prevent the possibility of splashing liquid nitrogen into your eyes or onto your skin.**



**The CryoCooler must be free of internal moisture before insertion into LN<sub>2</sub>. If present, moisture will freeze and prevent operation. The system was free of moisture when it left the factory; however, dry air, nitrogen, or other inert gas should be passed through the delivery hose to be sure it has remained dry.**

## Installation

1. Attach one end of the cable (provided with the CryoCooler) to the connector labeled **Sub Ambient Cooler** on the front panel of the analyzer. Attach the other end of the cable to the connector on the CryoCooler.



2. To verify the CryoCooler is operating properly before inserting it into LN<sub>2</sub>, enable manual control, right-click on the CryoCooler icon, and set the speed to 100%. The green light on the housing should come on and the rotor on the pump should be visibly turning. Turn the CryoCooler off using manual control, but leave the cable connected.
3. Fill the Dewar approximately two-thirds full with LN<sub>2</sub>.
4. Insert the CryoCooler into the Dewar. Boiling will result as the pump mechanism contacts the LN<sub>2</sub> so insert the CryoCooler slowly over a period of several minutes. This permits most of the resulting vapor to escape before the CryoCooler is finally centered on the Dewar opening.



**Insert the CryoCooler into the LN<sub>2</sub> slowly. Inserting it too quickly will cause the LN<sub>2</sub> to boil-off and possibly splash onto your hands.**

5. Place the Dewar on the floor near the front of the analyzer or beside the analyzer on its workbench.

6. Attach the delivery hose from the CryoCooler to the furnace inlet tube.



7. Secure the delivery hose by tightening the thumbwheel on the furnace connection.
8. Allow five minutes for thermal expansion and contraction effects to dissipate, then start the AutoChem analysis.

If the CryoCooler fails to respond immediately, it may be due to components having not yet thermally adjusted to their new temperature surroundings or to ice formation from previously condensed moisture. Wait a few minutes and try again. Failure this time is almost certainly due to ice formation.

## Routine Maintenance

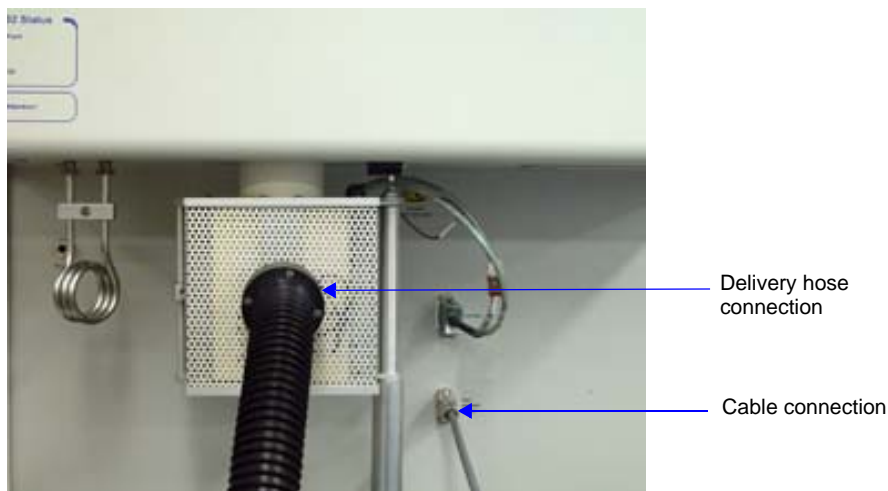
---

The buildup of ice and frost particles is likely in any device which uses liquid nitrogen in a laboratory environment. Repeated filling and evaporation alone can lead to an accumulation of ice crystals within the Dewar. The CryoCooler has been carefully designed to minimize problems from ice. Ice problems can be avoided almost entirely if, on a weekly basis, you empty the Dewar and thoroughly dry its interior.

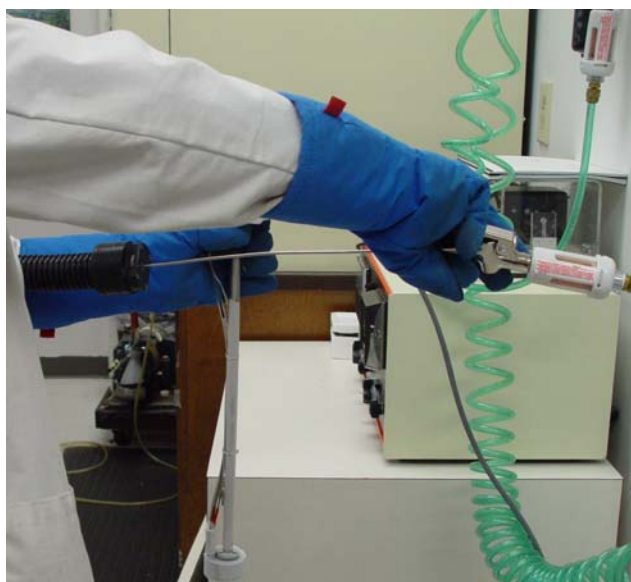
## Recovering from an Iced Condition

If ice buildup should occur in the CryoCooler, a thermal overload switch activates and cuts power to the pump motor. The green light will also be off in this condition. Perform the following steps to remove ice buildup:

1. Remove the CryoCooler from the Dewar.
2. Unplug the cable from the **Sub Ambient Cooler** connector on the front of the instrument to allow a few minutes for the thermal protector to reset itself.
3. Turn the thumbwheel on the delivery hose connection clockwise to loosen, then disconnect the hose from the analyzer.



4. Blow dry air or nitrogen into the hose and out of the pump mechanism, holding the CryoCooler nearly vertical.



5. Also blow into the pump inlet and into one of the holes of the member connecting the housing and the pump just above its connection point to the pump.
6. It is permissible to heat the pump mechanism with a laboratory heat gun until it is too warm to hold. When doing so, be careful not to heat the plastic delivery hose directly; it will melt if overheated.



**Do not apply heat directly to the delivery hose. Doing so may melt the plastic.**

7. Slowly insert the CryoCooler back into the Dewar and proceed with the sample analysis.



**The CryoCooler reservoir retains LN<sub>2</sub> for several days when not used. If its use is temporarily suspended, leave it plugged into the instrument but disconnected from the furnace inlet. An internal heater produces a slight outflow of gaseous nitrogen which prevents ice buildup in the delivery hose.**

### Replenishing the Dewar

You can replenish the Dewar without completely removing the CryoCooler in either of two ways:

- **By adding liquid nitrogen to the Dewar.**

Lift the CryoCooler a few inches above the neck of the Dewar, allowing it to rest on the rim of the neck. Then insert the nozzle of an LN<sub>2</sub> transfer system and refill the Dewar. The Model 021 LN<sub>2</sub> transfer system is available from Micromeritics (refer to **“ORDERING INFORMATION” on page 10-1**).



LN<sub>2</sub> Transfer  
Hose



**Insert the CryoCooler into the LN<sub>2</sub> Dewar slowly. Inserting it too quickly will cause the LN<sub>2</sub> to boil-off and possibly splash onto your hands.**

- **By attaching the CryoCooler to another Dewar.**

Remove the CryoCooler from the nearly empty Dewar and immediately insert it into another full Dewar.



## 10. ORDERING INFORMATION

The AutoChem 2950 HP Analyzer components and accessories can be ordered by:

- Telephone: 770/662-3636
- Email: customer.service@micromeritics.com
- Internet: www.micromeritics.com

When ordering, please use the information provided below.

### Analysis System Components and Accessories

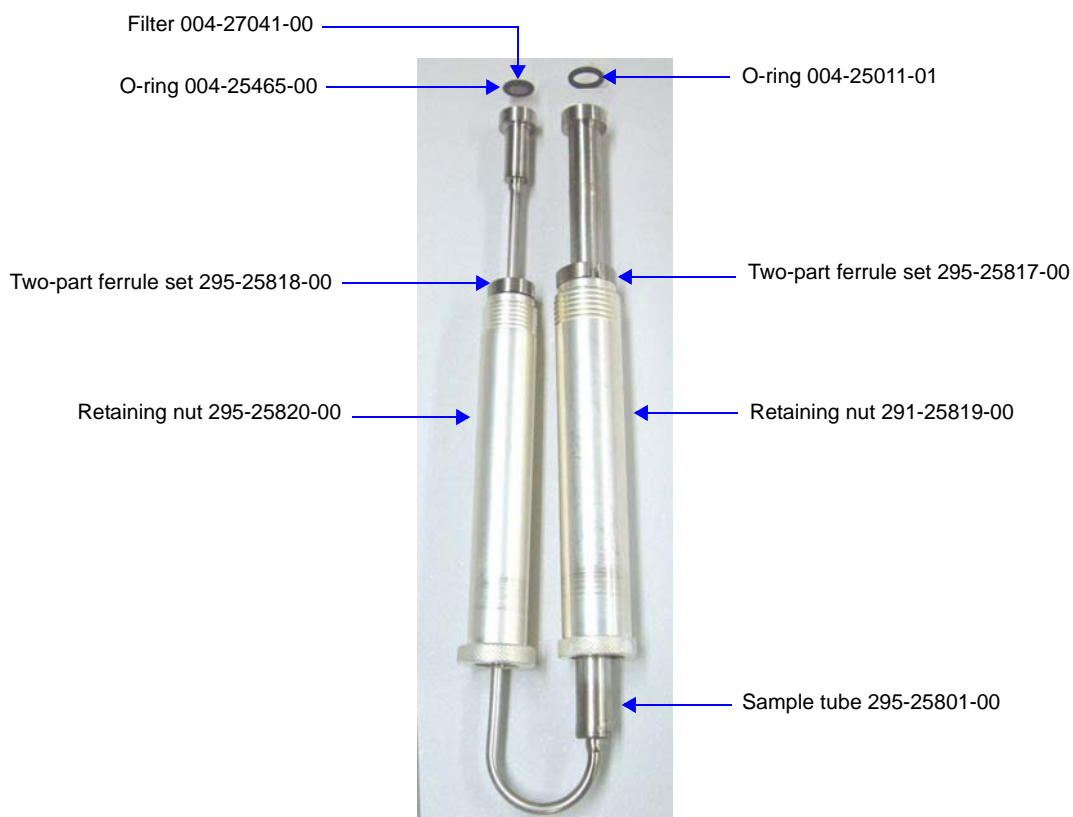
Part Number	Item and Description
295-42803-00	Operator's Manual, AutoChem 2950 HP Analyzer
008-42001-00	<i>Analytical Methods in Fine Particle Technology</i> , a comprehensive resource of information on particle analysis. This hardbound text describes theory, instrumentation, and applications pertaining to chemisorption and temperature-programmed reactions, as well as other analytical methods used in the characterization of catalysts and other materials.
291-34005-00	KwikCool; provides rapid cooling of the furnace, decreasing analysis time and increasing throughput.
029-00000-00	CryoCooler; allows furnace system to hold sample at approximately -120 °C, then ramp upwards. Includes bench top Dewar.
021-00000-00	Model 021 LN <sub>2</sub> transfer system, for easy filling of sample Dewars; includes 47-L Dewar, mobile platform, and pump with 100-cm discharge line
295-25810-00	Cold trap assembly
290-25846-00	Gas inlet line assembly; includes copper tubing, with ferrules and nuts (1/8-in. male fitting to connect to analyzer on one end, 1/8-in. female fitting to connect to regulator on opposite end)
290-25846-01	Same as 290-25846-00, but uses 5m 16-ft. tubing.
201-25818-00	Same as 290-25846-00, but uses stainless steel tubing.
201-25818-01	Same as 290-25846-00, but uses stainless steel 5m 16-ft. tubing.
930-54603-00	Brush, narrow; for cleaning sample tubes
004-54609-00	Brush, wide; for cleaning sample tubes
004-54618-00	Tool, for removing sample port O-ring

Part Number	Item and Description
291-31801-00	Cover, half, for cold trap Dewar. (Two needed per Dewar.)
004-61064-00	Dewar, 600 cm <sup>3</sup>
004-62102-00	Rotor for 6-port rotary valves
004-25940-00	Ferrule, one piece, 1/8th inch PEEK, for the quartz thermocouple sheath, which mounts through a 1/8th inch fitting
004-25938-00	Ferrule, one piece, 1/16-in. ID, for mounting the sample thermocouple in the same 1/8-in. fitting as the quartz thermocouple sheath
290-29800-00	Flowmeter, bubble, 10 cm <sup>3</sup>
004-16247-00	Fluid, gas bubble, for Flowmeter
004-32187-00	Gloves, cotton flannel, pair
290-62600-00	Injection loop, 0.5 cm <sup>3</sup> nominal, stainless steel
290-62601-00	Injection loop, 1.0 cm <sup>3</sup> nominal, stainless steel
290-62602-00	Injection loop, 5.0 cm <sup>3</sup> nominal, silver-solder joints in loop, stainless steel
004-32164-01	Quartz Wool, 4-in. x 6-in. bag
004-25549-00	Reducer, 1/8 in. tube x 1/4 in. tube, for regulators
004-16836-00	Reference Material, silver oxide
290-25842-00	Septum, external gas-sampling assembly
280-32800-00	Support, sample weighing
004-61602-00	Syringe, 1 cm <sup>3</sup>
004-61604-00	Syringe, 10 cm <sup>3</sup>
004-61606-00	Syringe, 0.1 cm <sup>3</sup>
004-61604-01	Spare needle, for syringe, pkg of 3
291-53904-00	Thermocouple, sample, pre-shaped for analyzer
292-32801-00	Protective shelf mat
004-62150-326	Gas regulator, CGA 326 fitting, 1500 psig (N <sub>2</sub> O)
004-62150-590	Gas regulator, CGA 590 fitting, 1500 psig (10% O <sub>2</sub> in He)
004-62150-540	Gas regulator, CGA 540 fitting, 1500 psig (O <sub>2</sub> )

Part Number	Item and Description
004-62150-350	Gas regulator, CGA 350 fitting, 1500 psig (CO or H <sub>2</sub> or 10% CO or 10% H <sub>2</sub> )
004-62150-580	Gas regulator, CGA 580 fitting, 1500 psig (Ar, He, or N <sub>2</sub> )
004-62230-32	Gas regulator, 2-stage, CGA 320 fitting, 30 psig (CO <sub>2</sub> )
004-62230-35	Gas regulator, 2-stage, CGA 350 fitting, 30 psig (CO, H <sub>2</sub> )
004-62230-54	Gas regulator, 2-stage, CGA 540 fitting, 30 psig (O <sub>2</sub> )
004-62230-58	Gas regulator, 2-stage, CGA 580 fitting, 30 psig (N <sub>2</sub> , He, Ar, Kr)
004-62230-59	Gas regulator, 2-stage, CGA 590 fitting, 30 psig (ultrapure air)
004-62200-58	Gas regulator, 2-stage, CGA 580 fitting, 100 psig (N <sub>2</sub> , He, Ar, Kr)
004-33601-00	Expansion Kit; adds an additional outlet to the gas regulator (includes fittings and instructions), maximum 1000 psig
004-32601-01	Expansion Kit; adds three additional outlets to the gas regulator (includes fittings and instructions), maximum 1000 psig
004-32601-02	Expansion Kit; adds three additional outlets to the gas regulator (includes fittings and instructions), maximum 3500 psig
004-54805-00	Extraction Tool, for inserting quartz wool into or for removing from sample tubes
292-33011-00	Mass Spectrometer, 100 AMU
292-33012-00	Mass Spectrometer, 200 AMU
292-33013-00	Mass Spectrometer, 300 AMU

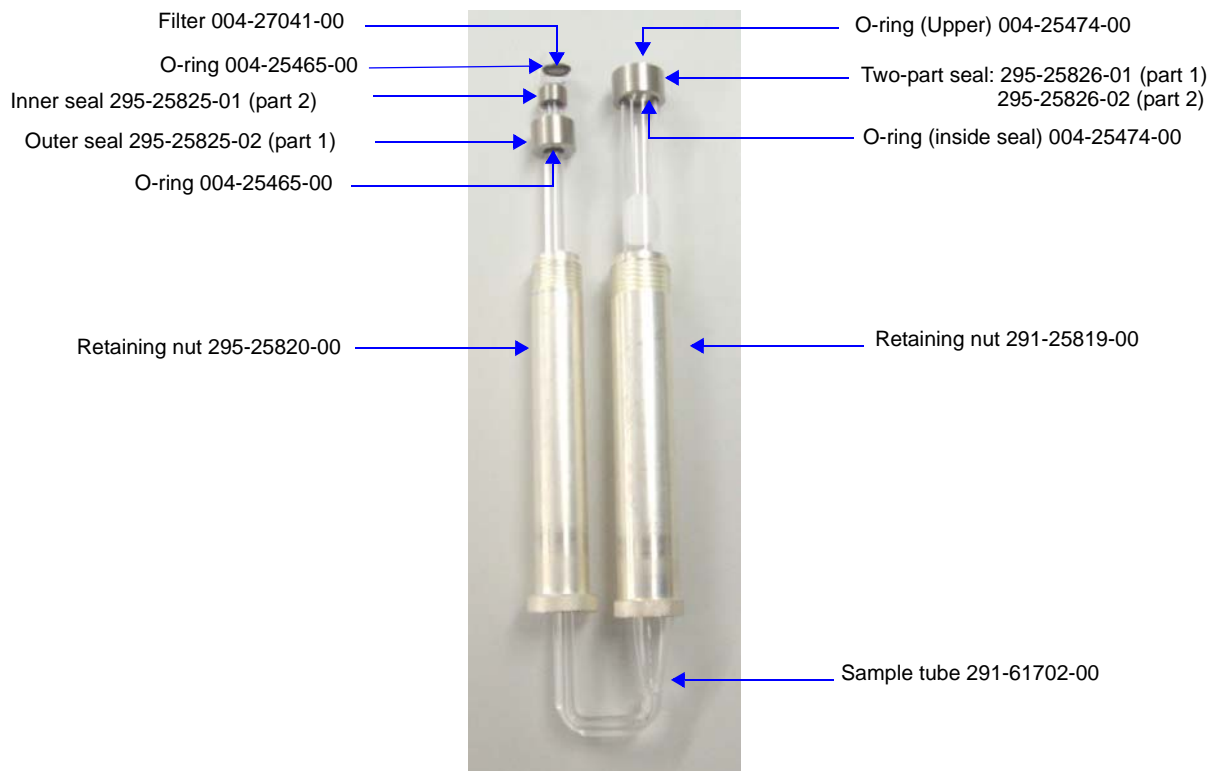
## Sample Tubes and Components

### Metal Tubes



Part Number	Item and Description
295-25801-00	Sample tube, stainless steel
004-27041-00	Filter, for metal sample tube, small stem, 20 micron, 1/4 D x 1/16 T
004-25465-00	O-ring, for metal sample tube, small stem, 010, Kalrez
295-25818-00	Two-part ferrule set, for metal sample tube, small stem
295-25820-00	Retaining nut, for metal sample tube, small stem
004-25011-01	O-ring, for metal sample tube, large stem, 011, Kalrez
295-25817-00	Two-part ferrule set, for metal sample tube, large stem
291-25819-00	Retaining nut, for metal sample tube, large stem

## Quartz Tubes



Part Number	Item and Description
291-61702-00	Sample tube, quartz
004-27041-00	Filter, for quartz sample tube, small stem, 20 micron, 1/4 D x 1/16 T
004-25465-00	O-ring, for quartz sample tube seal, small stem, 010 Kalrez
295-25825-01	Inner sample tube seal, for quartz sample tube, small stem, part 2
295-25825-02	Outer sample tube seal, for quartz sample tube, small stem, part 1
295-25820-00	Retaining nut, for quartz sample tube, small stem
004-25474-00	O-ring, for metal sample tube, large stem, 013 Kalrez
295-25826-01	Two-part sample tube seal, for quartz sample tube, large stem, part 1
295-25826-02	Two-part sample tube seal, for quartz sample tube, large stem, part 2
291-25819-00	Retaining nut, for quartz sample tube, large stem
004-32004-00	Stopper for quartz sample tube inlet (not shown)



## A. FORMS



### Determining the Sample Mass

---

Complete instructions for determining the mass of your sample and loading it into the sample tube are located in **“Determining the Mass of the Sample” on page 3-25**. This worksheet provides a format for recording the values necessary to calculate the sample’s mass. A space is also provided for recording the sample mass after analysis.

Some operators prefer to use the sample’s mass after the analysis; this is particularly desirable when a significant amount of sample preparation occurs in situ during the analysis. If you want the after-analysis value for the mass to be used in calculations, determine the after analysis sample mass, then open the sample information file and enter the after analysis mass in the Sample mass field before editing or reporting data for the analysis.

In some cases, the difference in the sample’s mass recorded after the analysis versus that recorded before the analysis may be significant in assessing the results.

**Sample identification:** \_\_\_\_\_

- |   |   |       |
|---|---|-------|
| 1 | Empty sample tube mass (including quartz wool and stopper): | _____ |
| 2 | Sample tube plus sample mass:                               | _____ |
| 3 | Sample mass (Step 2 - Step 1):                              | _____ |
| 4 | Sample tube plus sample mass after analysis (optional):     | _____ |
| 5 | Sample mass after analysis (Step 4 - Step 1) (optional)     | _____ |



## Manual Injections

When a series of manual injections is performed, it may be useful to list the planned injection volumes, then check each one off immediately after it is performed.

Injection Volume	Check when completed	Comments
1.		
2.		
3.		
4.		
5.		
6.		
7.		
8.		
9.		
10.		
11.		
12.		
13.		
14.		
15.		
16.		
17.		
18.		
19.		
20.		





## Current Gas Connections

---

Use this form to record the gases currently connected to the analyzer. Post the completed form on or near the analyzer.

This form may help you make sure that the correct gas for each port is identified in the software. Complete the form, then open the Gas Selections dialog from the Unit menu. Verify that the correct gas is selected for each port.

Unit number (S/N): \_\_\_\_\_

Date: \_\_\_\_\_

Operator: \_\_\_\_\_

Preparation Gas	Carrier	Loop Gas
1	1	1
2	2	2
3	3	3
4	4	4



## B. CALCULATIONS

### Volume at STP

---

Volume at standard reduction of gas data to Standard Temperature Pressure (STP) conditions (273.15K and 760 mmHg).

$$V_{STP} = V_a \left( \frac{273.15}{273.15 + T_a} \right) \times \left( \frac{P_a}{760 \text{ mmHg}} \right)$$

VSTP	=	Volume at STP, cm <sup>3</sup>
Va	=	Volume at ambient temperature and pressure, cm <sup>3</sup>
Ta	=	Ambient temperature, °C
Pa	=	Ambient pressure, mmHg

### Injection Loop Calibration

---

These equations are used during calibration of a dose loop

$$V_{S_{STP}} = V_s \left( \frac{273.15}{273.15 + T_a} \right) \times \left( \frac{P_a}{760 \text{ mmHg}} \right)$$

$$V_l = \left( \frac{A_l \times V_{S_{STP}}}{A_s} \right)$$

VSSTP	=	Volume of syringe at STP, cm <sup>3</sup>
VS	=	Physical volume of syringe, cm <sup>3</sup>
Ta	=	Ambient temperature, °C
Pa	=	Ambient pressure, mmHg
Vl	=	Loop volume (at loop temperature), cm <sup>3</sup>
Al	=	Average peak area of loop injections
AS	=	Average peak area of syringe injections

## Injection Loop Volume – Temperature Change

This equation is used to correct the apparent loop volume when the temperature of the loop heat zone is changed.

$$V_n = V_o \left( \frac{T_o + 273.15}{T_n + 273.15} \right)$$

V <sub>n</sub>	=	New Effective Loop Volume, cm <sup>3</sup>
V <sub>o</sub>	=	Old Loop Volume, cm <sup>3</sup>
T <sub>o</sub>	=	Old Loop Temperature, °C
T <sub>n</sub>	=	New Loop Temperature, °C

## Calibration Error (Goodness of Fit)

This equation is a means to determine whether the calibration curve is acceptable.

$$E_{\%} = \sum_{i=0}^N \frac{(Q_{m_i} - Q_{c_i})}{(N-1)}$$

E <sub>%</sub>	=	Percent error
Q <sub>m</sub>	=	Quantity measured
Q <sub>e</sub>	=	Quantity calculated
N	=	Number of measurements

## Percent Dispersion

This equation is used in a Pulse Chemisorption analysis to calculate the percent dispersion. Some understanding of the chemistry is required to select the proper stoichiometric factor and gram molecular weight.

$$PD = 100 \left( \frac{V_s \times SF_{calc}}{SW \times 22414} \right) GMW_{calc}$$

PD	=	Percent dispersion
V <sub>s</sub>	=	Volume sorbed (cm <sup>3</sup> at STP)
SF <sub>calc</sub>	=	Calculated stoichiometry factor
SW	=	Sample weight (g)
GMW <sub>calc</sub>	=	Gram Molecular Weight (g/g-mole)

## BET Surface Area

For each included point  $i$ , relative pressure  $P/P_{o_i}$  and specific volume adsorbed  $V_{STP_i}$  are calculated for a single point BET. Then the BET transformation  $B_i$  is calculated:

$$B_i = \frac{\frac{P}{P_{o_i}}}{\left(1 - \frac{P}{P_{o_i}}\right) V_{STP_i}}$$

where  $B_i$  is in units of  $\text{g/cm}^3$  STP.

A linear least squares fit is performed on the  $(B_i, P/P_{o_i})$  pairs where  $B_i$  is the dependent variable and  $P/P_{o_i}$  is the independent variable. The following are calculated:

Slope,  $S$ ,  $\text{g/cm}^3$  STP

Y-intercept,  $Y_{INT}$ ,  $\text{g/cm}^3$  STP

Error of the slope,  $SERR$ ,  $\text{g/cm}^3$  STP

Error of the y-intercept,  $YI_{ERR}$ ,  $\text{g/cm}^3$  STP

Correlation coefficient,  $C_C$

Using the results of the above calculations, the following are calculated:

**BET Surface Area ( $SA_{BET}$   $\text{m}^2/\text{g}$ ):**

$$SA_{BET} = \frac{CSA \times (6.023 \times 10^{23})}{(22414 \text{ cm}^3 \text{ STP}) \times (10^{18} \text{ nm}^2/\text{m}^2) \times (S + Y_{INT})}$$

CSA = adsorbate molecular cross-sectional area ( $\text{nm}^2$ )

**BET C value:**

$$C = \frac{S + Y_{INT}}{Y_{INT}}$$

**Volume of monolayer ( $\text{cm}^3/\text{g}$  STP):**

$$V_M = \frac{1}{C \times Y_{INT}} = \frac{1}{S + Y_{INT}}$$

**Error of the BET Surface Area (m<sup>2</sup>/g):**

$$BET_{ERR} = \frac{SA_{BET} \times (S_{ERR}^2 + YI_{ERR}^2)^{0.5}}{Y_{INT} + S}$$

**Langmuir Surface Area**

For each included point i, pressure P<sub>i</sub> and specific volume adsorbed VSTP<sub>i</sub> are calculated as for a single point BET. Then the Langmuir transformation L<sub>i</sub> is calculated:

$$L_i = \frac{P_i}{V_{STP_i}}$$

where L<sub>i</sub> is in units of g/cm<sup>3</sup> STP.

A linear least squares fit is performed on the (L<sub>i</sub>, P) pairs where L<sub>i</sub> is the dependent variable and P is the independent variable. The following are calculated:

Slope, S, g/cm<sup>3</sup> STP  
 Y-intercept, YINT, g/cm<sup>3</sup> STP  
 Error of the slope, SERR, g/cm<sup>3</sup> STP  
 Error of the y-intercept, YIERR g/cm<sup>3</sup> STP  
 Correlation coefficient, CC

Using the results of the above calculations, the following are calculated:

**Langmuir Surface Area (SALAN m<sup>2</sup>/g):**

$$SA_{LAN} = \frac{CSA \times (6.023 \times 10^{23})}{(22414 \text{ cm}^3 \text{ STP}) \times (10^{18} \text{ nm}^2/\text{m}^2) \times S}$$

**Volume of monolayer (cm<sup>3</sup>/g STP):**

$$V_M = \frac{1}{S}$$

**Langmuir b value:**

$$b = (Y_{INT})(V_M)$$

**Error of the Langmuir Surface Area (m<sup>2</sup>/g):**

$$LAN_{ERR} = \frac{SA_{LAN} \times S_{ERR}}{S}$$

**Total Pore Volume**

---

Total pore volume VPOR at a selected physisorption point is calculated:

$$V_{POR} = (V_{STP})(DCF)$$

DCF = adsorbate density conversion factor

**Volume Sorbed**

---

This equation is for loop or syringe injections to calculate volume removed from the injections by the sample.

$$V_S = NV_i - V_{na}$$

$V_s$  = Volume sorbed, cm<sup>3</sup>

$N$  = Number of injections

$V_i$  = Volume per injection, cm<sup>3</sup>

$V_{na}$  = Total volume not sorbed, cm<sup>3</sup>

**Peak Area Volume**

---

This is the basic data reduction equation. It converts the area under a peak to cm<sup>3</sup> of gas.

$$V_{pa} = A_p \times F_c$$

$V_{pa}$  = Peak area volume, cm<sup>3</sup>

$A_p$  = Peak area

$F_c$  = Calibration factor

## Stoichiometry Factor

The calculated stoichiometry factor is a weighted average. It is dependent on both the individual stoichiometry factor and number of moles of each active metal.

$$SF_{calc} = \left[ \left( \frac{F_1 \times SF_1}{W_{atomic1}} \right) + \left( \frac{F_2 \times SF_2}{W_{atomic2}} \right) + \dots + \left( \frac{F_N \times SF_N}{W_{atomicN}} \right) \right] \cdot (GMW_{calc})$$

- $SF_{calc}$  = Calculated stoichiometry factor
- $SF_N$  = Stoichiometry factor for metal N
- $F_1$  = Fraction of sample weight for metal N
- $W_{atomic1}$  = Gram molecular weight of first metal (g/g-mole)
- $GMW_{calc}$  = Gram molecular weight (g/g-mole)

## Gram Molecular Weight

The calculated Gram Molecular Weight is a weighted average of the number of moles of each active metal.

$$GMW_{calc} = \frac{1}{\left( \frac{F_1}{W_{atomic1}} \right) + \left( \frac{F_2}{W_{atomic2}} \right) + \dots + \frac{F_N}{W_{atomicN}}}$$

- $GMW_{calc}$  = Gram molecular weight (g/g-mole)
- $F_N$  = Fraction of sample weight for metal N
- $W_{atomicN}$  = Gram Molecular Weight of first metal (g/g-mole)

## Cross-Sectional Area

$$SA_{calc} = \left[ \left( \frac{F_1 \times SA_1}{W_{atomic1}} \right) + \left( \frac{F_2 \times SA_2}{W_{atomic2}} \right) + \dots + \left( \frac{F_N \times SA_N}{W_{atomicN}} \right) \right] \cdot (GMW_{calc})$$

- $SA_{calc}$  = Calculated cross-sectional area
- $F_N$  = Fraction of sample weight for metal N
- $SA_N$  = Cross-sectional area for metal N
- $W_{atomicN}$  = Gram molecular weight of first metal (g/g-mole)



## Metal Density

$$\rho_{calc} = \left[ \left( \frac{F_1 \times D_1}{W_{atomic1}} \right) + \left( \frac{F_2 \times D_2}{W_{atomic2}} \right) + \dots + \left( \frac{F_N \times D_N}{W_{atomicN}} \right) \right] \cdot (GMW_{calc})$$

- $D_{calc}$  = Calculated density  
 $F_N$  = Fraction of sample weight for metal N  
 $D_N$  = Density for metal N  
 $W_{atomicN}$  = Gram Molecular Weight of first metal (g/g-mole)

## Metallic Surface Area

This equation calculates the effective metallic surface area per gram of sample.

$$SA_{Metallic} = \left( \frac{V_s}{SW \times 22414} \right) \times (SF_{calc}) \times (6.123 \times 10^{23}) \times (SA_{calc})$$

- $SA_{Metallic}$  = Metallic surface area (m<sup>2</sup>/g of metal)  
 $V_s$  = Volume sorbed (cm<sup>3</sup> at STP)  
 $SF_{calc}$  = Calculated stoichiometry factor  
 $SA_{calc}$  = Calculated specific surface area  
 $SW$  = Sample weight (g)

## Active Particle Size

This equation calculates the active particle size.

$$APS = \frac{6}{(D_{calc}) \times \left( \frac{W_s}{GMW_{calc}} \right) \times (6.023 \times 10^{23}) \times (SA_{calc})}$$

- $APS$  = Active particle size  
 $D_{calc}$  = Calculated metal density (g/cm<sup>3</sup>)  
 $W_s$  = Sample weight, g  
 $GMW_{calc}$  = Gram molecular weight (g/g-mole)  
 $SA_{calc}$  = Calculated specific surface area (per gram of metal)

## First Order Kinetics

---

This series of steps calculates the First Order Kinetics (Heat of Desorption). At least two experiments must be run — commonly 3 experiments are run. The ramp rate must be different for each run. The data are plotted and the slope determined.

The general equation is:

$$\ln\left(\frac{\beta}{T_p^2}\right) = -\frac{E_d}{RT_p} + \ln\left(\frac{E_d A}{RC}\right)$$

Determine the slope  $s$  by plotting  $2 \ln T_p - \ln \beta$  vs.  $\frac{1}{T_p}$  then determine  $E_d$  using:

$$E_d = s \times R$$

$\beta$	= Ramp rate, degrees/min
$E_d$	= Heat of desorption, K joules/(mole K)
$R$	= Gas constant
$T_p$	= Temperature at peak max
$s$	= Slope
$A$	= The quantity adsorbed at saturation
$C$	= A constant related to the desorption rate

## C. GAS INFORMATION

### Relative Thermal Conductivity of Gases

---

Name	Chemical Formula	Conductivity (Relative to Air)
Air		1.00
Ammonia	NH <sub>3</sub>	0.92
Argon	Ar	0.68
Butane	C <sub>4</sub> H <sub>10</sub>	0.60
Carbon Dioxide	CO <sub>2</sub>	0.62
Carbon Monoxide	CO	0.97
Ethane	C <sub>2</sub> H <sub>6</sub>	0.79
Helium	He	5.84
Hydrogen	H <sub>2</sub>	7.07
Krypton	Kr	0.37
Methane	CH <sub>4</sub>	1.29
Neon	Ne	1.87
Nitric Oxide	NO	0.99
Nitrogen	N <sub>2</sub>	1.00
Nitrogen Dioxide	NO <sub>2</sub> or N <sub>2</sub> O <sub>4</sub>	1.51
Nitrous Oxide	N <sub>2</sub> O	0.65
Oxygen	O <sub>2</sub>	1.02
Sulfur Dioxide	SO <sub>2</sub>	0.38
Water Vapor	H <sub>2</sub> O	0.67

## Conversion Constants for the Mass Flow Controllers (MFCs)

Gas	Symbol	MFC Conversion Constant (H <sub>2</sub> = 1.000)
Acetylene	C <sub>2</sub> H <sub>2</sub>	0.6535
Air (mixture)		0.9901
Allene	C <sub>3</sub> H <sub>4</sub>	0.4752
Ammonia	NH <sub>3</sub>	0.7822
Argon	Ar	1.3861
Arsine	AsH <sub>3</sub>	0.7525
Boron Trichloride	BCl <sub>3</sub>	0.4356
Boron Trifluoride	BF <sub>3</sub>	0.5743
Bromine Pentafluoride	BrF <sub>5</sub>	0.2871
Bromine Trifluoride	BrF <sub>3</sub>	0.4356
Butane	C <sub>4</sub> H <sub>10</sub>	0.2871
Butene	C <sub>4</sub> H <sub>8</sub>	0.3267
Carbon Dioxide	CO <sub>2</sub>	0.7723
Carbon Monoxide	CO	0.9802
Carbon Tetrachloride	CCl <sub>4</sub>	0.3465
Carbon Tetrafluoride	CF <sub>4</sub>	0.4752
Carbonyl Fluoride	COF <sub>2</sub>	0.2673
Carbonyl Sulfide	COS	0.6733
Chlorine	Cl <sub>2</sub>	0.8218
Chloroform	CHCl <sub>3</sub>	0.4356
Chlorine Trifluoride	ClF <sub>3</sub>	0.4257
Cyanogen	C <sub>2</sub> N <sub>2</sub>	0.4950
Cyclopropane	C <sub>3</sub> H <sub>6</sub>	0.5050
Deuterium	D <sub>2</sub>	0.9901
Diborane	B <sub>2</sub> H <sub>6</sub>	0.5446
Dichlorosilane	SiH <sub>2</sub> Cl <sub>2</sub>	0.4356
Dimethylamine	(CH <sub>3</sub> ) <sub>2</sub> NH	0.6634
Dimethylether	(CH <sub>3</sub> ) <sub>2</sub> O	0.5842
Ethane	C <sub>2</sub> H <sub>6</sub>	0.5446
Ethyl Chloride	C <sub>2</sub> H <sub>5</sub> Cl	0.2871
Ethylene	C <sub>2</sub> H <sub>4</sub>	0.6139
Ethylene Oxide	C <sub>2</sub> H <sub>4</sub> O	0.5842

Gas	Symbol	MFC Conversion Constant (H <sub>2</sub> = 1.000)
Fluorine	F <sub>2</sub>	0.9208
Fluroform	CHF <sub>3</sub>	0.5644
Freon 11	CCl <sub>3</sub> F	0.3762
Freon 12	CCl <sub>3</sub> F <sub>2</sub>	0.3861
Freon 13	CClF <sub>3</sub>	0.4257
Freon 13 B1	CBrF <sub>3</sub>	0.4059
Freon 14	CF <sub>4</sub>	0.4703
Freon 21	CHCl <sub>2</sub> F	0.4554
Freon 22	CHClF <sub>2</sub>	0.5050
Freon 23	CHF <sub>3</sub>	0.5644
Freon 114	CCl <sub>2</sub> F <sub>4</sub> -CClF <sub>2</sub>	0.2554
Freon 115	CClF <sub>2</sub> -CF <sub>3</sub>	0.2713
Freon 116	CF <sub>3</sub> -CF <sub>3</sub>	0.2277
Germane	GeH <sub>4</sub>	0.6436
Helium	He	1.3762
Hexamethyldisizane	HMDS	0.1386
Hydrogen	H <sub>2</sub>	1.0000
Hydrogen Bromide	HBr	0.9703
Freon 113	CCl <sub>2</sub> F-CClF <sub>2</sub>	0.2277
Hydrogen Chloride (Dry)	HCl	0.9802
Hydrogen Fluoride	HF	0.9901
Hydrogen Iodide	HI	0.9505
Hydrogen Selenide	H <sub>2</sub> Se	0.8317
Hydrogen Sulfide	H <sub>2</sub> S	0.8416
Isobutane	C <sub>4</sub> H <sub>10</sub>	0.3069
Isobutylene	C <sub>4</sub> H <sub>8</sub>	0.3366
Krypton	Kr	1.3762
Methane	CH <sub>4</sub>	0.8020
Methylamine	CH <sub>3</sub> NH <sub>2</sub>	0.5644
Methyl Bromide	CH <sub>3</sub> Br	0.6436
Methyl Chloride	CH <sub>3</sub> Cl	0.6832
Methyl Fluoride	CH <sub>3</sub> F	0.7525
Methyl Mercaptan	CH <sub>4</sub> S	0.5842
Neon	Ne	1.3861

Gas	Symbol	MFC Conversion Constant (H <sub>2</sub> = 1.000)
Methyl Mercaptan	CH <sub>4</sub> S	0.5842
Neon	Ne	1.3861
Nitric Oxide	NO	0.9901
Nitrogen	N <sub>2</sub>	0.9950
Nitrogen Dioxide	NO <sub>2</sub>	0.7525
Nitrogen Trioxide	N <sub>2</sub> O <sub>3</sub>	0.4356
Nitrogen Trifluoride	NF <sub>3</sub>	0.5446
Nitrous Oxide	N <sub>2</sub> O	0.7426
Oxygen	O <sub>2</sub>	0.9802
Ozone	O <sub>3</sub>	0.7327
Pentaborane	B <sub>5</sub> Hg	0.2871
n Pentane	C <sub>5</sub> H <sub>12</sub>	0.2376
Perchloryl Fluoride	ClO <sub>3</sub> F	0.4455
Phosgene	COCl <sub>2</sub>	0.5050
Phosphine	PH <sub>3</sub>	0.7822
Phosphorous Pentafluoride	PF <sub>5</sub>	0.3465
Propane	C <sub>3</sub> H <sub>8</sub>	0.3861
Propylene (Propene)	C <sub>3</sub> H <sub>6</sub>	0.4653
Silane	SiH <sub>4</sub>	0.6733
Silicon Tetrachloride	SiCl <sub>4</sub>	0.3168
Silicon Tetrafluoride	SiF <sub>4</sub>	0.3960
Sulfur Dioxide	SO <sub>2</sub>	0.7228
Sulfur Hexafluoride	SF <sub>6</sub>	0.2970
Trichlorosilane	SiCHl <sub>3</sub>	0.3267
Trimethylamine	(CH <sub>3</sub> ) <sub>3</sub> N	0.3168
Tungsten Hexafluoride	WF <sub>6</sub>	0.2871
Uranium Hexafluoride	UF <sub>6</sub>	0.2178
Vinyl Bromide	C <sub>2</sub> H <sub>3</sub> Br	0.5248
Vinyl Chloride	C <sub>2</sub> H <sub>3</sub> Cl	0.5347
Vinyl Fluoride	C <sub>2</sub> H <sub>3</sub> F	0.5743
Xenon	Xe	1.3762

## D. FORMAT OF EXPORTED DATA

Data collected using the AutoChem II 2950 analyzer can be exported to a file for use in other applications. Data may be exported as an ASCII or Grams/32 (a peak-editing program used in earlier versions of the AutoChem application) format. Instructions for exporting data are contained in Chapter 5.

### ASCII Format

---

Data are exported in the following format when ASCII is chosen as the export type:

Header:

<sample id>  
<operator>  
<submitter>  
<sample mass>  
<instrument serial number>  
<ambient temperature>  
<ambient pressure>  
<Po of adsorbate>

After the Header is exported, the remaining data are determined by whether **Peak Summaries** or **Measurements** was specified in the report options set.

- For **Peak Summaries**, the peaks for each selected experiment in the file are summarized. Data are exported as follows:

<experiment number>, <start time (minutes)>, <stop time (minutes)>

- For **Each Peak**:

<experiment type>, <start time (minutes)>, <stop time (minutes)>,  
<temperature at maximum (°C)>, <area>

<experiment type> is one of the following: TPD", :TPR", "TPO", "Physisorption",  
"Chemisorption", "Level Cal.", "Vapor Cal.", "Loop Cal.", "Other"

- For **Measurements**, the data points for each checked experiment in the file are reported. Data are exported as follows:

<experiment number>, <start time (minutes)>, <stop time (minutes)>

**For Each Point:**

<time (minutes)>, <temperature (°C)>, <TCD signal>, <event flag>, <input signal 1>, <Input signal 2>

The TCD signal may be either calibrated or not calibrated based upon the selections made when the export operation was performed.

The *event flag* is either 1 or 0. The state of the event flag switches when a pulse chemisorption dose is started.

## Grams/32

When Grams/32 is chosen as the export type, five files are created for each experiment selected for export. The data in these files can be read only by the Grams/32 software. Table D-1 lists the names and contents of these files.

**Table D-1. Grams/32 Export File Names**

File Name	Contents
xxxnn.CGM	Calibrated TCD signal vs. time (same as xxxSGnn.SPC when no calibration is specified)
xxxSGnn.SPC	Uncalibrated TCD signal vs. time
xxxTMnn.SPC	Sample temperature vs. time
xxxT1nn.SPC	Auxiliary signal one vs. time
xxxT2nn.SPC	Auxiliary signal two vs. time

Where **xxx** is the first three characters of the input sample file name and **nn** is the number of the experiment within the sample.

For example, the *second* experiment of the sample file **ABC.SMP** would be exported into the following Grams/32 files:

ABC02.CGM  
ABCSG02.SPC  
ABCTM02.SPC  
ABCT102.SPC  
ABCT202.SPC

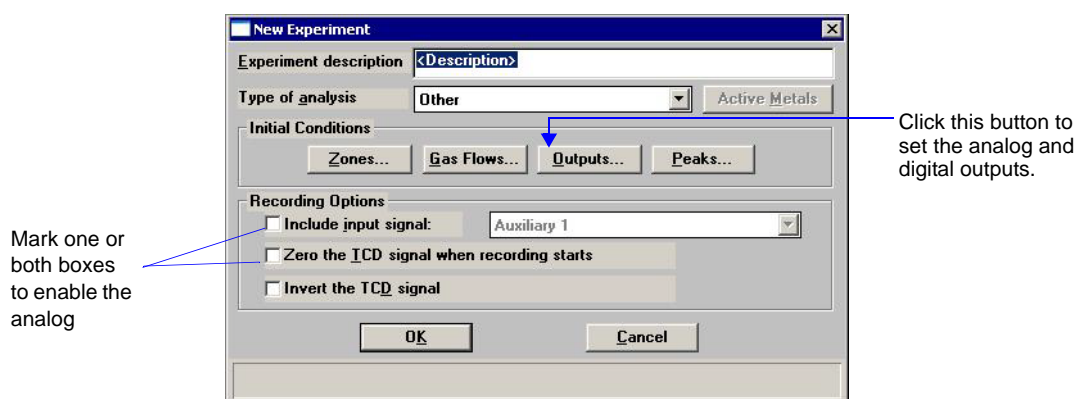


## E. AUXILLIARY INPUTS AND OUTPUTS

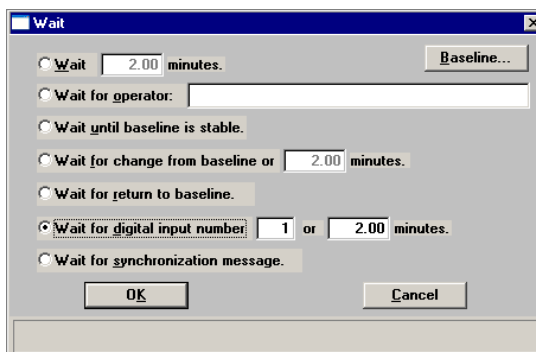
You can connect analog and digital signals to the AutoChem 2950 HP analyzer. Two connectors are available on the analyzer's right side panel; both are industry-standard "D" shell connectors. You must supply a shielded cable for any auxiliary signal you connect to the analyzer. All cables must use male pins in the connectors. The analog connector uses a 15-pin body; the digital connector uses a 25-pin body. The cable shield must connect to a metal shell "D" connector housing.

### Software

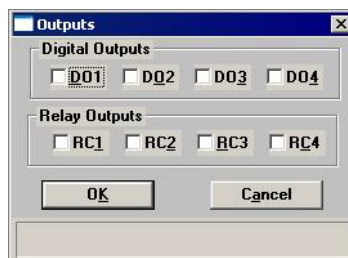
Analog inputs are enabled by marking one or both check boxes on the Insert Experiment dialog; this dialog is opened from the Analysis Conditions dialog of the sample information file (Advanced format only). Click **Insert**, then select **Experiment**. After an auxiliary input is enabled, it remains enabled for the duration of the experiment. Because an analysis can be comprised of many experiments, an input can be disabled or enabled at various points in the analysis, as determined by the selection on the New Experiment dialogs.



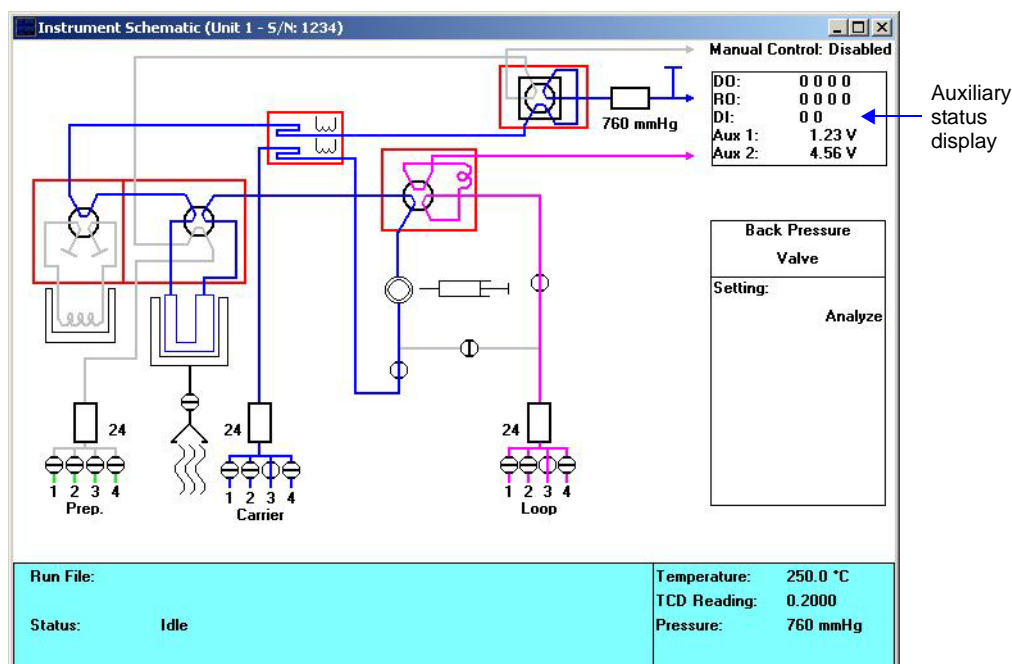
Digital inputs are controlled by inserting a **Wait** step in the analysis. On the Wait dialog (shown below), mark the radio button next to Wait for digital input number. Specify **1** or **2** to indicate the input signal. Enter a number of minutes to wait; if the signal is not received before that amount of time elapses, the analysis continues without the input.



Analog and digital outputs are enabled by marking the applicable check box(es) on the Outputs dialog. The Outputs dialog is available when you are setting the initial conditions for an experiment (by clicking the **Outputs** button on the New Experiment dialog), or at many other points in the analysis (by inserting a **Set Outputs** step in the Synopsis). The state of the auxiliary outputs can therefore be changed frequently throughout the analysis.



The state of the auxiliary inputs and outputs is displayed on the instrument schematic. Auxiliary signals can be incorporated in reports (overlaid over other data in graphs), in the Results view of the Analysis dialog (during analysis), and in the Peak Editor.



In the Auxiliary Status Display on the instrument schematic, a zero or a one is displayed to show the state of each digital output (DO), relay output (RO), and digital input (DI). Refer to [“Digital” on page E-3](#) for additional information.

The voltage is displayed for each analog input (AUX 1 and AUX 2).

## Analog

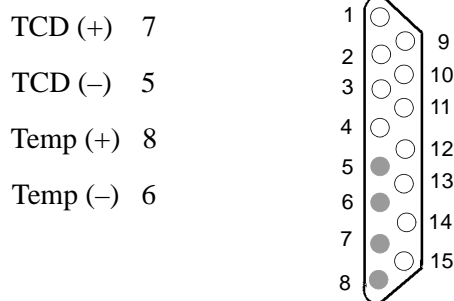
The 15-pin analog connector provides two output signals and two input signals.

## Outputs

The two output signals are:

- The TCD signal, which could be as high as 12 volts (positive or negative)
- The sample temperature signal, which is a buffered voltage from the sample thermocouple. The voltage ranges from  $-0.365$  V at  $-40$  °C; 0 at 0 °C; to 10.88 V at 110 °C. The signal follows the type-K thermocouple nonlinear characteristic.

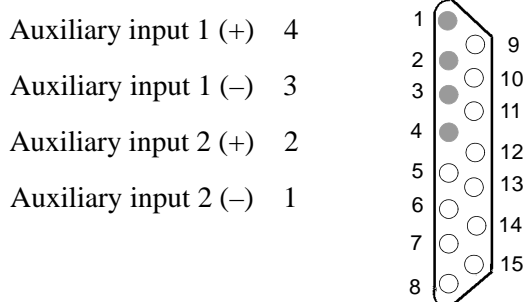
The signals are available at these pins:



## Inputs

The inputs are available for connection to other detectors, such as a mass spectrometer. The voltage range is  $-15$  to  $15$  volts. The software can record these signals at the same time as it records TCD and sample thermocouple data.

The signals connect to these pins:



All other pins in the 15-pin connector are floating (not connected). You should connect the metal shell of your connector to chassis ground and the cable shield to maintain shielding of the signals.

## Digital

The 25-pin digital connector provides floating signals to control and monitor external

hardware, such as auxiliary detectors or chart recorders.

The outputs consist of four sets of latching relay contacts and four opto-coupled photo transistors. The inputs are two optically isolated photo couplers.

The software sets these as logic 1 or logic 0, as defined by the analysis step. They are displayed on the instrument schematic.

A logic 1 causes the relay to connect the “common” contact to the “normally open” contact. The relay will retain this condition even when power to the instrument is disconnected. A logic 0 causes the relay to connect the “common” contact to the “normally closed” contact.

A logic 1 to the opto couple outputs causes the NPN photo transistor to be turned on. A logic 0 turns the NPN photo transistor off.

The two opto-coupled inputs are read by the instrument as logic 1 (when current flows through the opto coupler photo diodes) or logic 0 (when no current is flowing).

---

## Electrical Specifications

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### Relays

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Maximum voltage: 250 VAC, 48 VDC

Maximum switching current: 4A at 250 VAC; 3A at 30 VDC

Maximum switching current: 100 mA at 100 VDC

### Opto-coupled outputs

---

Maximum voltage, VCE = 70 VDC

Maximum reverse voltage, VCE = 7 VDC

Maximum current = 10 mA

### Opto-coupled inputs

---

Maximum reverse voltage = 3 v

Nominal forward voltage = 1.3 v at 10 mA

Minimum current required to be recognized by the 2950 analyzer = 1 mA

**Digital Connector Pin Designations**

Pin	Type	Function
1	Relay 1	normally closed
2	Relay 1	common
3	Relay 1	normally open
4	Relay 2	normally closed
5	Relay 2	common
6	Relay 2	normally open
7	Relay 3	normally closed
8	Relay 3	common
9	Relay 3	normally open
10	Relay 4	normally closed
11	Relay 4	common
12	Relay 4	normally open
13	Not used	Not used
14	Opto output 1	collector
15	Opto output 1	emitter
16	Opto output 2	collector
17	Opto output 2	emitter
18	Opto output 3	collector
19	Opto output 3	emitter
20	Opto output 4	collector
21	Opto output 4	emitter
22	Opto input 1	anode (+)
23	Opto input 1	cathode (-)
24	Opto input 2	anode (+)
25	Opto input 2	cathode (-)



## F. CALIBRATING THE MASS FLOW CONTROLLERS FOR EACH GAS

The AutoChem 2950 HP Analyzer uses Mass Flow Controllers (MFCs) to control the flow of gases. These MFCs require a conversion constant for each gas or gas mixture, to compensate for variations in gas flows resulting from variations in the gases' properties. Default MFC conversion constants are provided for a wide variety of gases is contained in the software. You can view this table by selecting **Options > Gas Defaults**. A more complete list of gases and their conversion constants can be found in **“Conversion Constants for the Mass Flow Controllers (MFCs)” on page C-2**.

In most cases, using the default MFC conversion constant yields accurate data. A new conversion constant can be determined if:

- You are using a unique gas mixture
- The gas to use is not included in the table
- You require a higher precision calibration of the MFCs for a given gas
- Unexpected analysis data lead you to believe that the default value is in error

### Gas Mixtures

---

To determine the conversion constant for a gas mixture, you must first obtain the conversion constant for each gas in the mixture. For each gas, you may either use the default conversion constant or determine a new constant using the method described in this appendix. A combination of default and calculated constants may be used.

After a constant is obtained for each gas in the mixture, use the formula on the worksheet at the end of this appendix to determine the constant for the mixture.

### Flow Measurement

---

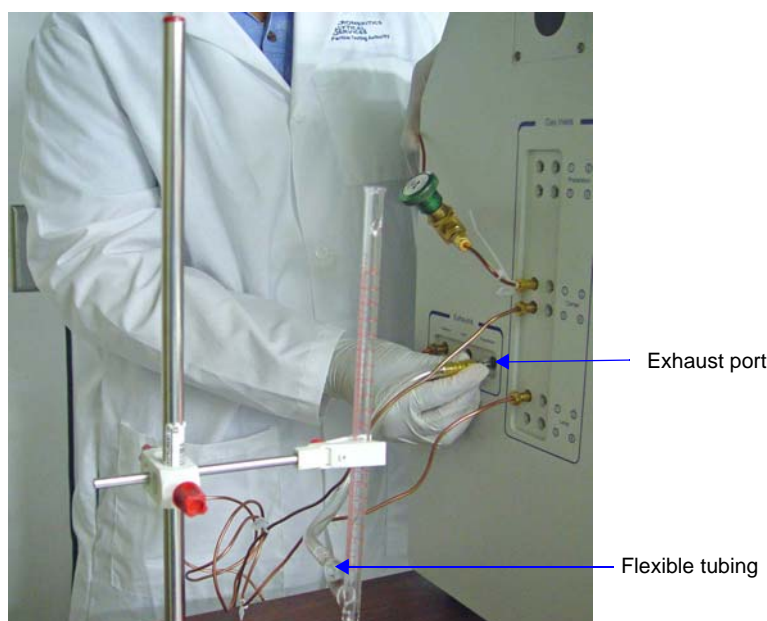
An external device that measures the flow of gas from the analyzer's exhaust is used during MFC calibration. A soap bubble burette is shipped with your instrument. For even more precise calibration, you may choose to use another type of flow meter; follow these instructions for obtaining a new conversion constant, substituting your flow meter for the bubble burette.

## Installing the Soap Bubble Burette



**These instructions are for assembling and using the burette supplied by Micromeritics. If you are using another type of flow meter, follow the meter manufacturer's assembly and operation instructions.**

1. Carefully unwrap the glass tube and the rubber bulb. Attach one end of the flexible tubing to the side arm of the glass tube.
2. Locate the exhaust port that corresponds to the MFC you are using for this calibration. Remove external plumbing or plug from that port and attach the bubble burette using the connector provided with the burette. For example, if you are using the Preparation Flow Controller, attach the bubble burette to the preparation gas exhaust port.





3. Fill the rubber bulb with leak detector fluid. An 8-oz. bottle of the fluid is included in the accessory kit. Attach the bulb to the bottom of the glass tube.



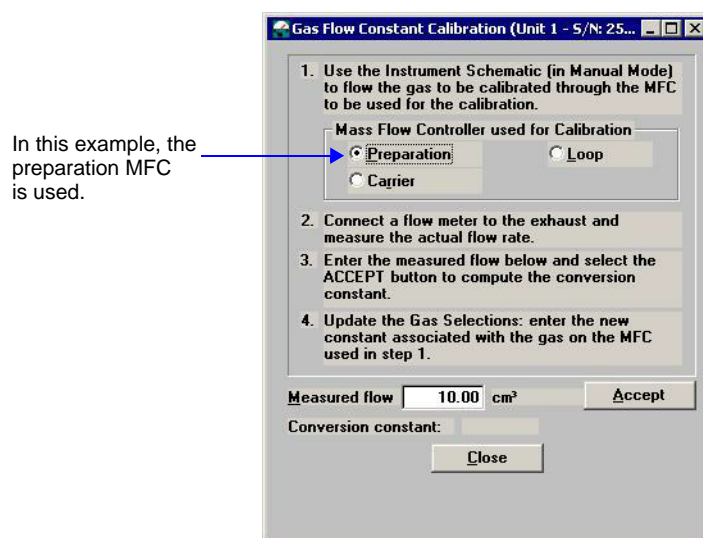
Attach bulb filled with leak detector fluid



The soap bubble burette must be held in an upright position. An example of a stand and clamp is shown in the illustration above.

## Determining the Constant for Individual Gases

1. Make a copy of the Gas Flow Constant Calibration Worksheet (at the end of this appendix). Use the worksheet to complete the following procedure.
2. If the gas tube used to determine a calibration factor is not attached to an inlet port, attach it before proceeding. Use an inlet port for the MFC you plan to use during this calibration. For example, if you plan to use the Preparation MFC, attach the gas to one of the preparation inlet ports.
3. Select **Unit > Gas Flow Constant Calibration**. On the dialog box, mark a radio button to indicate which MFC you plan to use. The MFCs are identical, so you should use the most convenient one.

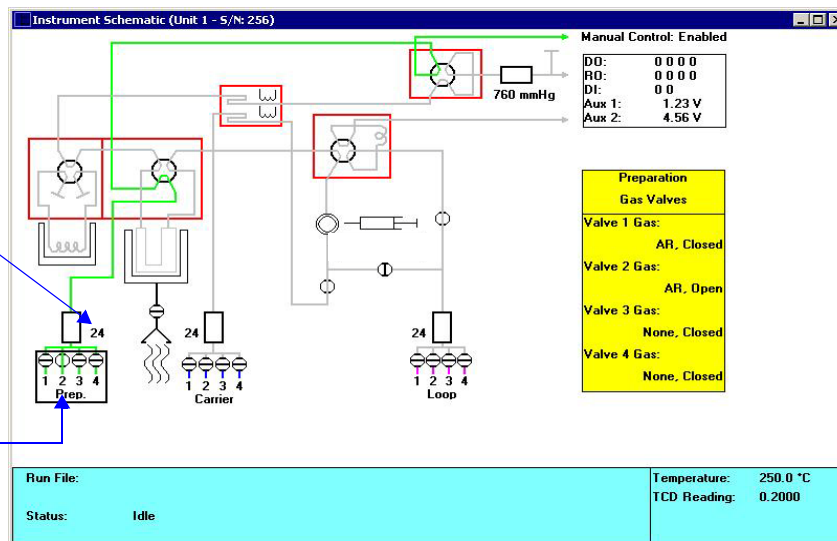


4. Flow the gas to be calibrated through the appropriate Mass Flow Controller by following these steps:
  - a. Select **Unit > Show instrument schematic**. Then, select **Unit > Enable manual control**. (The manual control function is explained on page 6-12.)

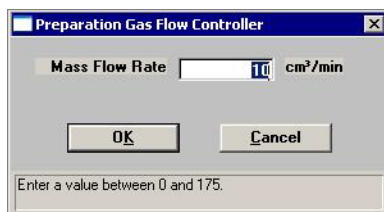
In this example, the preparation gas MFC will be used to obtain a calibration factor for the new gas.

The current flow rate is displayed beside the MFC icon.

In this example, the new gas is connected to preparation gas inlet port 2. Inlet valve 2 is opened to create a gas flow through the preparation MFC.



- b. On the instrument schematic, place the cursor over the icon that represents the inlet valves for the Flow Controller you are using. Click the right mouse button and select the Flow Gas (number) option for the inlet valve to which the new gas is connected.
- c. Place the cursor over the icon representing the Mass Flow Controller to use. Click the right mouse button, then select **Set Flow Rate**. A window opens allowing you to specify a flow rate.



Be sure to use a rate that can be verified by the flow meter you plan to use. Record the flow rate on line 1 of the worksheet. Click **OK** to close the dialog.

5. Make sure a flow meter is connected to the appropriate exhaust port. Measure the actual flow rate in standard  $\text{cm}^3/\text{min}$  and record the flow rate on line 2 of the worksheet.

If you are using the bubble burette shipped with your analyzer, observe a bubble as it rises through the glass tube. The lines on the glass tube indicate the beginning and ending points for measuring the progress of a bubble through the tube. Use a stopwatch to measure the amount of time that elapses from the moment the bubble passes the lower mark on the tube until the moment it passes the higher mark on the tube.

6. Convert the measured flow to Standard Temperature and Pressure (STP) conditions. The formula is provided on the worksheet; record the value on line 3.
7. Enter the measured flow rate at STP on the Gas Flow Constant Calibration dialog. Click **Accept** to calculate a conversion constant for this gas. Record the value on line 5.
8. Open the Gas Defaults table (on the Options Menu) and update it to include the new gas and its conversion constant. (If you have used this process to obtain a new constant for an existing gas in the table, update the conversion constant for that gas.)



**The gas you just added can then be selected for any port on the Gas Selections Dialog. If you plan to leave this gas connected to the port, be sure to change the gas selection for that port to the correct name.**

## Gas Flow Constant Calibration Worksheet

---

Follow the instructions for calibrating the analyzer's Mass Flow Controllers for a given gas, using this form to record measured values and perform calculations included in this process. Instructions are provided for single (individual) gases and gas mixtures.

### Single Gases

---

MFC being used (optional):

\_\_\_\_ Preparation

\_\_\_\_ Carrier

\_\_\_\_ Analysis (Loop)

Gas: \_\_\_\_\_

1. MFC flow rate (as set on instrument schematic): \_\_\_\_\_
2. Measured flow rate (using an external flow meter): \_\_\_\_\_
3. Convert the flow rate to Standard Temperature and Pressure (STP) using the following formula:

$$\text{Rate at STP} = \text{Rate measured (cm}^3\text{)} \times \frac{273.15 \text{ K}}{273.15 \text{ K} + \text{room temp } ^\circ\text{C}} \times \frac{\text{atmospheric pressure}}{760 \text{ mmHg}}$$

Measured rate at STP: \_\_\_\_\_

4. Enter the value from # 3 in the Measured Flow field of the Gas Flow Constant Calibration dialog, then click **Accept**.)
5. The new MFC Conversion Constant (displayed at the bottom of the Gas Flow Constant Calibration dialog) is: \_\_\_\_\_
6. Enter the new Conversion Constant for this gas (# 5) on the Gas Defaults table, which is accessed from the Options menu.

## Gas Mixtures

Obtain a conversion constant for each gas in the mixture. Use the conversion constant provided in the **Gas Defaults** table (on the Options menu) or in **“Conversion Constants for the Mass Flow Controllers (MFCs)” on page C-2**, or use the instructions above to obtain a new constant. Record the names and constants for each gas in the mixture in the table provided in this section. Use the following formula to calculate the conversion constant for the gas mixture.

$$M = \frac{1}{\left[\frac{P_1}{F_1 \times 100}\right] + \left[\frac{P_2}{F_2 \times 100}\right] + \left[\frac{P_n}{F_n \times 100}\right]}$$

- $M$  = the mixture conversion constant  
 $P$  = the percentage of gas n in the mixture, expressed as a whole number (example: for 15%, use 15, not 0.15)  
 $F$  = the conversion constant (factor) for gas n (From the Gas Defaults Table or from step 5 on this worksheet)

Enter the gas mixture in the Gas Defaults table; use M as the conversion constant.

Gas Name (n)	Conversion Constant (F)
1.	
2.	
3.	
4.	
5.	
6.	
7.	
8.	
9.	
10.	
11.	
12.	
13.	
14.	
15.	
16.	
17.	
18.	
19.	
20.	

Mixture name: \_\_\_\_\_ Constant (M): \_\_\_\_\_



## G. ATOMIC WEIGHTS AND CROSS-SECTIONAL AREAS FOR SELECTED METALS

Metal	Symbol	Atomic Weight (g/mole)	Cross-Sectional Area (sq nm)
chromium	Cr	51.996	0.0635
cobalt	Co	58.933	0.0662
copper	Cu	63.546	0.0680
gold	Au	196.967	0.08696
hafnium	Hf	178.490	0.0862
iridium	Ir	192.220	0.0769
iron	Fe	55.847	0.0613
manganese	Mn	54.938	0.0714
molybdenum	Mo	95.940	0.0730
nickel	Ni	58.710	0.0649
niobium	Nb	92.906	0.0806
osmium	Os	190.220	0.0629
palladium	Pd	106.400	0.0787
platinum	Pt	195.090	0.0800
rhenium	Re	186.207	0.0649
rhodium	Rh	102.906	0.0752
ruthenium	Ru	101.070	0.0613
silver	Ag	107.868	0.0869
tantalum	Ta	180.947	0.0800
thorium	Th	232.038	0.1350
titanium	Ti	47.900	0.0741
tungsten	W	183.850	0.0741
vanadium	V	50.942	0.0680
zirconium	Zr	91.220	0.0877





## H. PEAK DETECTION / INTEGRATION OPTIONS

Peak detection parameters can be customized using the Peak Detection/Integration Options dialog. If you customize this dialog when you create the sample file, you can control peak detection during analysis. You can also control peak detection after analysis by customizing this dialog in the Peak Editor. The defaults for the parameters on this dialog are usually acceptable, but they may be changed.

- **To open this dialog while you are creating the sample file:**  
With the sample file open in Advanced format, click on the Analysis Conditions tab. Click **Insert**, then select **Experiment**. Click **Peaks**.
- **To open the dialog while you are in Peak Editor:**  
Select the **Edit Peaks** tab. Then right-click in the graph area and choose **Peak Integration Options**.

**Peak Detection/Integration Options**

**Standard Settings**

Baseline Mode: ☒ Best Fit Baseline, ☐ Snap to Baseline

Threshold Y Level: 0.25

Peak Smoothing: 0

Number of points for smoothing. If Peak Smoothing value is 0, automatic selection is enabled (1% of data points). Its purpose is identifying peak edges - it does not affect peak height or area.

**Advanced Settings**

Sensitivity: 0.01

Max Shoulder Ratio: 33

Max Group Separation: 33

Minimum Peak Area: 1.0

Maximum Baseline Slope: 0.1

OK Cancel

The TCD detects and records all deviations from baseline, but only those which satisfy the criteria established in this dialog are reported as peaks.



**Peak detection is accomplished through a combination of noise, height, and area thresholds.**

---

## Baseline Mode

---

This feature affects how the Find All Peaks function works.

If **Best Fit Baseline** is selected, the bottom of the peaks is placed in the baseline that best describes the signal outside the range of the peaks. This assumes a linear baseline between the beginning and the end of the peak.

If **Snap to Baseline** is selected, the bottom of the peaks is moved to the signal recorded, not the best fit baseline between the peaks.

---

## Threshold Y Level

---

Sets the minimum height for peaks to be identified and included in the peak table. This value is expressed in terms of the trace's Y-axis units. Use a value of 0 (zero) to include all peaks.

---

## Smoothing

---

This feature allows the software to average the points before using them, so that noise spikes are ignored. Specify the number of points to average into a single value during the peak picking process.

The smoothing parameter can be turned off by setting the value at **1** or **0** (zero). A setting of **1** disables smoothing, and the peak edges are interpolated to the best X-axis value. A setting of **0** also disables smoothing, but the peak edges (the points where the peak begins and ends) are not interpolated. Instead, the nearest data point is used as the peak edge.

---

## Sensitivity

---

Sets the noise rejection level for identifying the peaks in a trace. Use a value from -100 to 100%. For example, a sensitivity level of 5% means that 5% of all local maxima in the trace are greater than the noise and are, therefore to be considered as peaks. A value of 100% identifies all local maxima (or minima for traces with transmission Y units) as peaks. One possible exception to note is the combination of other rejection parameters (such as Minimum Peak Area and Threshold Y Level) which can reject peaks even when the sensitivity is set to 100%.

The sensitivity can also be set to negative values to define a specific noise level (in Y units) for peak rejection. For example, a sensitivity setting of -2.5% sets the noise rejection to 2.5 V. This means that maxima with an amplitude of 2.5 V or less will be considered as baseline noise instead of as peaks. (As opposed to the Threshold Y Level rejection parameter which eliminates refined peaks by using their height above the baseline.)

A setting of **0** (zero) automatically sets a default noise level for the trace.

---

## Max Shoulder Ratio

---

There can also be “shoulder peaks” (also called combination peaks) within a group. (For a discussion of peak groups, see the description of Max Group Separation below.) Shoulders are usually small peaks that are overlapped on the front or the tail of a larger peak. These peaks can also be called “leaders” and “followers,” respectively. As with baseline groups, the areas of these peaks can be calculated incorrectly. If the larger “parent” peak has a long tail with a much smaller peak riding on it, most of the area under the trace belongs to the parent peak. However, if the area of these peaks was determined using baseline grouping, the smaller peak would be calculated by using vertical drop lines at the edges. This would give the parent peak too little area, and the rider peak too much.

The software can detect these shoulder peaks. The areas of shoulder peaks are calculated by drawing a skimmed baseline from the leading edge to the trailing edge. Either an exponential or a straight skim line can be used. The remaining area between the shoulder peak baseline and the group baseline is considered to be part of the parent peak.

The **Max Shoulder Ratio** parameter is used to specify whether the peaks that are overlapped in the front or the tail of much larger peaks should be identified as shoulder peaks. To use shoulder peak detection, use a non-zero value for the **Max Shoulder Ratio** parameter. After a baseline group has been identified, the software looks for peaks within the group that satisfy the following shoulder peak criteria.

Shoulders must have a significantly higher Y value at one edge than the other. More importantly, the height of the shoulder above the common value must be much smaller than the height of the “parent” (larger) peak above the same valley. It must be smaller by the **Max Shoulder Ratio** setting. For example, a setting of 33 implies that shoulders must be smaller than 33% of their parents in terms of height above the common valley. The areas for shoulder peaks are calculated by drawing a skimmed baseline from the left edge to the right edge of the peak. The remaining area between the shoulder peak baseline and the group common baseline is considered to be part of the parent peak.

Shoulder peaks can only be calculated within a group of peaks. (See the description of peak groups in the section below on **Max Group Separation**.) If the **Max Group Separation** parameter is set to **0** (no groups), a **Max Shoulder Ratio** parameter value is not used. Use a value of zero to specify no shoulder peak detection.

A value of 33% works well with most data. Use a setting of zero to treat shoulder peaks with a perpendicular drop to the common group baseline instead of a skimmed baseline.

If the software detects unwanted baseline noise peaks, try increasing the Sensitivity setting. Conversely, if some peaks are not detected, decrease the value.

## Max Group Separation

---

The software normally calculates peak areas by drawing a “valley-to-valley” baseline from the leading edge to the trailing edge of every identified peak. However, in many traces, the valleys between peaks do not always drop back to the original baseline. If a valley-to-valley baseline is used for this type of peak, the calculated area does not accurately reflect the true area under the peak.

The software provides a parameter that allows the calculation of baseline Groups.” A group of peaks is defined by a common baseline that extends from the leading edge of the first peak in the group to the trailing edge of the last. The areas of grouped peaks are calculated by dropping vertical lines from the peak edges down to the group baseline. (Note there may also be “Report Groups” defined by the method and assigned group letters from A through Z. Unlike these Baseline Groups, the Report Groups need not be next to one another. The two types of groups are not related.)

The Max Group Separation parameter is used to determine which peaks in a trace have a common baseline. When using peak grouping, the software compares the width (actually double the largest half width) of every identified peak in a trace to the width of the following peak. The Max Group Separation parameter specifies a percentage of the smallest of these two widths in X units. If the edges of two adjacent peaks differ by less than this value, the two peaks constitute a group and are given a common baseline. For example, if two adjacent peaks in the trace have largest half widths of 1 and 1.5 respectively, and the Max Group Separation parameter is set at 20%, then a difference of less than 0.4 X units between the adjacent edges of these peaks would make them a group with a common baseline. If the same two peaks have adjacent edges that are greater than 0.4 X units apart, they do not define a group, and each peak has its own separate baseline.

The areas for grouped peaks are calculated by drawing imaginary vertical lines from the peak edges to the common baseline. Any peaks that share common edges are automatically considered a group and are given a common baseline for any Max Group Separation setting greater than 0. To specify no peak grouping (each identified peak has its own baseline), use a setting of 0. A value of 33% for this parameter works well with most data. Use a setting of zero to force all baselines to be drawn from peak valley to valley.

## Minimum Peak Area

---

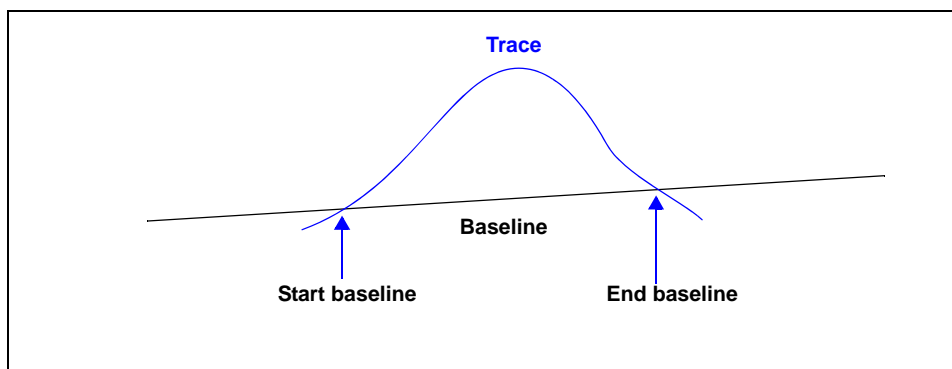
This parameter sets the minimum area required for refined, processed peaks to be recognized, identified, and included in the peak table. Any peak with a calculated peak area smaller than the current setting is not detected. Values for this parameter are expressed in terms of the trace X-axis units multiplied by the Y-axis units (e.g., millivolt-minutes).

Setting the Sensitivity parameter to large values (greater than 20%) can cause noise “spikes” (or “dips”) on the sides of major peaks to be identified as peaks themselves. If the areas of the peaks are smaller than the Minimum Peak Area parameter, major peaks in the trace may not be identified at all. Exercise caution when using high Sensitivity settings with the Minimum Peak Area parameter.

If peaks are not detected by the software, lower the setting with a smaller value. You must adjust this parameter in conjunction with the Peak Sensitivity and/or Threshold Y parameters above. Peak rejection is accomplished through a combination of peak height and peak area rejection parameters. If the software is not detecting your peak(s) of interest, you probably need to decrease both parameters.

## Maximum Baseline Slope

The maximum baseline slope indicates how the software determines the start and end of the baseline. The software algorithm scans the trace and looks for the first time when the slope of the trace is larger than the maximum baseline slope. Then it scans from the end of the trace backward and looks for the first time the slope is larger than the maximum baseline slope. It records these two points as the start and end of the baseline.



Basically, the Maximum Baseline Slope value is used to decide how flat the trace would need to be to set the baseline of the current peak.



## I. ERROR MESSAGES

Error messages are listed numerically. If the **Action** response instructs you to contact your service representative, record the error message and make backup copies of any files involved in the operation.



The 1000-series error messages (used primarily for software testing) are not included in this appendix. These errors should not occur during normal operation. If you receive a 1000 series error message or an error message not listed in this appendix: record the error message, make backup copies of any files involved, then contact your service representative.

### 2200 Series

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**2201- Cannot execute report subsystem.**

**2202- Cannot execute Peak Editor subsystem.**

*Cause:* The software for the report (or Peak Editor) subsystem has been corrupted and cannot be started.

*Action:* Reinstall the AutoChem software.

**2204- Communication attempt with (service) failed (code=n). Please check application directory for file.**

*Cause:* Inter-process communication with (service) did not succeed.

*Action:* Confirm that the service is located in the application directory. If it is not, reinstall the application. If the problem persists, contact your Micromeritics service representative.

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## 2400 Series

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**2401- FATAL ERROR: (error message)**

*Cause:* An internal processing and/or hardware error has occurred.

*Action:* Contact your Micromeritics service representative if you continue to receive this message

**2430- Error accessing file (file name), error code = [n]**

*Cause A:* Hard disk may be damaged.

*Action A:* Contact your service representative.

*Cause B:* A software error occurred when the file was accessed.

*Action B:* Contact your service representative.

*Cause C:* The name specified contains one or more invalid characters.

*Action C:* Enter a valid file name; do not use characters such as \* or?.

**2431- Error writing file (file name), error code = [n].**

*Cause:* The hard disk does not have enough space left to perform the operation.

*Action:* Copy files not used regularly from the hard disk to a diskette, CD, or network drive, delete them from the hard disk, and then try the operation again.

**2432- Invalid response from MMI 'File\_READ' request.**

*Cause:* An internal processing and/or hardware error has occurred.

*Action:* Contact your service representative if you continue to receive this error message



**2433- New entries have been found in this directory. Refresh the directory information?**

*Cause:* Several files (sample information, analysis conditions, or report options) have been added to this directory by some function other than the analysis program.

*Action:* Select **Yes** to update the directory information with data from each new file. This operation may take a minute.

Select **No** if you do not want to spend the time updating the directory information. This option may be feasible if a large number of files have been copied into the directory and you know the name of the file to access.

**2434- File (file name) - Subset # [n] wrote wrong amount of data.**

*Cause:* An internal processing and/or hardware error has occurred.

*Action:* Contact your service representative if you continue to receive this error message.

**2436- Path specification (path name) is invalid.**

*Cause:* You entered an invalid path name and/or extension.

*Action:* Type a valid path name (including the proper extension) and press **Enter**.

**2437- File (file name) does not exist.**

*Cause:* You entered a file specification that does not exist.

*Action:* Enter an existing file specification or select a file name from the list box

**2438- Disk drive (letter): is inaccessible.****2439- Could not register file.****2440- Subset not found.****2441- Seek within file failed.****2442- Had header in subset file.**

- 2443- Subset owner denied access.**  
**2444- Not a valid file format.**  
**2445- Subset wrote the wrong amount of data.**  
**2446- Error reading data.**  
**2447- Error writing data.**

*Cause:* An unexpected error occurred when you tried to access a data file.

*Action:* Contact your service representative.

**2448- File directory (path name) is invalid. Resetting to the installation directory.**

*Cause:* A working directory specified in the .INI file is invalid. The directory may have been deleted or moved to a different location.

*Action:* The installation directory will be substituted. The next time you open a file, use the Directories list to move to the correct directory.

**2449- This field does not contain a valid file specification.**

*Cause:* You entered an invalid file name.

*Action:* See the description of file naming conventions in your operating system or Windows manual and reenter the name.

**2450- Sample Defaults may not be edited while this operation is in progress. Do you wish to save and close the Sample Defaults edit session?**

*Cause:* You are in the process of initiating an automatic analysis (an analysis in which sample files are created using the defaults) while editing the defaults.

*Action:* Finish your edit session of the defaults and close the dialog. Then restart the automatic analysis.

**2452- Attempt to write MICATTR.DIR in read only mode. (file name)**

**2453- Attempt to append MICATTR.DIR in read only mode. (file name)**

*Cause:* The Read-Only attribute is turned on in the application's MICATTR.DIR file (this file exists in each folder containing sample or parameter files).

*Action:* Use Windows Explorer to access the folder containing the MICATTR.DIR file and disable the Read-Only option.

**2454- Too many selections for a print-to-file operation. Only the first (number) selections will be processed. Please reselect the remainder.**

**2455- Too many selections for an export-to-file operation. Only the first (number) selections will be processed. Please reselect the remainder.**

*Cause:* You selected too many files for this operation.

*Action:* Select only the number of files specified in the message.

**2456- Insufficient file handles available. Application cannot continue.**

*Cause:* You have more than 50 files open at the same time.

*Action:* Refer to the manual for your operating system and set the limit for open files to 50 or greater.

**2457- Results cannot be displayed. More than (number) windows are currently displaying or printing results.**

*Cause:* You have too many windows open in the application.

*Action:* Close some of the open windows.

**2458- An instrument is performing a critical operation. Wait a few moments before exiting the application.**

*Cause:* You are attempting to exit the application while the instrument is performing a critical operation. This operation must be completed before the application can be stopped.

*Action:* Wait a short time and attempt to stop the application again.

**2459- An instrument is busy. A delay in restarting this application could result in loss of data. Continue with program Exit? (Yes, No)**

*Cause:* You are attempting to exit the application while an analysis is in progress. While this is possible, the data collected while the application is inactive will not be permanently recorded until the application is restarted. A power failure to the instrument could cause some data to be lost. Continued data collection may also exhaust the memory of the controller.

*Action:* If you are not concerned with the potential for loss of data should a power failure occur, select Yes to continue; otherwise select **No**.

**2460- Fatal Communications error on (Unit n - S/N: nnnn)**

*Cause:* There was a fatal error in the communications between the application and the instrument. All displays for that instrument will be closed.

*Action:* Check that the instrument is connected to the computer on the IP address configured in the Setup program. Stop the 2950 software and then restart it. If this error persists, contact your Micromeritics Service Representative.

**2461-No active instruments. The application will unconditionally terminate.**

*Cause:* At least one instrument must be active for the application to operate. The initialization of all of the instruments configured with the Setup program has failed. The application stops.

*Action A:* Usually this message is preceded by another message giving the reason for the instrument's failure to initialize. See the instructions for that message.

*Action B:* Ensure that the instrument is attached to the Control Module on the IP address configured with the Setup program. Verify that the instrument has the power switch in the ON position and that the light on the front panel is illuminated. If the application continues to fail in its attempts to initialize the instrument, contact your Micromeritics Service Representative.

**2477- Unit n - S/N: nnnn did not properly initialize.**

*Cause:* The software was unable to initialize this instrument; this is usually caused by one of the conditions listed in the error messages above.

*Action:* Correct the problem as described above, then restart the application.

**2478- Error copying sequential data segment.**

*Cause:* An internal processing and/or hardware error occurred while accessing a portion of a sample file.

*Action:* Confirm that the media being accessed does not contain errors; for example, to use a utility such as ScanDisk.

Contact your service representative if you continue to receive this message.

**2479- Unit n - S/N: nnnn The instrument is busy performing an operation of which this application is unaware. Do you want to cancel?**

*Cause:* During initialization, the instrument was found to be analyzing, but the analysis program has no record of it.

*Action:* Click **Yes** to cancel the analysis and synchronize the analysis program with the instrument's status. Click **No** to abort the initialization and allow the analyzer to continue the analysis.

**2480- File <name> cannot be analyzed. It is currently being edited.**

*Cause:* You are trying to start an analysis using a file that is open for editing.

*Action:* Finish editing the file, save and close it, then start the analysis.

**2481- Error accessing the sample information file <name>.**

*Cause:* An unexplained error prevents you from accessing this file.

*Action:* The hard disk drive may be corrupt. Run diagnostics.

**2482- File cannot be opened for writing. It is already in use.**

*Cause:* You attempted to open a file that is currently being used (either by this application or another).

*Action:* Locate the application using the file (in the Micromeritics application, use the Windows menu item to get a list of all windows, one of which may contain this file).

**2483- An analysis cannot be performed on (file name). It is open for editing and contains errors.**

*Cause:* You are trying to start an analysis using a file that contains errors and is open for editing.

*Action:* Finish editing this file, save and close it, then start the analysis.

**2484- The edit session for <name> must be saved before the analysis. Save changes and proceed with the analysis? (yes, no)**

*Cause:* You are trying to start an analysis using a file that contains unsaved changes and is open for editing.

*Action:* Select Yes to save the changes and proceed with the analysis; select No to cancel the analysis and continue editing the Sample Information file.

**2485- The service test file has an invalid status and cannot be used for this analysis.**

*Cause:* The selected service test file has a status other than No Analysis.

*Action:* Select a different service test file or create a new one and use Replace All to copy parameters from the file you originally selected.

**2486- Could not construct (name) report type. Program will terminate.**

**2487- Could not start report generator. Error code (number). Program will terminate.**

*Cause:* An internal processing and/or hardware error has occurred.

*Action:* Contact your service representative if you continue to receive this message.

**2488- File (file name) cannot be opened for editing. It is already in use.**

*Cause:* The file you specified is already open for editing.

*Action:* Check the windows list to locate the other edit session.

**2489- File (file name) cannot be opened for writing. It is already in use.**

*Cause:* The file you specified in a **Save As** operation is already open for edit.

*Action:* Select a different file for the **Save As** operation.

**2490- No '.INI' file present. Application will terminate.**

*Cause:* The ASCII file containing initialization information and system options information used during program startup does not exist.

*Action:* Run the AutoChem Setup program (located on the applications CD), select Change the number of units and enter the pertinent information.

**2491- Highlighted fields contain errors. Please correct the errors before closing.**

*Cause:* The highlighted fields contain invalid entries. You will not be able to close the dialog box until you correct the errors.

*Action:* Check the entries, correct the errors, and close the dialog box.

**2492- This field's entry is invalid.**

*Cause:* The highlighted field contains an invalid entry.

*Action:* Check the entry and correct the error.

**2493- An entry is required for this field.**

*Cause:* This field contains an invalid entry.

*Action:* Enter or select an appropriate value.



**2494- Value is out of the valid range.**

*Cause:* The value you entered in the highlighted field is outside the valid range of values.

*Action:* Check the entry and enter or select a value within the indicated range. The valid range is shown in the information bar across the bottom of the dialog.

**2495- Value is out of the valid range. Enter a value between (value) and (value).**

*Cause:* The value you entered in the highlighted field is outside the valid range of values.

*Action:* Check the entry and enter a value between the range indicated.

**2496- Invalid number.**

*Cause:* The number you entered in the highlighted field is invalid.

*Action:* Check the entry and enter or select a valid number.

**2497- This field contains an invalid character.**

*Cause:* You entered an invalid character in the highlighted field.

*Action:* Check the entry and enter valid characters.

**2498- The requested change to the Sample's status is invalid at this time.**

*Cause:* A request to change the file's status (for example, from automatically collected to manually entered data) could not be done.

*Action:* Contact your service representative if you continue to receive this message. Record the name of the sample file in which the problem occurred.

**2499- Sequence number must contain at least 3 digits.**

*Cause:* You tried to enter a sequence number that did not contain at least three digits.

*Action:* Enter a sequence number that contains at least three digits.

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## 2500 Series

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**2500- All sample files that can be created using the sequence number pattern already exist. You may want to modify the next sequence number.**

*Cause:* No more sample information files can be created using the currently specified file sequence number.

*Action:* Select **Options > Sample defaults** from the main menu and enter a new sequence number.

**2501- System resources have reached a dangerously low level. Please close some windows to avoid loss of data.**

*Cause:* A large number of windows is open and consuming the system resources available to all applications.

*Action:* Close any unneeded windows to free system resources. Contact your service representative if you continue to receive this error message.

**2502- Error writing to file (name) during print. Error code: (number).**

*Cause:* An error occurred in the file being written to during a print operation.

*Action:* Ensure that there is sufficient space on the drive containing the file.

**2503- Error converting file (name). Could not create DIO intermediate file.**

*Cause A:* Insufficient space is available on the hard disk. The DIO file is placed in the directory specified by the TEMP environment variable.

*Action A:* Determine if there is sufficient space on the drive where the TEMP directory is located.

*Cause B:* An internal processing and/or hardware error has occurred.

*Action B:* Contact your service representative.

**2504- Cannot create output file for sample (name).**

*Cause:* Insufficient space may be available on the hard disk.

*Action:* Ensure that sufficient space is available. Contact your service representative if you continue to receive this message.

**2505- Error Logger cannot be initialized. Error code (number). Program will exit.**

*Cause:* An internal processing and/or hardware error has occurred.

*Action:* Contact your service representative.

**2506- (sample file) Output device (name) is not installed. Printing cannot be accomplished.**

*Cause:* The selected output device is not installed.

*Action:* Select a different output device in the System Configuration dialog. Install the device using the Control Panel, Printers operation.

**2508- (sample file) Overlay file (name) was not found. It will not be included in the reports.**

*Cause:* The specified overlay file could not be found.

*Action:* Ensure that the file specified as an overlay does exist.

**2509- (sample file) Error opening file (name): (error). Reports cannot be produced.**

*Cause:* An error occurred while the program was opening a file necessary to the report operation.

*Action:* Use the name given in the error message to investigate. Contact your service representative if you continue to receive this error message.

**2510- (sample file) Error parsing reports from file (name). Reports cannot be produced.**

*Cause A:* One or more data entry fields in the sample file may contain an invalid character (such as a single quote or double quotes).

*Action A:* Review the data entry fields (for example, the Sample field) and remove the invalid character.

*Cause B:* The system was unable to create the usual temporary files during the report, possible due to insufficient disk space.

*Action B:* Check the space available on the hard disk.

*Cause C:* An internal processing error occurred.

*Action C:* Contact your service representative.

**2511- Print job (name) has been cancelled due to insufficient disk space. Delete unnecessary files and restart the report.**

*Cause:* The disk drive does not have enough space for the temporary file required by the Windows Print Manager. Therefore, printing of the requested report has been canceled.

*Action:* Delete unnecessary files from the disk. You will require at least five megabytes of free space for normal operation.

**2512- Print job (name) has been canceled.**

*Cause:* The requested print job was canceled at your request.

*Action:* None required.

**2513- Unable to read the calibration file (file name).**

*Cause:* The calibration file selected for loading is not valid or cannot be read.

*Action:* Confirm the media containing the alleged calibration file has no problems.

**2514- Unable to write the calibration file (file name).**

- Cause:* An attempt to Save calibration data has failed due to possible media problems
- Action A:* Confirm the media containing the alleged calibration file has no problems.
- Action B:* Choose an alternate media to Save the calibration data.

**2515- Warning: Changing the calibration information will affect the performance of the instrument. Only qualified service personnel should do this. Do you wish to proceed?**

- Cause:* You are in the process of performing a calibration.
- Action:* Calibration operations should only be done under the direction of qualified service personnel.

**2516- Warning: Keeping a backup copy of the calibration data is recommended by Micromeritics. Would you like to do so now?**

- Cause:* You have completed a calibration procedure.
- Action:* Save the calibration data to a file.

**2517- Canceling this dialog will reset the calibration state to what it was when this dialog was first opened. Are you sure you want to cancel?.**

- Cause:* You have completed a calibration procedure, but you have not accepted the new calibration data.
- Action:* If the calibration operation was successful, click **Accept**.

**2521- Unable to program controller.**

- Cause:* A hardware malfunction has occurred.
- Action:* Contact your local service representative.

**2522- Invalid controller application file.**

*Cause:* The application's control file has been corrupted or deleted.

*Action:* Reinstall the AutoChem program.

**2523- Programming the controller failed.****2524- CRC check failed on programming controller.****2525- Unknown error programming controller.****2526- Controller download was not successful.****2527- Controller CRC error on boot block.****2528- Controller DRAM error.****2529- Controller Com1: error.****2530- Controller Com2: error.****2531- Controller debug port error.**

*Cause:* An internal processing and/or hardware error has occurred.

*Action:* Contact your service representative if you continue to receive this message.

**2532- The instrument contains a different software version. Do you want to reset it?**

*Cause:* The application has discovered that a different version of software is already running on the instrument.

*Action:* If there is no chance that some instrument other than the 2950 is connected to the instrument, select Yes to reset the instrument and download the proper software. Otherwise, select No to leave the instrument unchanged.

**2533- Mass initialization failed.**

*Cause:* The instrument initialization found that the instrument hardware configuration did not meet the minimum requirements.

*Action:* Contact your service representative.

**2534- Error opening file (name) for printing. Error code: (number)**

*Cause:* An error occurred in the selected file for print output.

*Action:* Ensure that sufficient space is available on the drive containing the file.

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**4200 Series**

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**4200- At least two experiments are needed for this report.**

*Cause:* The report options for this report do not include the selection of at least two experiments.

*Action:* Open the sample file and choose at least two experiments for this report.

**4202- Null connection, unable to start Transaction.****4203- Advise for unknown transaction received.****4204- Unable to create DDE conversation <number>.****4205- Unable to create DDE transaction <number>.****4206- Callback for unknown transaction received.****4207- DDE error number <number>.**

*Cause:* A DDE operation was not properly executed.

*Action:* Ensure that the other application is loaded and in a state which supports DDE operations. If the problem persists, exit all applications and Windows, then restart the applications.

*Cause:* The DDE command was not specified correctly.

*Action:* Review the DDE server application manual for the specific command and enter it correctly in the DDE command step. If the problem persists, exit all applications and Windows, then restart the applications.



**4208- An error occurred while loading the application control information. Data entry cannot be performed. (Code #)**

*Cause:* An error occurred accessing the control information disk file required by this application.

*Action:* The disk drive may have failed or be corrupt. Run diagnostics on the disk drive.

**4209- New steps cannot be inserted in the synopsis at any point that precedes the step that is currently being executed.**

*Cause:* The analysis has already progressed past the point at which you attempted to insert a step; therefore the step would not be performed.

*Action:* Insert the new analysis step after the step that is currently being executed.

**4210- No new steps may be inserted. Analysis is complete.**

*Cause:* The analysis for this sample file has ended. You cannot insert a step in a sample file whose analysis is complete.

*Action:* The analysis conditions for a completed sample cannot be changed. You can, however, create a new sample file and use the **Replace** button to copy the values from the completed file. Then, add the step to the new sample file. A new sample can be analyzed using the new (edited) sample file.

**4213- This analysis can only be executed when Service Test mode is enabled.**

*Cause:* You have attempted to analyze a sample using an analysis conditions set which may only be used under the supervision of an authorized Micromeritics Service Representative. These analysis conditions are provided to assist in the diagnosis of certain instrument problems.

*Action:* Contact your Micromeritics Service Representative for advice on your current problem.

**4214- This analysis exceeds the maximum allowed length.**

*Cause:* The current analysis conditions script contains the maximum number of allowed steps (1000). You cannot add another step.

*Action:* Divide the analysis steps into two or more sample information files. Allow the sample tube to remain undisturbed on the analyzer while you repeatedly start analyses using these sample information files in sequence. When you divide the steps between sample information files, plan your divisions so that the ending conditions of one file and the initial experiment conditions of the next file facilitate the continuing analysis. The data from the various sample information files can be integrated using the Peak Editor, since multiple sample information files can be edited at once. Note that the maximum number of experiments that can be loaded into Peak Editor at one time (regardless of the number of sample files) is 16.

**4219- At least one experiment must be selected and at least one box checked.**

*Cause:* You are attempting to export the collected data from a Sample file, but not all of the required fields on this dialog box have been completed correctly.

*Action:* Select at least one experiment to be exported and check either Peak summaries, Measurements, or both.

**4220- Unexpected I/O error accessing data for an experiment in sample <name>.**

*Cause:* An unexpected disk error occurred while you were attempting to access experiment data.

*Action:* The hard disk drive may have failed or may be corrupted. Run diagnostics.

**4221- Cannot append an experiment to Sample file <name>. Maximum size may be exceeded.**

*Cause:* You selected Save As from the experiment editor and elected to append the loaded experiments to an existing file. Either the existing Sample file already contains the maximum number of allowed experiments or the hard disk is full.

*Action:* Verify that there is at least 20 megabytes of disk space on the target disk drive and that the target Sample file contains fewer than 20 experiments.

**4222- The Active Metal Table Options dialog box does not specify a stoichiometry factor for element <name>. One (1.0) was substituted for this value in the calculations.**

*Cause:* The Pulse Chemisorption report was selected, and requires a valid stoichiometry factor for calculations. The Active Metal Table does not contain a valid stoichiometry factor for the specified element.

*Action:* Open the sample file in Advanced format. Click on the Report Options tab. Select the Pulse Chemisorption report, then click the **Edit** button. Edit the Active Metal Table for this file, specifying a valid stoichiometry factor for the metal named in the error message. Save and close the file, then print the report.

**4223- No element in the Active Metals Table Options has a '% of Sample Weight' value greater than zero.**

*Cause:* The Pulse Chemisorption report was selected. None of the metals in the Active Metal Table are designated as being part of this sample.

*Action:* Edit the Active Metal Table in the Pulse Chemisorption report options and specify a '% of Sample Weight' greater than zero for at least one metal.

**4224- The first peak is larger than the last. This report assumes inverse chemisorption.**

- Cause:* This message appears on the Pulse Chemisorption Report when an inverse chemisorption experiment is reported.
- Action:* None required; informational only. If this experiment is not actually an inverse chemisorption experiment, it is not appropriate for this type of data reduction.

**4225- No reports were selected.**

- Cause:* A report was requested, but there are no reports selected in the sample information file.
- Action:* Open the sample information file and edit the report options. Make sure that at least one report type is selected (double-click on a report to select it; a check mark appears next to each selected report).

**4226- No experiments were selected for inclusion in this report.**

- Cause:* The report type that was selected for printing requires that you specify which experiments should be included in the report. No experiments were selected for this report.
- Action:* Open the sample file in Advanced format. Click on Report Options, then on the selected report type. Click **Edit** and choose the experiments to include in this report. If none of the experiments within the Sample file are applicable to this type of report, deselect the report type from the list shown on the Report Options dialog.

**4227- The data requested on this report are not available.**

- Cause:* The data requested on a report, such as the Auxiliary 1 signal, are not contained within any of the selected experiments.
- Action:* Open the sample file in Advanced format. Click the Report Options tab, then deselect this report type. Or, click the report type, then click the Edit button. When the editing dialog for this report type opens, change the options so that they request data contained within the file.

**4228- This sample does not contain an experiment which can be used in a <name> calibration.**

*Cause:* You are attempting to load a sample which does not have valid peaks. A **User Defined Peaks calibration** will accept any experiment type with valid peaks defined.

*Action:* Select a sample file that contains the appropriate experiment type.

**4229- The calibration data for experiment <name> may already be loaded. You may delete duplicates after loading.**

*Cause:* Before you can reload the peak information for a sample file into the TCD Calibration dialog, you must first delete all previously loaded peaks.

*Action:* Delete all peaks from the dialog, then try loading the sample file again.

**4230- The destination of a Grams/32 export must be a file.**

*Cause:* You selected a destination other than File for exporting in the Grams/32 format.

*Action:* Perform the export operation again and choose File as the destination. You must use a File destination to export Grams/32 data.

**4231- An error occurred exporting experiment <number> from the sample '<file name>'.**

*Cause:* You were attempting to export data to the Grams/32 application when an error occurred. Either the disk file required by the export could not be created or the named experiment contains invalid data.

*Action:* Ensure that sufficient disk space (at least 20 MB) to contain the exported file is available. Also examine the selected experiment with the Peak Editor to ensure that it contains valid data.

**4232- At least two peaks are required to determine heat of desorption.**

*Cause:* The heat of desorption is determined by fitting data obtained at different temperature ramp rates. At least two data points must be present for the fit to be performed.

*Action:* Repeat the analysis with at least two experiments that have different ramp rates. Refer to Appendix J for a tutorial on performing a TPD analysis.

**4233- Default mass spectrometer file directory (path name) is invalid. Resetting to the installation directory.**

*Cause:* A working directory specified in the .INI file is invalid. The directory may have been deleted or moved to a different location.

*Action:* The installation directory will be substituted. The next time you open a file, use the Directories list to move to the correct directory.

**4234- File (name) is not a mass spectrometer data file.**

*Cause:* You selected a file that does not contain appropriate mass spectrometer data.

*Action:* Choose a file that contains data collected by the Pfeiffer Vacuum (formerly Balzers) mass spectrometer using the QuadStar software.

**4235- The mass spectrometer file (file name) cannot be opened for reading.**

*Cause:* A file-access error occurred when you attempted to access the mass spectrometer file.

*Action:* Ensure that you have selected a valid file name, and that you have the required permissions to read mass spectrometer files.

**4236- The calibration data file (name) was not created from an imported mass spec file.**

*Cause:* You attempted to load a calibration file containing data collected on an instrument different from the current one.

*Action:* If selecting a TCD calibration, choose a file containing calibration data collected on the current instrument. If selecting a mass spec calibration, choose a file containing imported mass spec data.

**4237- Peak processing cannot be performed. The mass spectrometer data cannot be imported.**

*Cause:* The mass spectrometer data did not include identifiable peaks.

*Action:* Select a different mass spectrometer file.

**4238- Peak Editor cannot be started.**

*Cause:* You attempted to open the Peak Editor, but one of its components is missing.

*Action:* Reinstall the software.

**4239- A calibration from instrument S/N <number> is required to calibrate experiment <name>.**

*Cause:* In order to associate a calibration file with a Sample file, both files' analyses must have been performed on the same instrument. The instrument must be connected to the control module for its calibration files to be available.

*Action:* Ensure that the instrument with the designated serial number is connected to the computer and is active.

**4240- Error writing calibration information. The calibration of experiment <name> could not be changed.**

*Cause:* The calibration information for an experiment could not be changed because of an error writing to the disk file.

*Action:* The hard disk drive may have failed or it may be corrupt. Run diagnostics. Check that no other application has write access to the sample information file.

**4241- TCD Calibration is required for the calculations of this report.**

*Cause:* This report requires that TCD calibration be applied to the sample file.

*Action:* BET, Langmuir, and Total Pore Volume reports require inclusion of a TCD calibration. Use the Edit Peak view of the Peak Editor to assign a TCD calibration to the file.

**4243- Ambient Temperature should be > 10 °C.**

*Cause:* The value for the ambient temperature is out of range; it is possible that the sample file has been corrupted.

*Action:* Call your Micromeritics service representative.

**4244- Average Syringe Area should be > 0.**

*Cause:* There is no peak data for the Loop calibration.

*Action:* Using the Peak Editor, edit the peaks, inserting new peaks if possible.

**4245- The peak table contains [nn] peaks. Exactly 6 peaks are required for loop calibration.**

*Cause:* The number of peaks for the Loop calibrating is less than six; six are required.

*Action:* Using the Peak Editor, edit the peaks. Insert enough peaks to total six.



**4246- There are no peaks available for this experiment.**

*Cause:* You tried to generate a report on an experiment that has no peaks available.

*Action:* Open the Peak Editor and generate peaks for this experiment.

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## 4300 Series

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**4375- Cannot insert peak with negative height.**

*Cause:* You attempted to insert a peak below the peak set's baseline.

*Action:* Insert the new peak above the peak set's baseline and/or adjust the baseline.

**4376- Auto Fit cannot be executed unless a valid peak-set is selected.**

*Cause A:* You attempted to auto-fit a peak set, but there is no peak set current active.

*Action A:* Click on the peak or peak set in the peak-fit table to select a peak set.

*Cause B:* You attempted to auto-fit, but there are not peak sets in the experiment.

*Action B:* Insert a new peak set into the experiment. Then choose **Auto Fit** to find all peaks in the set.

**4377- This operation cannot be performed on a negative peak set.**

*Cause A:* You attempted to insert a peak in a negative peak set.

*Cause B:* You attempted to move the boundaries of a negative peak set.

*Cause C:* You attempted to change the shape of a peak found within a negative peak set.

*Cause D:* You attempted to optimize the peaks of a negative peak set.

*Cause E:* You attempted to auto-fit the peaks of a negative peak set.

*Action:* Invert the graph to make the peak set positive, then repeat the operation.

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## 6200 Series

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**6209- File cannot be opened for writing. It is already in use.**

*Cause:* You are attempting to access a file that is in use by some other part of the application.

*Action:* Close the window or application which is currently using the file and try again.

**6219- Warning: the instrument (Unit <n> - S/N: nnn) is uncalibrated.**

*Cause:* During initialization, the instrument determined that no calibration data exists for this instrument.

*Action:* Before performing any analysis or experiment, load the calibration information from the factory-supplied disk.

**6221- Unit - S/N: has an invalid status/calibration file and cannot be initialized.**

*Cause:* The status/calibration file, which tracks the status of the instrument, cannot be read by the AutoChem software.

*Action:* Reinstall the AutoChem software to make sure that the file is installed correctly.

**6224- Unit - S/N: has an invalid status/calibration file and cannot be initialized.**

*Cause:* The AutoChem software cannot read the file that tracks the instrument's status.

*Action:* Reinstall the AutoChem software to make sure that the file is set up correctly.

**6226- No instrument calibration is available on Unit n - S/N: nnn.**

*Cause:* The unit could not be initialized because no factory-supplied calibration file has been loaded.

*Action:* Load the factory-supplied calibration file.

**6231- The sample has an invalid status and cannot be used for this analysis.**

*Cause:* Only sample files with a status of **No Analysis** can be used for analysis. The sample file you have selected has a status other than **No Analysis**.

*Action:* Select a sample file with a status of **No Analysis**.

**6232- Warning: The furnace is open, and sample/furnace temperature is above ambient. Do you want to continue? (Yes, No)**

*Cause:* The sample's temperature (or that of the furnace) was above ambient, and/or the furnace was open when you attempted to start the analysis.

*Action:* If the furnace/temperature condition is correct, click Yes. Otherwise, click No, correct the problem, then restart the analysis.

**6233- Warning: The furnace is open. Do you want to continue? (Yes, No)**

*Cause A:* You tried to start an analysis with the furnace door open, or the furnace is not connected to the analyzer.

*Action A:* If the furnace condition is correct, click Yes. Otherwise click No, correct the problem, and restart the analysis.

*Cause B:* You tried to start an analysis with the furnace door closed, but the interlock switch inside the furnace has failed.

*Action B:* Contact a Micromeritics Service Representative.

**6234- Warning: The sample/furnace temperature is above ambient. Do you want to continue? Yes, No**

*Cause:* The sample's temperature (or that of the furnace) was above ambient when you attempted to start the analysis.

*Action:* If the sample's temperature is correct, click Yes. Otherwise, click No, correct the problem, and restart the analysis.

**6264- Warning: The CryoCooler needs to be re-filled. Do you want to continue? (yes, no)**

*Cause:* You are starting an analysis with a low level of liquid in the CryoCooler.

*Action:* Select **Yes** to proceed with the analysis if the CryoCooler is not required. Select **No**, refill the CryoCooler, and restart the analysis if the CryoCooler is needed for this analysis.

**6267- Inter-application message cannot be delivered. Select Continue to resume analysis.**

**6268- Analysis paused. An inter-application message could not be delivered.**

**6269- Analysis paused. An inter-application message failed. The Receiver was busy.**

**6270- Analysis paused. An inter-application message failed. The Receiver refused the request.**

**6271- Analysis paused. An inter-application message failed for an unknown reason.**

*Cause A:* A DDE operation was not properly executed.

*Action A:* Ensure that the other application is loaded and in a state which supports DDE operations. If the problem persists, exit all applications and Windows, then restart the applications.

*Cause B:* The DDE command was not specified correctly.

*Action B:* Review the DDE server application manual for the specific command and enter it correctly in the DDE command step. If the problem persists, exit all applications and Windows, then restart the applications.

**6273- Warning. For the chosen total flow rate and blending proportions, the carrier and/or loop MFC will be set to a flow rate less than 10 cm<sup>3</sup>/min. Blending performance may not be acceptable. Do you wish to keep these settings? (Yes, No)**

*Cause:* You requested blending of the carrier and loop gasses, but the proportions of the gasses and the flow rate of the mixture chosen caused at least one of the MFCs to be set to a rate less than 10 cm<sup>3</sup>/min. The accuracy of the MFC at such low flow rates may be unacceptable for some applications.

*Action:* Click **Yes** if reduced accuracy is acceptable. Click *No* and change the proportions or total flow rate.

**6275- Instrument (Unit, S/N) initialization failure (code)**

*Cause:* An internal error has occurred.

*Action:* Record the contents of the error message, then contact your Micromeritics service representative.

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## 6300 Series

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**6300- Warning. The back pressure heat zone has been reporting an invalid temperature for one minute.**

*Cause A:* Heater cable is loose or disconnected.

*Action A:* Connect the heater cable.

*Cause B:* There is a failure in the hardware or electronics.

*Action B:* Establish an Idle state in the instrument, turn the instrument power OFF, then turn it back ON. Allow the software to be initialized. If the problem recurs after the software has been active for about one minute, contact your Micromeritics Service Representative.

**6301- Warning. The Back Pressure rotary valve did not operate correctly.**

*Cause:* There is a failure in the hardware or electronics.

*Action:* Establish an Idle state in the instrument, turn the instrument power OFF, then turn it back ON. Allow the software to be initialized. If the problem recurs after the software has been active for about one minute, contact your Micromeritics Service Representative.

**6302- Warning. Calibration of the back pressure heat zone failed. Previous calibration will be used.**

*Cause:* An attempt to manually control the Back Pressure valve resulted in incorrect operation, possibly due to a mechanical problem.

*Action:* Try the operation again 2-3 times. If the problem persists, lower the temperature of the associated heat zone and then try the operation again. If this is not successful, exit the software program and then turn the analyzer's power off. After a few seconds, turn the analyzer's power back on and then restart the software program. The valve should be re-initialized. Check that the valve moves freely using Manual Control. If the valve is still not operating correctly, contact a Micromeritics Service Representative.

**6303- Warning. An attempt to operate a valve has been prevented due to high pressure (code [n] of [n]),**

*Cause A:* The measured temperature when the high calibration value was entered was less than the measured temperature when the low calibration value was entered.

*Action A:* Repeat the calibration, ensuring that the entered temperatures are accurate.

*Cause B:* The temperature is being measured improperly by the instrument.

*Action B:* Contact your Micromeritics service representative if you continue to receive this message.

**6304- Warning. A back pressure operation time-out has occurred.**

*Cause A:* More than 30 minutes has elapsed since the beginning of a back-pressure operation without the pressure target being reached.

*Action A:* Check the gas regulator pressure.

**6305- Error. Operation suspended. The back pressure zone has been reporting an invalid temperature for one minute.**

*Cause A:* Heater cable is loose or disconnected.

*Action A:* Connect the heater cable.

*Cause B:* A failure has occurred in the hardware or electronics.

*Action B:* Establish an Idle state in the instrument, turn the instrument power OFF, then turn it back ON. Allow the controller software to be initialized. If the problem recurs after the software has been active for about one minute, contact your Micromeritics Service Representative.

If you are able to correct the problem, you may resume the analysis; otherwise, cancel it. Before you resume the analysis, you must reestablish the TCD temperature using Manual Control. After you enable Manual Control, click on the detector heat zone and set the temperature to the correct value.

*Cause C:* The heat zone is calibrated incorrectly.

*Action C:* Contact your Micromeritics Service Representative.



**6306- Error. Operation suspended. The Back Pressure rotary valve did not operate correctly.**

*Cause:* An attempt to control the back pressure valve during an analysis resulted in an incorrect operation, possibly due to a mechanical problem. If analysis is resumed before the problem is corrected, the current step will NOT be completed.

*Action:* Use manual control to try to operate the valve. If the problem persists, lower the temperature of the associated heat zone and then try the operation again. If this is not successful, exit the software program and then turn the analyzer's power off. After a few seconds, turn the analyzer's power back on and then restart the software program. The valve should be re-initialized. Check that the valve moves freely using Manual Control. Restart the analysis. If the problem still persists, contact a Micromeritics Service Representative.

**6307- Error. Operation suspended. An attempt to operate a valve has been prevented due to high pressure ([n] of [n]).**

*Cause:* While a portion of the analyzer was at an elevated pressure an attempt was made to change a valve in the elevated region.

*Action:* Do not attempt to operate a valve when it is in the elevated pressure region.

**6308- Error. Operation suspended. A back pressure operation time-out occurred.**

*Cause A:* More than 30 minutes has elapsed since the beginning of a back-pressure operation without the pressure target being reached.

*Action A:* Check the gas regulator pressure.

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## 6400 Series

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**6400- Warning. The flow sensed by the carrier flow sensor is below 5 cm<sup>3</sup>/min.**

*Cause:* The gas flow in the analysis path carrier gas plumbing is inadequate. Either it is blocked, or the gas source (its associated valves and gas supplies) is not activated.

*Action:* Ensure that the analysis gas Mass Flow Controller is set for a flow greater than 5 cm<sup>3</sup>/min., that one of its valves is open, and that the gas supply to the open valve is operating correctly.

**6402- Warning. The flow of the carrier Mass Flow Controller is below 5 cm<sup>3</sup>/min.**

*Cause:* The gas flow in the carrier gas plumbing is inadequate. Either it is blocked, or the gas source (its associated valves and gas supplies) is not activated.

*Action:* Ensure that the carrier Mass Flow Controller is set for a flow greater than 5 cm<sup>3</sup>/min., that one of its valves is open, and that the gas supply to the open valve is operating correctly.

**6404- Warning. The flow of the loop Mass Flow Controller is below 5 cm<sup>3</sup>/min**

*Cause:* The gas flow in the analysis gas plumbing is inadequate. Either it is blocked, or the gas source (its associated valves and gas supplies) is not activated.

*Action:* Ensure that the analysis Mass Flow Controller is set for a flow greater than 5 cm<sup>3</sup>/min., that one of its valves is open, and that the gas supply to the open valve is operating correctly.

**6405- Warning. The flow of the carrier Mass Flow Controller differs from the target flow by 10% or more.**

*Cause:* The gas flow in the carrier gas plumbing is inadequate. Either it is blocked, or the gas source (its associated valves and gas supplies) is not activated.

*Action:* Ensure that the carrier Mass Flow Controller is set for a flow greater than 5 cm<sup>3</sup>/min., that one of its valves is open, and that the gas supply to the open valve is operating correctly.

**6407- Warning. The flow of the loop Mass Flow Controller differs from the target by 10% or more.**

*Cause:* The gas flow in the loop gas plumbing is inadequate. Either it is blocked, or the gas source (its associated valves and gas supplies) is not activated.

*Action:* Ensure that the loop Mass Flow Controller is set for a flow greater than 5 cm<sup>3</sup>/min., that one of its valves is open, and that the gas supply to the open valve is operating correctly.

**6408- Warning. The detector heat zone has been reporting an invalid temperature (reading) for one minute.****6409-Warning. The loop heat zone has been reporting an invalid temperature (reading) for one minute.****6410- Warning. The analysis heat zone has been reporting an invalid temperature (reading) for one minute.****6411- Warning. The cold trap heat zone has been reporting an invalid temperature (reading) for one minute.**

*Cause A:* Heater cable is loose or disconnected.

*Action A:* Connect the heater cable.

*Cause B:* There is a failure in the hardware or electronics.

*Action B:* Establish an Idle state in the instrument, turn the instrument power OFF, then turn it back ON. Allow the software to be initialized. If the problem recurs after the software has been active for about one minute, contact your Micromeritics Service Representative.

**6415- Warning. The furnace thermocouple is not connected.**

*Cause A:* The cable connecting the furnace thermocouple to the front panel of the instrument has been disconnected.

*Action A:* Connect the cable (the connector is shaped so that it can only go in one way).

*Cause B:* There is a failure in the hardware or electronics.

*Action B:* Contact your Micromeritics Service Representative.

**6416- Warning. The sample thermocouple is not connected or operating properly.**

*Cause:* Either the sample thermocouple is not connected, or it is not operating properly.

*Action:* Remove the top cover of the instrument and inspect the sample thermocouple cable to insure that it is properly connected. If everything appears correct, substitute a different thermocouple. If the problem persists, there is a failure in the hardware or electronics. Contact a Micromeritics Service Representative.

**6417- Warning. System pressure has been above the allowed limit for more than 10 seconds.****6418- Warning. System pressure has been above the tolerance level for more than one minute.**

*Cause:* Gas flow is restricted at some point in the system.

*Action:* Examine the instrument schematic display to determine the current flow path from the carrier Mass Flow Controller, then examine the instrument's components that correspond to the flow path. Use Manual Control to identify the location of the restriction. A typical source of blockage is a sample that is too tightly packed in the tube. Clear the restriction, if possible. If necessary, contact a Micromeritics Service Representative.

**6419- Warning. Manual Control of the furnace is not supported when the Sample Temperature system is active.**

*Cause:* The operator has attempted a Manual Control operation during an automatic analysis. The current step involves the sample temperature system.

*Action:* Do not attempt manual operations during an analysis. To alter the sample temperature system manually during the analysis, suspend the analysis, perform the operation, then resume the analysis. (The current step is skipped and the analysis is resumed on the next step.)

**6420- Warning. Manual Control of a flow-related item is not supported when the Flow Management system is active.**

*Cause:* You have attempted a Manual Control operation during an automatic analysis. The current step involves a flow-related item.

*Action:* Do not attempt manual operations during an analysis. To alter a flow-related item manually during the analysis, suspend the analysis, perform the operation, then resume the analysis. (The current step is skipped and the analysis is resumed on the next step.)

**6421- Warning. Manual Control of a TCD-related item is not supported when the Signal Management system is active.**

*Cause:* You have attempted a Manual Control operation during an automatic analysis. The current step involves a TCD-related item.

*Action:* Do not attempt manual operations during an analysis. To alter a TCD-related item manually during the analysis, suspend the analysis, perform the operation, then resume the analysis. (The current step is skipped and the analysis is resumed on the next step.)

**6422- Warning. Manual Control of a heat zone is not supported when the Signal Management system is active.**

*Cause:* You have attempted a Manual Control operation during an automatic analysis. The current step involves a heat zone.

*Action:* Do not attempt manual operations during an analysis. To alter a heat zone manually during the analysis, suspend the analysis, perform the operation, then resume the analysis. (The current step is skipped and the analysis is resumed on the next step.)

**6425- Error. Operation suspended. The flow of the carrier Mass Flow Controller is below 5 cm<sup>3</sup>/min.**

*Cause:* The gas flow in the carrier gas plumbing is inadequate. Either it is blocked, or the gas source (its associated valves and gas supplies) is not activated.

*Action:* Ensure that the carrier Mass Flow Controller is set for a flow greater than 5 cm<sup>3</sup>/min., that one of its valves is open, and that the gas supply to the open valve is operating correctly.

If you are able to correct the problem, you may resume the analysis; otherwise, cancel it. Before you resume the analysis, you must re-establish the TCD temperature using Manual Control. After you enable Manual Control, click on the detector heat zone and set the temperature to the correct value.

**6427- Error. Operation suspended. The flow of the prep Mass Flow Controller differs from the target by 10% or more.**

*Cause:* The gas flow in the prep plumbing is inadequate due to either a blockage or the gas source (its associated valves and gas supplies) is not activated.

*Action:* Ensure that the prep Mass Flow Controller is set for a flow, that one of its associated valves is open, and that the gas supply to the open valve is operating correctly.

If you are able to correct the problem, you may resume the analysis; otherwise, cancel it. Before you resume the analysis, you must re-establish the TCD temperature using Manual Control. After you enable Manual Control, click on the detector heat zone and set the temperature to the correct value.

**6428- Error. Operation suspended. The flow of the carrier Mass Flow Controller differs from the target by 10% or more.**

*Cause:* The gas flow in the carrier plumbing is inadequate due to either a blockage or the gas source (its associated valves and gas supplies) is not activated.

*Action:* Ensure that the carrier Mass Flow Controller is set for a flow, that one of its associated valves is open, and that the gas supply to the open valve is operating correctly.

If you are able to correct the problem, you may resume the analysis; otherwise, cancel it. Before you resume the analysis, you must re-establish the TCD temperature using Manual Control. After you enable Manual Control, click on the detector heat zone and set the temperature to the correct value.

**6430- Error. Operation suspended. The flow of the loop Mass Flow Controller differs from the target by 10% or more.**

*Cause:* The gas flow in the loop gas plumbing is inadequate. Either it is blocked, or the gas source (its associated valves and gas supplies) is not activated.

*Action:* Ensure that the loop Mass Flow Controller is set for a flow greater than 5 cm<sup>3</sup>/min., that one of its valves is open, and that the gas supply to the open valve is operating correctly.

If you are able to correct the problem, you may resume the analysis; otherwise, cancel it. Before you resume the analysis, you must re-establish the TCD temperature using Manual Control. After you enable Manual Control, click on the detector heat zone and set the temperature to the correct value.

**6431- Error. Operation suspended. The detector heat zone has been reporting an invalid temperature (reading) for one minute.**

**6432- Error. Operation suspended. The loop heat zone has been reporting an invalid temperature (reading) for one minute.**

**6433- Error. Operation suspended. The analysis heat zone has been reporting an invalid temperature (reading) for one minute.**

**6434- Error. Operation suspended. The cold trap heat zone has been reporting an invalid temperature (reading) for one minute.**

*Cause A:* Heater cable is loose or disconnected.

*Action A:* Connect the heater cable.

*Cause B:* A failure has occurred in the hardware or electronics.

*Action B:* Establish an Idle state in the instrument, turn the instrument power OFF, then turn it back ON. Allow the controller software to be initialized. If the problem recurs after the software has been active for about one minute, contact your Micromeritics Service Representative.

If you are able to correct the problem, you may resume the analysis; otherwise, cancel it. Before you resume the analysis, you must reestablish the TCD temperature using Manual Control. After you enable Manual Control, click on the detector heat zone and set the temperature to the correct value.

*Cause C:* The heat zone is calibrated incorrectly.

*Action C:* Contact your Micromeritics Service Representative.

**6438- Error. Operation suspended. The furnace thermocouple is not connected.**

*Cause A:* The cable connecting the furnace thermocouple to the front panel of the instrument has been disconnected.

*Action A:* Connect the cable (the connector is shaped so that it cannot be improperly inserted).

*Cause B:* There is a failure in the hardware or electronics.

*Action B:* Contact your Micromeritics Service Representative.

If you are able to correct the problem, you may resume the analysis; otherwise, cancel it. Before you resume the analysis, you must reestablish the TCD temperature using Manual Control. After you enable Manual Control, click on the detector heat zone and set the temperature to the correct value.



**6439- Error. Operation suspended. The sample thermocouple is not connected or operating properly.**

*Cause:* Either the sample thermocouple is improperly connected or it is not functioning properly.

*Action:* Remove the top cover of the instrument and inspect the sample thermocouple cable to ensure that it is properly connected. If the connect appears correct, substitute a different thermocouple. If the problem persists, there is a failure in the hardware or electronics. Contact a Micromeritics Service Representative.

If you are able to correct the problem, you may resume the analysis; otherwise, cancel it. Before you resume the analysis, you must re-establish the TCD temperature using Manual Control. After you enable Manual Control, click on the TCD heat zone and set the temperature to the correct value.

**6440- Error. Operation suspended. System pressure has been above the allowed limit for more than 10 seconds.**

*Cause:* Gas flow has been restricted at some point in the system.

*Action:* Examine the instrument schematic display to determine the current flow path from the carrier Mass Flow Controller, then examine the instrument's components that correspond to the flow path. Use Manual Control to identify the location of the restriction. A typical source of blockage is a sample that is too tightly packed in the tube. Clear the restriction, if possible. If necessary, contact a Micromeritics Service Representative.

If you are able to correct the problem, you may resume the analysis; otherwise, cancel it. Before you resume the analysis, you must re-establish the TCD temperature using Manual Control. After you enable Manual Control, click on the detector heat zone and set the temperature to the correct value.

**6441- Error. Operation suspended. An invalid instruction has been encountered (code # at # in experiment # after step #).**

*Cause:* A software error has occurred.

*Action:* Carefully record the entire text of this error message. On a diskette or network drive, save a copy of the sample file being analyzed. Contact your Micromeritics Service Representative.

**6442- Error. Operation suspended. After 200 attempts, the instrument has failed to detect two consecutive peaks of equal area.**

**6443- Error. Operation suspended. After 200 attempts, the instrument has failed to detect two consecutive peaks of zero area.**

*Cause:* The sample tube contains little or no sample, or the sample is not reacting with the analysis gas.

*Action:* If you are able to correct the problem, you may resume the analysis; otherwise, cancel it. Before you resume the analysis, you must re-establish the TCD temperature using Manual Control. After you enable Manual Control, click on the detector heat zone and set the temperature to the correct value.

**6444- Error. Operation suspended. A signal wait time-out has occurred.**

*Cause:* The time allowed for a Wait step expired before the condition(s) of the Wait step were met.

*Action:* Examine the analysis conditions for this analysis to determine if sufficient time was allowed for the conditions of this Wait step to be met. Determine if the analysis should proceed. You may either resume the analysis or cancel it. Before you resume the analysis, you must re-establish the TCD temperature using Manual Control. After you enable Manual Control, click on the detector heat zone and set the temperature to the correct value.

**6445- Error. Operation suspended. A sample temperature operation time-out has occurred.**

*Cause:* The time allowed for a sample temperature operation to be complete expired before the target temperature was reached. The control software calculates a time limit based upon the ramp rate and the difference between the current temperature and the target temperature.

*Action:* Examine the current sample temperature. If it is acceptable for the purposes of the current analysis, you may choose to resume the analysis; otherwise, cancel it. Before you resume the analysis, you must re-establish the TCD temperature using Manual Control. After you enable Manual Control, click on the detector heat zone and set the temperature to the correct value.

**6446- Error. Operation suspended. A gas flow operation time-out has occurred.**

*Cause:* Stable gas flow in a Mass Flow Controller was not achieved within two minutes.

*Action:* Ensure that the gas supply for the open valve associated with the Mass Flow Controller is set to the correct pressure. Use Manual Control to detect the cause of the problem.

If you are able to correct the problem, you may resume the analysis; otherwise, cancel it. Before you resume the analysis, you must re-establish the TCD temperature using Manual Control. After you enable Manual Control, click on the detector heat zone and set the temperature to the correct value.

**6447- Error. Operation suspended. An invalid parameter has been encountered (code # #).****6448- Error. Operation suspended. The requested script (ID = #) is not located in the instrument.****6449- Error. Operation suspended. There is insufficient room for the script (ID = #) (error code #).**

*Cause:* A software error has occurred.

*Action:* Carefully record the entire text of this error message. On a diskette or network drive, save a copy of the sample file being analyzed. Contact your Micromeritics Service Representative.

**6450- Warning. The requested gas flow is not possible because all inlet valves for this MFC are closed.**

*Cause:* A Manual control operation to set a gas flow in a Mass Flow Controller was requested, but all associated valves are closed.

*Action:* Use Manual Control to open the gas valve, then repeat the Set Flow Rate operation.

**6452- Error. Operation suspended. Sample temperature ramping/control has failed because the actual sample temperature differs from the temperature control element by more than 100 degrees C.**

*Cause:* The sample temperature has differed significantly from that of the furnace for at least 10 minutes.

*Action:* Ensure that the sample thermocouple is properly installed and that the furnace is closed.

If you are able to correct the problem, you may resume the analysis; otherwise, cancel it. Before you resume the analysis, you must re-establish the TCD temperature using Manual Control. After you enable Manual Control, click on the detector heat zone and set the temperature to the correct value.

**6453- Error. Operation suspended. An operation was requested that is not currently valid.**

*Cause:* A software error has occurred.

*Action:* Carefully record the entire text of this error message. On a diskette or network drive, save a copy of the sample file being analyzed. Contact your Micromeritics Service Representative.

**6454- Error. Operation suspended. The flow control interface board (channel <number>) was not ready within the allowed time.**

**6455- Error. Operation suspended. The heat zones interface board (channel <number>) was not ready within the allowed time.**

*Cause:* A hardware error has occurred.

*Action:* Carefully record the entire text of this error message. Save a copy of the sample file being analyzed on a diskette or network drive and contact your Micromeritics Service Representative.

**6456- Error. Operation suspended. A heat zone <code number> did not reach its target of <number> (current temperature = <number>) within the allowed time of <number> minutes.**

**6457- Error. Operation suspended. The furnace did not reach a temperature between <number> and <number> in the allowed time of <number> minutes.**

**6458- Error. Operation suspended. Sample temperature equilibration is not allowed because sample temperature control is not active (code).**

*Cause:* A hardware error has occurred.

*Action:* Carefully record the entire text of this error message. On a diskette or network drive, save a copy of the sample file being analyzed. Contact your Micromeritics Service Representative.

If you are able to correct the problem, you may resume the analysis; otherwise, cancel it. Before you resume the analysis, you must re-establish the TCD temperature using Manual Control. After you enable Manual Control, click on the detector heat zone and set the temperature to the correct value.

**6459- Warning. A gas flow blockage has been detected, return to ambient pressure is being done.**

*Cause:* The difference in pressure readings of the analyzer's pressure transducer and the back-pressure controller's transducer has been above 50 psia for more than 10 seconds.

*Action:*



The analysis is canceled and the pressure control is disabled to allow excess gas to vent from the system. Take extreme caution before removing the sample tube. Excess pressure may be stored in the sample tube and may present a dangerous condition. Appropriate personal protection should be used when removing the sample tube.

**6460- Operation canceled. A gas flow block age has been detected, return to ambient pressure is being done.**

*Cause:* The difference in pressure readings of the analyzer's pressure transducer and the back-pressure controller's transducer has been above 50 psia for more than 10 seconds.

*Action:*



The analysis is canceled and the pressure control is disabled to allow excess gas to vent from the system. Take extreme caution before removing the sample tube. Excess pressure may be stored in the sample tube and may present a dangerous condition. Appropriate personal protection should be used when removing the sample tube.

**6461- Operation canceled. The furnace was opened during an analysis, return to atmospheric pressure is being done.**

*Cause:* The furnace door was opened while an analysis was active.

*Action:*



The analysis is canceled and the pressure control is disabled to allow excess gas to vent from the system. Take extreme caution before removing the sample tube. Excess pressure may be stored in the sample tube and may present a dangerous condition. Appropriate personal protection should be used when removing the sample tube.

**6463- Warning. The Cold Trap rotary valve did not operate correctly.**

*Cause:* An attempt to manually control the Cold Trap valve resulted in incorrect operation, possibly due to a mechanical problem.

*Action:*

Try the operation again 2-3 times. If the problem persists, lower the temperature of the associated heat zone and then try the operation again. If this is not successful, exit the software program and then turn the analyzer's power off. After a few seconds, turn the analyzer's power back on and then restart the software program. The valve should be re-initialized. Check that the valve moves freely using Manual Control. If the valve is still not operating correctly, contact a Micromeritics Service Representative.

**6464- Warning. The Preparation rotary valve did not operate correctly.**

*Cause:* An attempt to manually control the Preparation (also known as the Analysis) valve resulted in incorrect operation, possibly due to a mechanical problem.

*Action:* Try the operation again 2-3 times. If the problem persists, lower the temperature of the associated heat zone and try the operation again. If this is unsuccessful, exit the program and turn the analyzer's power off. After a few seconds, turn the analyzer's power back on and restart the program. The valve should be reinitialized. Check that the valve moves freely using Manual Control. If the valve is still not operating correctly, contact a Micromeritics Service Representative.

**6465- Warning. The Inject rotary valve did not operate correctly.**

*Cause:* An attempt to manually control the Inject (also known as the loop) valve resulted in an incorrect operation, possibly due to a mechanical problem.

*Action:* Try the operation again 2-3 times. If the problem persists, lower the temperature of the associated heat zone and try the operation again. If this is unsuccessful, exit the program and then turn the analyzer's power off. After a few seconds, turn the analyzer's power back on and restart the program. The valve should be reinitialized. Check that the valve moves freely using Manual Control. If the valve is still not operating correctly, contact a Micromeritics Service Representative.

**6467- Warning. The KwikCool option is not installed.**

- Cause:* An attempt to control manually the KwikCool option failed because the KwikCool option was not detected at the time the control was attempted. If analysis is resumed before the problem is corrected, the current step will NOT be completed.
- Action:* Confirm that the KwikCool option cable is properly connected to the analyzer. It is recommended that the power to the analyzer be turned off when you attach the KwikCool connector to the analyzer (or detach it). Once again, use Manual Control to attempt the KwikCool operation. If the problem persists, you may choose to continue operations without using the KwikCool. Or, you may try exiting the software program and then turning the analyzer's power off. After a few seconds, turn the analyzer's power back on and then restart the software program. If the problem cannot be corrected, contact a Micromeritics Service Representative.

**6468- Error. Operation suspended. The Cold Trap rotary valve did not operate correctly.**

- Cause:* An attempt to control the Cold Trap valve during an analysis resulted in an incorrect operation, possibly due to a mechanical problem. If analysis is resumed before the problem is corrected, the current step will NOT be completed.
- Action:* Use manual control to try to operate the valve. If the problem persists, lower the temperature of the associated heat zone and then try the operation again. If this is not successful, exit the software program and then turn the analyzer's power off. After a few seconds, turn the analyzer's power back on and then restart the software program. The valve should be re-initialized. Check that the valve moves freely using Manual Control. Restart the analysis. If the problem still persists, contact a Micromeritics Service Representative.



**6469- Error. Operation suspended. The Preparation rotary valve did not operate correctly.**

*Cause:* An attempt to control the Preparation (also known as the Analysis) valve during an analysis resulted in an incorrect operation, possibly due to a mechanical problem. If analysis is resumed before the problem is corrected, the current step will NOT be completed.

*Action:* Use manual control to try to operate the valve. If the problem persists, lower the temperature of the associated heat zone and then try the operation again. If this is not successful, exit the software program and then turn the analyzer's power off. After a few seconds, turn the analyzer's power back on and then restart the software program. The valve should be re-initialized. Check that the valve moves freely using Manual Control. Restart the analysis. If the problem still persists, contact a Micromeritics Service Representative.

**6470- Error. Operation suspended. The Inject rotary valve did not operate correctly.**

*Cause:* An attempt to control the Inject (also known as the loop valve) during an analysis resulted in an incorrect operation, possibly due to a mechanical problem. If analysis is resumed before the problem is corrected, the current step will NOT be completed.

*Action:* Use manual control to try to operate the valve. If the problem persists, lower the temperature of the associated heat zone and then try the operation again. If this is not successful, exit the software program and then turn the analyzer's power off. After a few seconds, turn the analyzer's power back on and then re-start the software program. The valve should be re-initialized. Check that the valve moves freely using Manual Control. Restart the analysis. If the problem still persists, contact a Micromeritics Service Representative.

**6472- Error. Operation suspended. The KwikCool option is not installed.**

*Cause:* An attempt to operate the KwikCool option during an analysis failed because the KwikCool option was not detected at the time the operation was attempted. If analysis is resumed before the problem is corrected, the analysis will continue without the KwikCool.

*Action A:* Confirm that the KwikCool option cable is properly connected to the analyzer. It is recommended that the power to the analyzer be turned off when you attach the KwikCool to the analyzer (or when you detach it).

*Action B:* Use Manual Control to attempt the KwikCool operation that was requested during the analysis. If this is not successful, exit the software program and then turn the analyzer's power off. After a few seconds, turn the analyzer's power back on and then restart the software program. Restart the analysis. If the problem persists, contact your Micromeritics Service Representative.

**6473- Warning. The CryoCooler option is not installed.**

*Cause:* You attempted to change the CryoCooler's settings but the CryoCooler was not detected.

*Action A:* Verify that the CryoCooler is physically connected to the instrument and that the retaining screws on the connector are securely tightened.

*Action B:* Exit the 2950 application, turn off the instrument, then restart the application. If the CryoCooler still does not respond, contact your Micromeritics service representative.

**6474- Warning. The CryoCooler is almost empty. Temperature control may not be stable.**

*Cause:* The liquid level in the CryoCooler is low. It may be impossible to achieve subambient temperatures unless cryogen is added.

*Action:* If the current analysis requires subambient temperatures, suspend the analysis, refill the CryoCooler with liquid nitrogen, then resume the analysis.

**6475- Warning. Manual control of the CryoCooler is not supported when the Sample Temperature system is active.**

*Cause:* You attempted to change the CryoCooler's settings while a temperature ramp was in progress.

*Action:* Wait until the temperature ramp is finished before adjusting the CryoCooler's settings.

- 6476- Warning. Calibration of the detector heat zone failed. The previous calibration will be used.**
- 6477- Warning. Calibration of the loop heat zone failed. The previous calibration will be used.**
- 6478- Warning. Calibration of the analysis heat zone failed. The previous calibration will be used.**
- 6479- Warning. Calibration of the cold trap heat zone failed. The previous calibration will be used.**
- 6483- Error. analysis cannot proceed. System pressure has been above the allowed limit for more than 10 seconds.**

*Cause:* Gas flow has been restricted at some point in the system.

*Action:* Examine the instrument schematic display to determine the current flow path from the carrier Mass Flow Controller, then examine the instrument's components that correspond to the flow path. Use Manual Control to identify the location of the restriction. A typical source of blockage is a sample that is too tightly packed in the tube. Clear the restriction, if possible. If necessary, contact a Micromeritics Service Representative.

If you are able to correct the problem, you may resume the analysis; otherwise, cancel it. Before you resume the analysis, you must re-establish the TCD temperature using Manual Control. After you enable Manual Control, click on the detector heat zone and set the temperature to the correct value.

**6485- Instrument is connected to a Eurotherm furnace.**

*Cause:* A Eurotherm furnace is erroneously plugged into the instrument that has a new style CPU card..

*Action:* Contact your Micromeritics Service Representative.



## J. TUTORIALS

This appendix contains tutorials for performing a:

- typical analysis, page [J-1](#)
- TPR analysis, page [J-10](#)
- TPD analysis, page [J-14](#)
- TPO analysis, page [J-19](#)
- BET analysis, page [J-23](#)
- Pulse chemisorption analysis, page [J-32](#)
- Loop calibration, page [J-38](#)

### Typical Analysis

The instructions in this section are provided to familiarize you with the software and analyzer. More detailed instructions for performing each analysis type are found later in this appendix.

Analysis conditions sets are included with the software for TPR, TPO, TPD, Pulse Chemisorption, and BET experiments. For this tutorial, you may perform any one of these analyses, using any sample you choose. Using a reference sample is recommended.

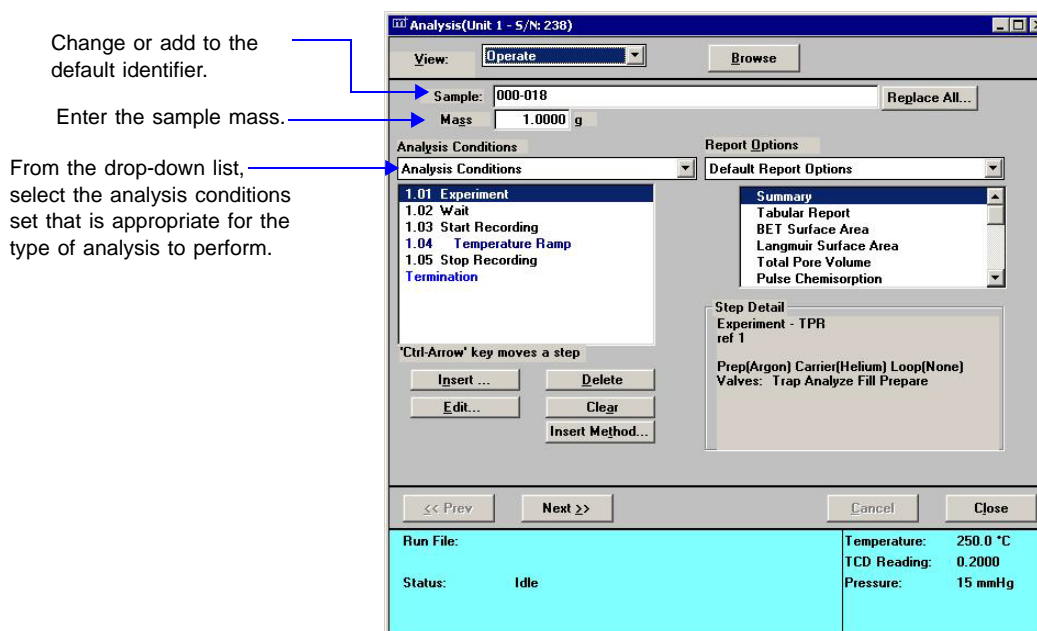


**As always, the operator is responsible for making sure that safe combinations of samples and gases are used.**

In this example, we use Cupric Oxide (15-20 mg) to perform a TPR experiment. This analysis uses a mixture of hydrogen with argon as the carrier gas. No loop gas is used.

1. Prepare and determine the mass of the sample, then load it on the analyzer. Make other preparations for the type of analysis you plan to perform (such as preparing the cold trap and Dewar, and positioning the thermocouple) as directed in Chapter 3.
2. Make sure that the correct gases are connected. Also check to be sure that the Gas Selections table (on the Unit menu) accurately reflects the current gas connections.
3. Select **Unit > Start Analysis**; the Analysis dialog sequence opens. You are first presented a dialog from which to choose a sample file. In this example, you are creating a new sample file; click **OK** to accept the default.

- Click **Yes** to create the file. The Analysis dialog opens containing the sample file you are creating; all fields contain default values. Complete the dialog as shown below. (Basic sample file format is shown; Advanced format may also be used.)

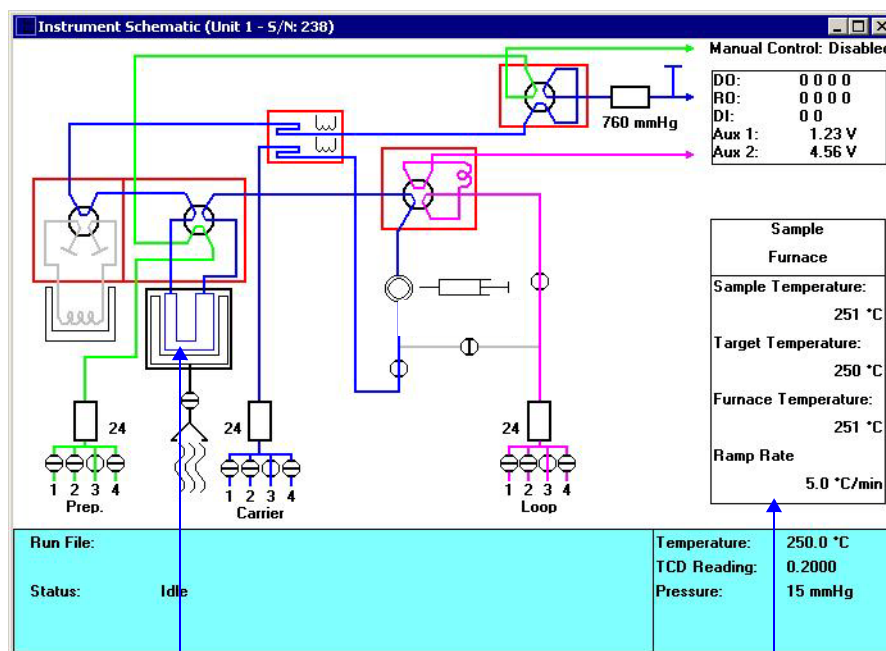


- Click **Next** three times to accept the calibration and reports defaults (although these can be changed when you are performing your own analyses) and to verify the caution information.
- Click **Start** and the analyzer begins the experiment.

## Observe the Analysis

The Analysis window contains four different views in which to observe the progress of your analysis. You select the view from the View drop-down list. The Instrument Schematic view shows the current status of the internal components of the instrument. The Results view displays the results, periodically updating the view as data are collected. The Sample Log view lists the completed steps contained in the current experiment, as well as any error messages that may have occurred during the analysis. During the time your analysis is running, open each view and explore the system's features.

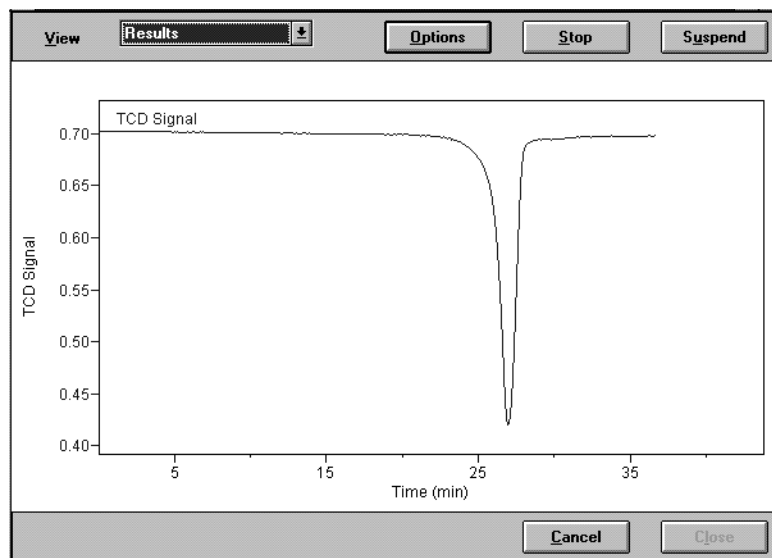
- Select the **Instrument Schematic** view and click on the sample tube illustration. The box at the lower right corner of the schematic shows updated details about the temperatures related to the sample furnace heat zone. Clicking on other parts of the schematic causes their details to be displayed in the detail box.



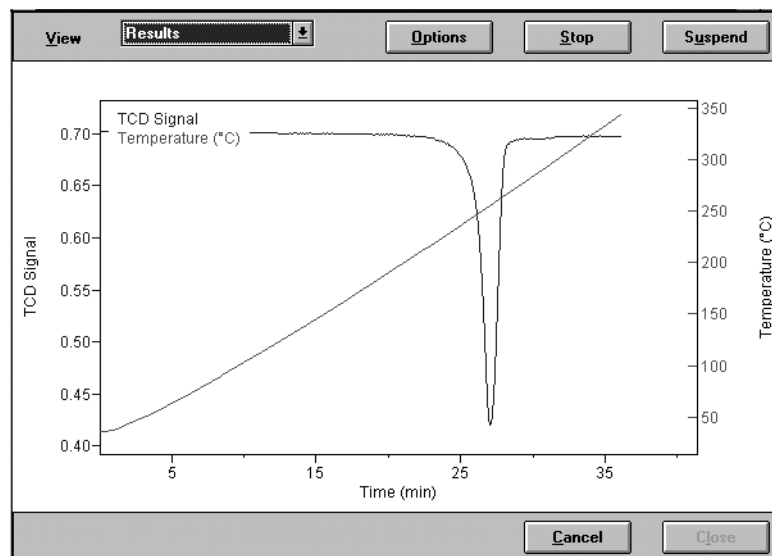
Sample tube

Detail box

- Select the **Results** view to observe the peaks **after** the baseline stabilizes and recording begins. This illustration shows the Results view of a TPR analysis on CuO in progress.



In the following illustration, Y-axis Temperature readings have been added to the display. (Instructions for making such changes to the display are given below.)



Note that the display is rescaled automatically as the experiment progresses.



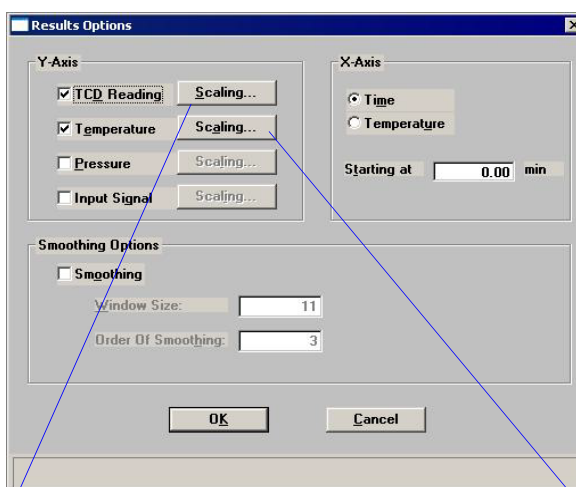
- While the **Results** view is displayed, click **Options**. A dialog opens that allows you to change the presentation of the results.

**Y-axis**

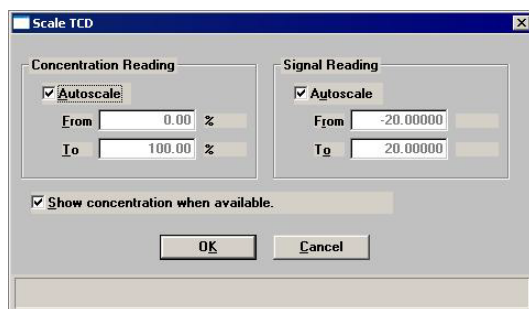
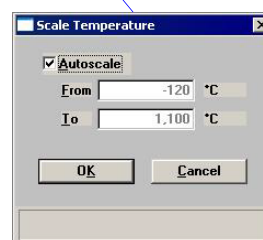
Mark the check box(es) for the results to display.

**X-axis**

Mark a radio button to choose Time or Temperature for the X-axis. You can also specify the starting value for the X-axis.



Click **Scaling** to open a dialog that enables you to control the scale of the selected axis.



Click **Scaling** for the TCD reading to specify whether or not to show the concentration (when available) and whether or not to autoscale the concentration and/or signal readings.

- Select the **Sample Log** view to review a list of each step as the analyzer begins it. The Sample Log also lists any error messages that occur during the analysis.

## Ending the Analysis

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During an analysis, the software executes each step in the analysis conditions set. The last step in every analysis is the Termination step. The Termination step lets you specify the ending state of the analyzer. The Termination step is completed when the analyzer reaches the state specified.



**Do not close the Analysis dialog until you have finished observing the data using its various views.**

After the Termination step is completed, you can continue to examine the data using the various views of the Analysis dialog, or you can close the Analysis dialog by clicking **Next**.

After you close the Analysis dialog, you can no longer use its views to assess this sample's data. However, you can view this sample's data in reports, by using the Peak Editor, and (if you use Advanced format for your sample files) by opening the sample information file to view the collected data.

## Printing Reports

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Report options are selected (and edited if required) from the sample information file. These reports can be generated automatically to a choice of destinations by selecting the report option on the third dialog of the Start Analysis series of dialogs (Chapter 6).

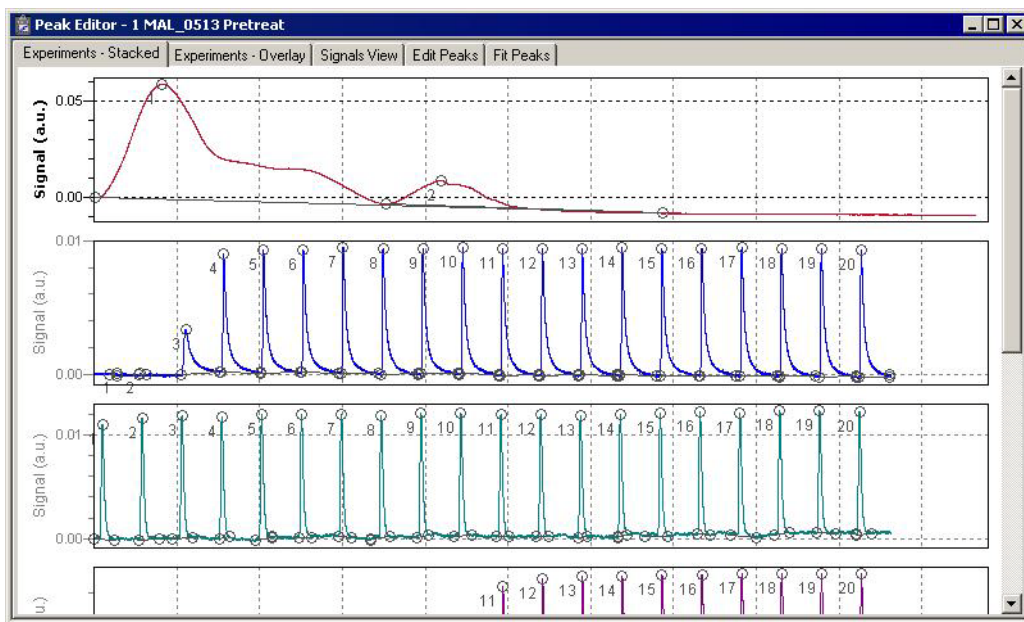
Generate partial reports at any time during the analysis or additional reports after the analysis. To do so, select Start Report from the Report menu. Select the sample file to report on, then click **OK**. In the reports list box, select the reports to print, then click **OK**.

Refer to Chapter 7 **REPORT MENU** for additional information on reports.

## Editing the Peaks

A set of peak-picking parameters is contained in the sample information file. The software automatically detects peaks that meet these criteria, and all such peaks are included in the results. In some cases, the automatically detected peaks accurately represent the results. However, it is often helpful to edit the peaks using the Peak Editor. Peak Editor can be used at any time during or after an analysis.

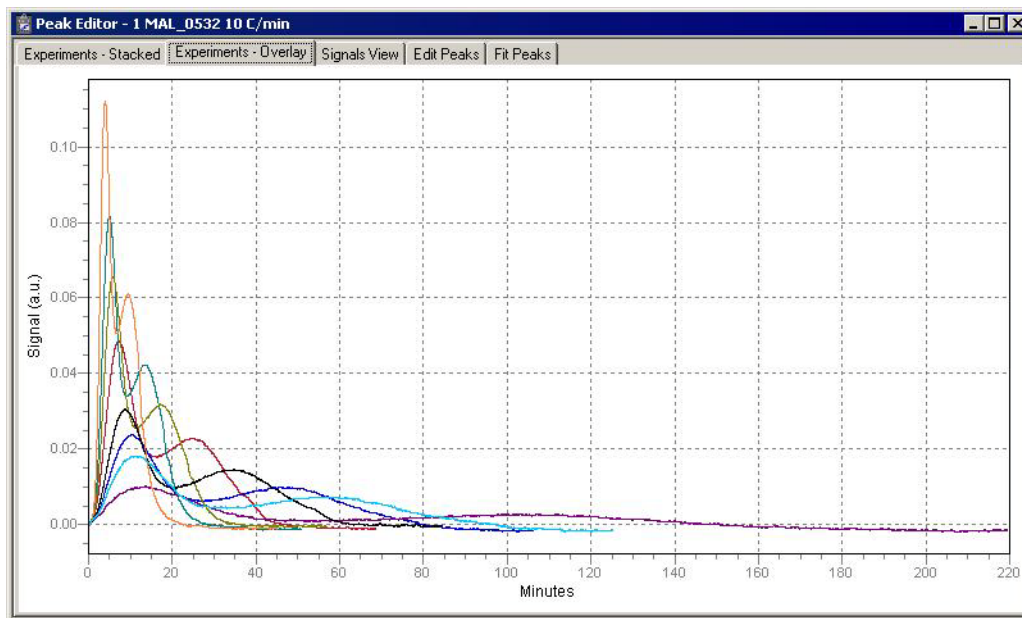
1. Select **Report > Peak Editor**; a files list box is displayed. Select the sample file, then click **OK**.



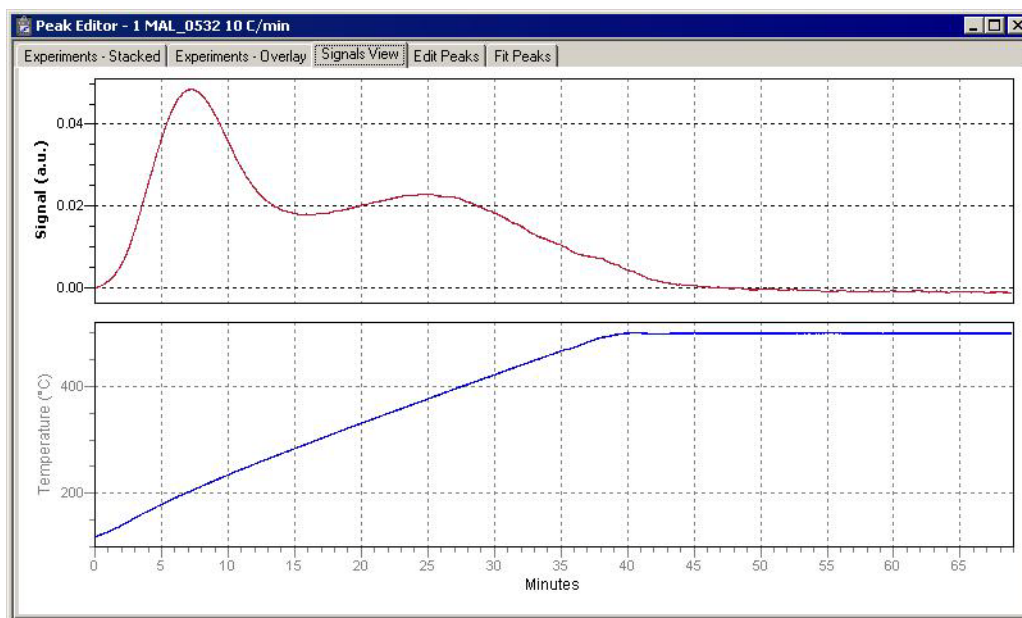
The Peak Editor defaults to the Experiments - Stacked view. This view shows the signals for all experiments contained in the file in a stacked fashion, one after the other. This example shows two experiments.

2. Right-click in the graph area to display a menu of the functions available while the Peak Editor is in this view. Refer to **“Peak Editor” on page 7-16** for a description of these functions.

3. Click **Experiments - Overlay**; this view displays the results of all experiments (in this example, the same two shown previously) in an overlaid manner.

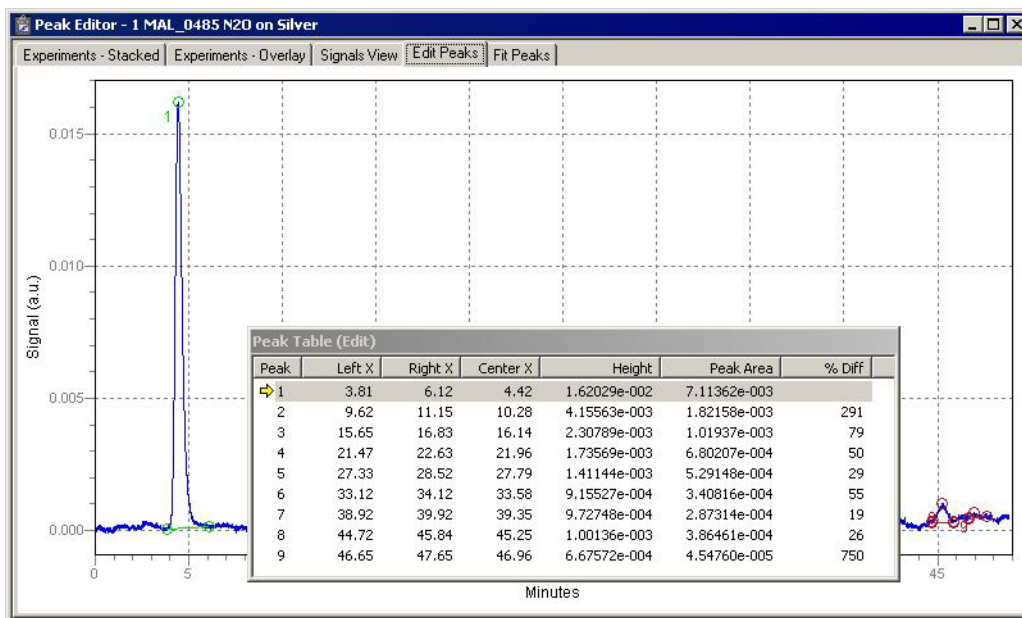


4. Right-click in the graph area to view the functions available for this view.
5. Click **View Signals**; this view shows the currently selected experiment in context with the temperature conditions that occurred during the analysis (as well as auxiliary signals, if they were recorded).



6. Right-click in the graph area to view the functions available in this view.

- Click **Edit Peaks**; the selected experiment is shown with a peak table detailing peak information positioned on top.



When multiple peaks are displayed, the peak markings for the selected peak are always green.

- Right-click in the graph area to view the functions available for editing the peaks.
- Click **Fit Peaks**; this view shows the selected experiment with a peak table positioned on top.

This view allows you to define a set of peaks; for example to create a table containing only the peaks of interest.

- Right-click in the graph area to view the functions available for creating peak sets. Experiment with some of the options; you can delete the peak sets when finished.

## Example Analyses

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This section contains a description and instructions for performing each type of analysis. The descriptions provide a very basic overview of each type of analysis. Recommendations for gases, ramp rates, etc. are generalizations which are valid for many types of samples, but not all. The examples provided are the same as the standard analysis conditions sets that are included in the software.

### TPR Analysis

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#### Description

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Temperature-Programmed Reduction (TPR) determines the number of reducible species present in the catalyst and reveals the temperature at which reduction occurs. An important aspect of TPR analyses is that the sample need not have any special characteristics other than containing reducible metals.

The TPR analysis begins by flowing analysis gas (typically hydrogen in an inert carrier gas such as nitrogen or argon) over the sample, usually starting at ambient temperature. While the gas is flowing, the temperature of the sample is increased linearly with time and the consumption of hydrogen by adsorption/reaction is monitored. Changes in the concentration of the gas mixture are determined. This information yields the hydrogen uptake volume.

#### Preparation

---

##### Pretreatment

Oxidize by flowing O<sub>2</sub> over the sample.

##### Analysis

Flow 5-10% hydrogen/argon OR 5-10% hydrogen/ nitrogen, while ramping the temperature. The analyzer records hydrogen consumption as a function of temperature. Nitrogen is sometimes used because it is more economical than argon. However, argon is recommended over nitrogen because the resultant peak(s) show no reaction between sample and gas.

##### Cold Trap

A cold trap is not required if you use argon and only traces of water are formed as a product of the reduction, and if the internal analyzer temperature zones are maintained at 100 °C or higher during the entire experiment. This method is preferred for longer experiments, because it eliminates the need to replenish the Dewar.

When you use argon, the need for a cold trap may vary. In some cases a small amount of water may be found, so the cold trap may be needed. Best baseline stability and lowest noise always result from using a cold trap.

A cold trap is needed for the entire analysis if you are using nitrogen, because water vapor and nitrogen have different thermal conductivities

## Procedure



**Before performing an analysis, make sure you have adequately prepared the sample and analyzer. Refer to “Preparing for an Analysis” on page 3-15.**

The following steps are provided as an example of how to perform a TPR analysis of copper oxide. Make the appropriate modifications to these steps for the material to be analyzed.

1. Obtain the sample mass, then install the loaded sample tube on the analyzer. If the analysis begins below ambient, either place a Dewar of coolant around the sample tube, or close the furnace around the sample tube and install the CryoCooler. If the analysis begins at ambient, close the furnace around the sample tube.
2. Install the cold trap (if you are using one), then place a Dewar filled with coolant around it. Make sure that the Dewar contains enough coolant to cover the cold trap loops.

A mixture of isopropyl alcohol (IPA) and liquid nitrogen (LN<sub>2</sub>) is the recommended coolant for this experiment. Place the isopropyl alcohol in a Dewar and slowly pour LN<sub>2</sub> into the Dewar as you stir the mixture. Continue to add and stir the mixture until it becomes a slush. The mixture must be capable of achieving a temperature of about -90 °C.



**Extreme caution should be used when mixing the IPA/LN<sub>2</sub>. If the Dewar is damaged during this process, the mixture could be expelled from the Dewar, causing injury.**

3. Create a sample information file for this analysis. (Refer to “Creating Sample Information Files” on page 3-11.)
  - a. Select **File > Open > Sample Information**. Enter a file name or accept the default; click **OK**, then **Yes** to confirm.
  - b. Complete the Sample Information dialog. Enter the correct mass and complete the optional descriptive fields (comments, operator, etc.).
  - c. Click the Analysis Conditions tab and insert the experiment steps listed below:

Step to Insert	Dialog or subdialog(s)	For this parameter:	Use this value:
Experiment	New Experiment	Experiment Description	Type an appropriate description.
		Type of analysis	Select <b>Temperature Programmed Reduction</b> from the drop-down list.
	Zones	Block Filament Cold Trap Analysis Loop	100 175 110 110 110
	Gas Flows	Prep	Helium
		Carrier/Reference	10H2-Ar OR 10H2-N2
		Loop	None
		Valve states	Trap, Prepare, Fill, Bypass
		Flow rate (all gases)	50 cm <sup>3</sup> /min
	Outputs	Use defaults	
	Peaks	Use defaults	
Wait	Wait	Select: <b>Wait for operator</b>	Type: <i>Add sample and set up cold trap.</i>
Change Gas Flows	Gas Flows	Prep	None
		Carrier	10H2-Ar
		Loop	None
		Valve states	Trap, Analyze, Fill, Prepare
Wait	Wait	Select: <b>Wait until baseline is stable.</b>	
Recording	Start Recording	One measurement every:	1.0 seconds
Temperature Ramp	Temperature Ramp	Ramp Type	Sample Ramp
		End Temperature	400
		Rate	10.0
		Hold Time	0.00
The software automatically inserts a Stop Recording step when a Start Recording step is inserted. Make sure that you insert the Temperature Ramp step within the Start/Stop Recording loop.			



Step to Insert	Dialog or subdialog(s)	For this parameter:	Use this value:
Termination	Termination (Click <b>Edit</b> , if necessary)	Return to ambient	Yes
		Detector	Off
		Gas Flows	Not applicable for low pressure.  For high pressure experiments, select the valve states used in the experiment. Otherwise, an error will occur.
		Zones	Not applicable

- d. Click on the Report Options tab and set the values to be used.
  - e. Click **Save** then **Close**.
4. Start the analysis. (Descriptions of the Unit menu options are found in Chapter 6, **UNIT MENU**.)
    - a. Select **Unit > Start Analysis**. From the Files list box, select the sample file you just created.
    - b. You can edit the sample file, if needed. For this example, no editing is required; click **Next** to continue.
    - c. The second dialog appears. From the drop-down list, select the calibrations associated with each experiment in the sample file (if any). For this example, select **None**.



**Calibration files can also be associated with a sample file after analysis through the Peak Editor using Set Calibration.**

- d. Click **Next**; the next Analysis dialog, the reports dialog, appears; no changes are needed.
- e. Read the cautionary dialog and make any necessary changes, then click **Start** to start the analysis.

As the temperature increases, the copper oxide is reduced, the water produced by the reaction is collected in the cold trap (if used), and the amount of hydrogen consumed is detected and transmitted to the software. Use the Results view to display a chromatogram of the hydrogen consumed from the detector signal as a function of the ramping temperature.

A hydrogen consumption peak which corresponds to the reduction capacity of copper oxide is displayed. The maximum peak should occur at approximately 280 °C. This temperature varies highly, depending on the CuO particle size. Larger particle size shifts

the temperature upward to 330 °C or more. Under certain combinations of sample, hydrogen concentration, and flow rate, two peaks may appear due to the transition state of  $\text{Cu}^{2+}$  to  $\text{Cu}^+$  to Cu.

5. Allow the TCD signal to return to the initial baseline after the peak has been recorded. When the analysis concludes, remove the sample tube.



**Use the *cotton* gloves provided in the accessory kit to remove the sample tube before it has cooled. You may use latex gloves to handle the sample tube when it has cooled.**

## TPD Analysis

### Description

Temperature-Programmed Desorption (TPD) analyses determine the number, type, and strength of active sites available on the surface of a catalyst from measurement of the amount of gas desorbed at various temperatures.

After the sample has been outgassed, reduced, or otherwise prepared, a steady stream of analysis gas flows over the sample and reacts with the active sites. (Alternatively, a step-like stream of gas is used when Pulse Chemisorption is used to react with active sites.) Programmed desorption begins when the temperature is ramped linearly over time while a constant stream of inert carrier gas passes over the sample.

At a certain temperature, the heat will overcome the activation energy, breaking the bond between the adsorbate and adsorbent. The adsorbed species will then desorb. If different active metals are present, they usually will desorb the reacted species at different temperatures. The desorbed molecules enter the stream of inert carrier gas and are swept to the detector, which measures the gas concentrations. The volume of desorbed species combined with the stoichiometry factor, and the temperature at which pre-adsorbed species desorb, yield the number and strength of active sites.

## Preparation

### Pretreatment

Degas by flowing inert gas (such as helium, argon, or nitrogen) over the sample while ramping the temperature.

### Analysis

Choose a gas mixture from the following table based on the results needed. Flow the gas over the sample, then ramp the temperature beginning at ambient.

Results Needed	Carrier Gas	Analysis Gas
Active Metal Surface Area H <sub>2</sub> Chemisorption	argon	hydrogen
Active Metal Surface Area CO Chemisorption	helium	carbon monoxide
Acidity	helium	ammonia (15% maximum NH <sub>3</sub> blended in helium)

### Cold Trap

Not required.



**Before performing an analysis, make sure you have adequately prepared the sample and analyzer using the instructions in Chapter 3.**

Before performing an analysis, make sure you have adequately prepared the sample and analyzer using the instructions in [“Preparing for an Analysis” on page 3-15](#).

## Procedure

Instructions given here are specific for analyzing zeolite with ammonia (use 15% maximum NH<sub>3</sub> blended in helium) at 3 to 20 °C/min. You should adjust the values given in the example to accommodate the analysis you are performing.

1. Obtain the sample mass, then install the loaded sample tube and thermocouple on the analyzer. Close the furnace around the sample tube.
2. Create a sample file for this analysis.
  - a. Select **File > Open > Sample Information**. Type a file name or accept the default; click **OK**, then **Yes** to confirm.
  - b. Complete the Sample Information dialog. Enter the correct mass and complete the optional descriptive fields (comments, operator, etc.).
  - c. Click the Analysis Conditions tab and insert the experiment steps listed below:

Step to Insert	Dialog or subdialog(s)	For this parameter:	Use this value:
Experiment	New Experiment	Experiment Description	Type an appropriate description.
		Type of analysis	Select <b>Other</b> from the drop-down list.
	Zones	Block	100
		Filament	175
		Cold trap	110
		Analysis	110
		Loop	110
	Gas Flows	Prep	Helium
		Carrier/Reference	Helium
		Loop	None
		Flow rate	50 cm <sup>3</sup> /min
		Valve states	Bypass, Prepare, Fill, Prepare
	Outputs	Use defaults	
	Peaks	Use defaults	
Temperature Ramp	Temperature Ramp	Type	Sample Ramp
		End Temperature	350
		Ramp Rate	50.0
		Hold Time	10.00
Experiment	New Experiment	Experiment description	Type an appropriate description.
		Type of analysis	Select <b>Temperature Programmed Desorption</b> from the drop-down list.
	Zones	Block	100
		Filament	175
		Cold trap	110
		Analysis	110
		Loop	110
	Gas Flows	Prep	Helium
		Carrier	Helium
		Loop	None
		Flow rate	50 cm <sup>3</sup> /min
		Valve states	Bypass, Prepare, Fill, Prepare

Step to Insert	Dialog or subdialog(s)	For this parameter:	Use this value:
Experiment <i>(continued)</i>	Outputs	Use defaults	
	Peaks	Use defaults	
Temperature Ramp	Temperature Ramp	Type	Return to Ambient
Wait	Wait	Select: <b>Wait for operator</b>	Type: <b>Add sample and set up cold trap.</b>
Change Gas Flows	Gas Flows	Prep	Ammonia (15% maximum NH <sub>3</sub> blended in helium)
		Carrier	Helium
		Loop	Ammonia (15% maximum NH <sub>3</sub> blended in helium)
		Flow rate	50 cm <sup>3</sup> /min
		Valve states	Bypass, Prepare, Fill, Prepare
Wait	Wait	Select: <b>Wait</b>	Type: <b>60.0 minutes</b>
Change Gas Flows	Gas Flows	Prep	None
		Carrier	Helium
		Loop	Ammonia (15% maximum NH <sub>3</sub> blended in helium)
		Valve states	Bypass, Analyze, Fill, Bypass
Wait	Wait	Select: <b>Wait</b>	Type: <b>30.00 minutes</b>
Wait	Wait	Select: <b>Wait until baseline is stable.</b>	
Recording	Start Recording	One measurement every	1.0 seconds
Temperature Ramp	Temperature Ramp	Type	Sample Ramp
		End Temperature	500
		Rate	3.0
		Hold Time	0.0
The software automatically inserts a Stop Recording step when a Start Recording step is inserted. Make sure that you insert the Temperature Ramp step within the Start/Stop Recording loop.			
Wait	Wait	Select: <b>Wait</b>	Type: <b>30.00 minutes</b>
Temperature Ramp	Temperature Ramp	Type	Return to Ambient

Step to Insert	Dialog or subdialog(s)	For this parameter:	Use this value:
Repeatedly insert the last several steps from the step that is double-outlined above ( <i>Wait until baseline is stable</i> step). Use a different ramp rate for each repetition; after ramping at 3°/min. and 5°/min., use 10, 15, and 20°/min. (This shifts the peak as a function of the Heat of Desorption.)			
Termination	Termination (Click <b>Edit</b> , if necessary)	Return to ambient	Yes
		Detector	Off
		Zones	Not applicable
		Gas Flows	Not applicable for low pressure.  For high pressure experiments, select the valve states used in the experiment. Otherwise, an error will occur.

- d. Click the Report Options tab and set the values. Click **Save**, then **Close**.
3. Start the analysis. (Descriptions of the Unit menu options are found in Chapter 6, **UNIT MENU**.)
    - a. Select **Unit > Start Analysis**. From the Files list box, select the sample file you just created.
    - b. You can edit the sample file, if needed. For our example, no editing is required; click **Next** to continue.
    - c. The second dialog appears. From the drop-down list, select the calibrations associated with each experiment in the sample file (if any). For this example, select **None**.
    - d. Click **Next**; the next Analysis dialog, the reports dialog, appears. For this example, no changes are needed.
    - e. Read the cautionary dialog and make any necessary changes, then click **Start** to start the analysis.

When the analysis ends, the furnace begins to lower the sample to room temperature. To speed cooling of the sample, use the optional CryoCooler or KwikCool. Remove the sample tube when the analysis is complete.



**Use the cotton gloves provided in the accessory kit to remove the sample tube before it has cooled. You may use latex gloves to handle the sample tube when it has cooled.**

## TPO Analysis

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### Description

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Temperature-Programmed Oxidation (TPO) examines the extent to which a catalyst can be re-oxidized. Generally, TPO analyses are used to measure the degree of reduction of certain oxides.

Usually the sample is pretreated and the metal oxides are reduced to the base metal. Then the reactant gas is applied to the sample in pulses or (alternatively) as a steady stream. The AutoChem 2950 measures the uptake of the reactant gas.

TPO may be performed after TPR is performed. When the TPR experiment concludes, the sample is returned to room temperature. (This process can be accelerated using the optional CryoCooler or KwikCool.) Then, the analysis gas is changed to 2-5% O<sub>2</sub> + He. This gas mixture is flowed through the sample at ambient temperature, then the temperature is ramped up to the same maximum temperature used for the preceding TPR analysis. The portion of the sample that had been reduced is re-oxidized, and the degree of reduction can be calculated.

### Preparation

---

#### Pretreatment

TPR

#### Analysis

2 to 5% oxygen/helium is flowed through the sample while temperature is ramped, beginning at ambient temperature.

#### Cold Trap

None needed



**Before performing an analysis, make sure you have adequately prepared the sample and analyzer by following the steps described earlier in this chapter.**



**For the following example, a TPO is performed on WO<sub>3</sub>. Because TPR is used as the preparation for TPO, the TPR process was included in this example.**

## Procedure

1. Obtain the sample mass, then install the loaded sample tube and thermocouple on the analyzer. Close the furnace around the sample tube.
2. Create a sample file for this analysis.
  - a. Select **File > Open, Sample Information**. Type a file name or accept the default; click **OK**, then **Yes** to confirm.
  - b. Complete the Sample Information dialog. Enter the correct mass and complete the optional descriptive fields (comments, operator, etc.)
  - c. Click on the Analysis Conditions tab and insert the experiment steps in the following table:

Step to Insert	Dialog or subdialog(s)	For this parameter:	Use this value:
Experiment	New Experiment	Experiment Description	Type an appropriate description.
		Type of analysis	Select <b>Temperature Programmed Reduction</b> from the drop-down list.
	Zones	Block	100
		Filament	175
		Cold trap	110
		Analysis	110
		Loop	110
	Gas Flows	Prep	Helium
		Carrier	10 H <sub>2</sub> -Ar
		Loop	None
		Flow rate	50 cm <sup>3</sup> /min
		Valve states	Trap, Prepare, Fill, Analyze
	Outputs	Use defaults	
	Peaks	Use defaults	
Temperature Ramp	Temperature Ramp	Type	Return to Ambient
Change Gas Flows	Gas Flows	Prep	None
		Carrier/Reference	10 H <sub>2</sub> -Ar
		Loop	None
		Valve states	Bypass, Analyze, Fill, Prepare



Step to Insert	Dialog or subdialog(s)	For this parameter:	Use this value:
Wait	Wait	Select: <b>Wait for operator</b>	Type: <b>Position cold trap Dewar and close furnace.</b>
Wait	Wait	Select: <b>Wait until baseline is stable.</b>	
Recording	Start Recording	One measurement every	1.0 seconds
Temperature Ramp	Temperature Ramp	Type End temperature Rate Hold time	Sample Ramp 980 10.0 0.00
The software automatically inserts a Stop Recording step when a Start Recording step is inserted. Make sure that you insert the Temperature Ramp step within the Start/Stop Recording loop.			
Change Gas Flows	Gas Flows	Prep	Helium
		Carrier/Reference	Helium
		Loop	None
		Valve states	Bypass, Prepare, Fill, Analyze
Temperature Ramp	Temperature Ramp	Type	Return to Ambient
Experiment	New Experiment	Experiment Description	Type an appropriate description.
		Type of analysis	Select <b>Temperature Programmed Oxidation</b> from the drop-down list.
	Zones	Block	100
		Filament	175
		Cold trap	110
		Analysis	110
		Loop	110
	Gas Flows	Prep	Helium
		Carrier/Reference	2 02-He
		Loop	None
		Flow rate	50 cm <sup>3</sup> /min
		Valve states	Bypass, Prepare, Fill, Analyze
	Outputs	Use defaults	
	Peaks	Use defaults	
Wait	Wait	Select: <b>Wait until baseline is stable.</b>	

Step to Insert	Dialog or subdialog(s)	For this parameter:	Use this value:
Recording	Start Recording	One measurement every:	1.0 seconds
Temperature Ramp	Temperature Ramp	Type	Sample Ramp
		End Temperature	980
		Rate	20.0
		Hold Time	0.00
The software automatically inserts a Stop Recording step when a Start Recording step is inserted. Make sure that you insert the Temperature Ramp step within the Start/Stop Recording loop			
Change Gas Flows	Change Gas Flows	Prep	Helium
		Carrier	Helium
		Loop	None
		Valve states	Bypass, Prepare, Fill, Analyze
Temperature Ramp	Temperature Ramp	Type	Return to Ambient
Termination	Termination (Click <b>Edit</b> , if necessary)	Return to ambient	Yes
		Detector	Off
		Zones	Not applicable
		Gas Flows	Not applicable for low pressure.  For high pressure experiments, select the valve states used in the experiment. Otherwise, an error will occur.

- d. Click the Report Options tab and set the values to use.
  - e. Click **Save**, then **Close**.
3. Start the analysis. (Descriptions of the Unit menu options are found in Chapter 6.)
    - a. Select **Unit > Start Analysis**. From the Files list box, select the sample file you just created.
    - b. You can edit the sample file if needed. For this example, no editing is needed; click **Next** to continue.
    - c. The second dialog appears. From the drop-down list, select the calibrations associated with each experiment in the sample file (if any). For this example, select **None**.
    - d. Click **Next**; the next Analysis dialog, the reports dialog, appears. For our example, no changes are needed.

- e. Read the cautionary dialog and make any necessary changes, then click **Start** to start the analysis.



**When the analysis ends, the furnace begins to lower the sample to room temperature. To speed cooling of the sample, use the KwikCool or the optional CryoCooler.**

4. As the temperature increases, the sample is oxidized, and the software calculates the volume of oxygen taken up.
5. Allow the TCD signal to return to the initial baseline after the peak has been displayed.



**Use the *cotton* gloves provided in the accessory kit to remove the sample tube before it has cooled. You may use latex gloves to handle the sample tube when it has cooled.**

6. When the displayed sample temperature reaches the ambient temperature, open the furnace. Using gloves, remove the sample tube.

## BET Surface Area Analysis

### Description

The BET Surface Area analysis lets you evaluate total surface area of the catalyst before and after chemisorption. Loss of activity, which may occur due to the blocking of pores during the chemisorption reaction, as well as the occurrence of sintering of the support, can be studied.

After outgassing the sample, a mixture of 30% nitrogen and 70% helium is applied to the sample which is immersed in a liquid nitrogen (LN<sub>2</sub>) bath. The amount of nitrogen adsorbed (usually measured on desorption) at LN<sub>2</sub> temperatures is used to calculate total surface area.

Because nitrogen uptake is a function of pore size, sample sintering can cause results to show reduced surface area. Therefore, it may be helpful to perform BET as the first and last experiments in your analysis, in order to check for sintering of the support.

---

**Preparation**

---

<b>Pretreatment</b>	Flow helium over the sample to remove impurities, usually at the maximum temperature tolerated by the sample (for example, the temperature at which the sample was calcined).
<b>Analysis</b>	Flow 30% N <sub>2</sub> /He, using a Dewar of liquid nitrogen, to measure the uptake of nitrogen. Then, remove the LN <sub>2</sub> Dewar and replace it immediately with a Dewar of water at ambient temperature. The amount of N <sub>2</sub> desorbed is measured and the BET equation is used to calculate the active surface area.
<b>Cold Trap</b>	Install the external long delay path. Do not use a cold trap Dewar.
<b>Pressure regulator</b>	For BET Analysis, gas cylinders should be set to a level above 40 psig (276 kPag).
<b>Furnace temperature</b>	During pretreatment, select a temperature high enough to remove any contaminants or moisture, but not so high as to cause sintering or fusing of the sample. Hold the temperature long enough to remove any contaminants or moisture. Maintain the target temperature for at least 30 minutes to ensure adequate degassing. Make sure the Termination step is set to return the sample temperature to 0 °C.

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**Calibration**

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BET experiments require a separate calibration consisting of three manual injections of nitrogen through the septum. You can perform this calibration either before or after the BET analysis is performed. As with all TCD calibrations, you will use the Peak Editor to associate the calibration file with the analysis file. For this reason, it is necessary that the data on the manual injections are collected in a separate experiment from the BET experiment. The injections may be included in the same analysis as the BET experiment, OR they may be performed as a completely separate analysis. You should use the same recording options (zero, invert) for the Calibration experiment and the BET experiment.

The method below includes the calibration injections as a separate experiment which follows the BET experiment in the analysis.

## Injection Size

You should inject three volumes of gas with the goal of approximating the volume of gas taken up by the sample. Use one injection slightly larger than the volume you expect, one injection slightly smaller than the volume you expect, and one injection between these two volumes. The largest injection should be two to three times greater than the smallest injection. Because you must use the same syringe for all three injections, be sure to select a syringe that can accommodate all three injection volumes. You can estimate the volume of gas uptake if you know the approximate surface area (SA) of your sample. Use the following formula to determine the approximate volume ( $V_m$ ) to inject.

$$V_m(\text{cm}^3 \text{ STP}) = \frac{SA(\text{m}^2)}{(6.023 \times 10^{23} \text{ molecule/mole})(16.21 \times 10^{-20} \text{ m}^3/\text{molecule})} \times 22414 \text{ cm}^3/\text{mole}$$

So:

$$V_m(\text{cm}^3 \text{ STP}) = SA(\text{m}^2) \times 0.229$$

For example, a sample of 0.1 g of 50 m<sup>2</sup>/g material has a surface area of 5 m<sup>2</sup>. Therefore, the volume of the gas which will be adsorbed is approximately 1.145 cm<sup>3</sup> STP.

## Procedure



**Before performing an analysis, make sure you have adequately prepared the sample and analyzer using the instructions in Chapter 3.**

1. Obtain the sample mass, then install the loaded sample tube and thermocouple on the analyzer.
2. Make sure the Delay Path is installed in the Cold Trap port.
3. Create a sample file containing the appropriate analysis conditions and report options. (Descriptions of the options on each dialog are found in Chapter 5.)
  - a. Select **File > Open > Sample Information**. Type a file name or accept the default; click **OK**, then **Yes** to confirm.
  - b. Complete the Sample Information dialog using appropriate values.
  - c. Click on the Analysis Conditions tab and insert the experiment steps listed on the following page.

Step to Insert	Dialog or subdialog(s)	For this parameter:	Use this value:
Experiment	New Experiment	Experiment Description	Type an appropriate description.
		Type of analysis	Select <b>BET Surface Area</b> from the drop-down list.
	Zones	Block	100
		Filament	175
		Cold trap	110
		Analysis	110
		Loop	110
	Gas Flows	Prep	Helium
		Carrier	Helium
		Loop	None
		Flow rate	50 cm <sup>3</sup> /min
		Valve states	Trap, Prepare, Fill, Analyze
	Outputs	Use defaults	
	Peaks	Use defaults	
	Recording options	Select: <b>Invert the TCD Signal</b>	
Temperature Ramp	Temperature Ramp	Type	Sample Ramp
		End Temperature	350
		Rate	25.0
		Hold Time	Set to amount of time it takes to prepare sample.
		Enable KwikCool	No
Temperature Ramp	Temperature Ramp	Type	Return to Ambient
		Enable KwikCool	Yes
Change Gas Flows	Gas Flows	Prep	None
		Carrier	30 N <sub>2</sub> -He
		Loop	None
		Valve states	Trap, Analyze, Fill, Prepare
Wait	Wait	Select: <b>Wait until baseline is stable.</b>	
Wait	Wait	Select: <b>Wait for operator</b>	Type: <b>Click OK to proceed with BET measurement.</b>
Recording	Start Recording	One measurement every:	0.1 second
Dose	Dose	Select: <b>Get BET Point</b>	

Step to Insert	Dialog or subdialog(s)	For this parameter:	Use this value:
The software automatically inserts a Stop Recording step when a Start Recording step is inserted. Make sure that you insert the Dose step and the Temperature Ramp step within the Start/Stop Record loop.			
Termination	Termination (Click <b>Edit</b> , if necessary)	Return to ambient	Yes
		Detector	Off
		Zones	Not applicable
		Gas Flows	Not applicable

To include the calibration data collection (three manual injections through the septum) within this analysis but as a separate Experiment, also insert the following steps (**before the Termination step**):

Step to Insert	Dialog or subdialog(s)	For this parameter:	Use this value:
Experiment	New Experiment	Experiment Description	Type an appropriate description.
		Type of analysis	Select <b>Other</b> from the drop-down list.
	Zones	Block	100
		Filament	175
		Cold trap	110
		Analysis	110
		Loop	110
	Gas Flows	Prep	None
		Carrier	30 N2-He
		Loop	None
		Flow rate	25 cm <sup>3</sup> /min
		Valve states	Trap, Analyze, Inject, Prepare
	Outputs	Use defaults	
	Peaks	Use defaults	
Wait	Wait	Select: <b>Wait until baseline is stable.</b>	
Recording	Start Recording	One measurement every:	0.1 second
Repeat	Start Repeat Sequence	Select: <b>Repeat</b>	Type: <b>3 times</b>
Wait	Wait	Select: <b>Wait for operator.</b>	<b>Type: Prepare manual N2 injection. Click OK.</b>

Step to Insert	Dialog or subdialog(s)	For this parameter:	Use this value:
Dose	Dose	Select: <b>Manual Injection</b>	
The software automatically inserts a Stop Repeat step when a Start Repeat step is inserted. Make sure that you insert the Wait step within the Start/Stop Repeat loop.			
The software automatically inserts a Stop Recording step when a Start Recording step is inserted. Make sure that you insert the Dose step and the Temperature Ramp step within the Start/Stop Record loop.			

- d. Click the Report Options tab and set the values to use.
  - e. Click **Save**, then **Close**.
4. Start the analysis. (Descriptions of the Unit menu options are found in Chapter 6.)
    - a. Select **Unit > Start analysis**. From the Files list box, select the sample file you just created.
    - b. You can edit the sample file if needed, but for our example, editing is not required; click **Next** to continue.
    - c. The second dialog box appears. From the drop-down list, select the calibrations associated with each experiment in the sample file (if any). For this example, select **None**.
    - d. Click **Next**; the next Analysis dialog appears: the reports dialog. For our example, no changes are needed.
    - e. Read the cautionary dialog and make any necessary changes, then click **Start** to start the analysis. During the analysis, you are prompted to change the Dewar.



**Change the Dewar rapidly. It is recommended that you hold the water Dewar in one hand and remove the LN<sub>2</sub> Dewar with the other to minimize the time required.**

5. When the analysis ends, remove the sample tube from the analyzer, place the cap on the tube, and dry off the outside of the tube with a paper towel.



**Use the *cotton* gloves provided in the accessory kit to remove the sample tube before it has cooled. You may use latex gloves to handle the sample tube when it has cooled.**

6. Weigh the sample tube, sample, and caps. Subtract this mass from the mass you obtained for the sample tube and caps. The resulting mass is the dry sample mass (the “after analysis mass”). This mass will be used when you calculate the BET surface area, as described in the following sections.



## Generating the BET Report

---

The following tasks must be performed before you can generate the BET report:

- Edit the sample file and the calibration file to ensure that peaks are properly marked
- Create a TCD calibration file
- Associate the TCD calibration file with the sample file

After completion of these tasks, select **Start Report** from the Reports menu to generate the report. Be sure that **BET Surface Area** is selected (preceded with a check mark).

### Edit the peaks in the sample file

1. Select **Reports > Peak Editor**; the Edit Sample Peaks dialog is displayed.
2. Select the BET sample file you used with the analysis, then click **OK**.
3. Click the experiment, then click **Edit Peaks**.
4. Right-click in the graph area and choose **Mark All Peaks**; all peaks that meet the current peak-picking parameters are marked.
5. If required, edit the edges of the desorption peak as follows:



**The desorption peak is used to determine the surface area because the desorption process starts with the adsorbate equilibrated on the surface. Also, it is simple and convenient to develop a calibration for desorption.**

- a. Drag the cursor over the entire baseline of the peak to enlarge the editing area.
  - b. Click on the baseline of the left side of the peak, right-click and choose **Mark left edge**.
  - c. Repeat step b for the right side of the peak.
6. As you adjust the peak edges, the values associated with the peak (listed in the peak table) are also adjusted. When you are satisfied with the appearance of the peak, right-click and choose **Save**.

### Edit the peaks in the calibration file

1. Select Peak Editor from the Reports menu; the Edit Sample Peak dialog is displayed.
2. Select the file containing the calibration data you used with the analysis, then click **OK**.
3. Edit the peaks in the same manner as described for the sample file.

## Create a TCD Calibration file

1. Select **Unit > TCD Calibration > New**; the TCD Calibration dialog box is displayed.

2. Click **Load Calibration Data**; the Load Calibration Sample File dialog is displayed.
3. Select the sample file containing the calibration data, then click **OK**. The data in the file are inserted into the appropriate fields of the TCD Calibration window.
4. The values in the Peak Volume column are defaults. The correct values for the volume of each injection at STP must be determined using the following formula:

$$V_{STP} = V(\text{injected}) \left( \frac{273.15}{273.15 + \text{ambient temp}} \right) \times \left( \frac{\text{ambient pressure}}{760 \text{ mmHg}} \right)$$

Determine the volumes for each calibration peak listed in the table.

5. Enter the converted values (cm<sup>3</sup> STP) into the table.

Assess the Peak Area plot, Goodness of Fit, and Coefficients to decide if the calibration file is acceptable. Use your laboratory's standards to determine what level of linearity is acceptable. As a general guideline, however, you should use a calibration file with a very low value for Goodness of Fit (less than 0.1), when 1st or 2nd Degree is specified.

To delete any peaks that are outliers (highlight the peak data in the table and click **Delete Peak**). To evaluate the results when you change the degree (select a different degree from the drop-down list).

6. Enter a new description into the **Description** field.
7. When you are satisfied with all values in the file, click **Save**, then **Close**

**Associate the Calibration file with the Sample file**

1. Select **Reports > Peak Editor**; the Edit Sample Peak dialog is displayed.
2. Select the BET sample file you used with the analysis, then click **OK**.
3. Click on the experiment, then right-click in the graph area and choose **Set Calibration**; the Set TCD Calibration dialog box is displayed.
4. Click the down arrow to the right of the Calibration field and choose the Calibration file you just created; click **OK**.
5. Repeat steps 3 and 4 for each experiment.
6. Click **Save**, then **Close**.

## Pulse Chemisorption

---

### Description

---

A Pulse Chemisorption analysis determines active surface area, percent metal dispersion, and active particle size by applying measured pulses of reactant gas to the sample, depending upon what technique is used. Pulse Chemisorption is performed in the same manner as TPD, except that the sample is dosed with the analysis gas using the injection loop until each active site has reacted. After the active sites have completely reacted, the discretely injected gas volumes emerge unchanged. If TPD is performed after Pulse Chemisorption, additional information about the distribution of active sites and the strength of active sites is collected.

The amount chemisorbed is the difference between the total amount of reactant gas injected and the amount that did not react with the active sites of the sample. The size of each pulse is determined by the loop, which is located on an electrically operated valve.

### Loop Volume

---

In general, it is desirable for the sample to require at least two doses of gas, but no more than ten doses, before the reaction ends (although this may vary from lab to lab). Some factors that influence the number of doses required are sample size, the active surface area per unit of sample, and the size of the loop. If your pulse chemisorption analysis requires more doses or fewer doses than you prefer, you can change either the sample size or the loop size.

Three loops of different sizes are provided with the analyzer; each loop must be calibrated before its use. To replace or install a loop, refer to **Changing the Injection Loop** on page 9-44. As an alternative to physically replacing the loop, adjust the loop volume by adjusting its temperature. If you change the loop volume by changing its temperature, results will be more accurate if you recalibrate the loop volume at the new temperature.

### Loop Calibration

---

Before data from an analysis that uses an injection loop can be reduced, a loop calibration experiment must be performed. You can perform a loop calibration before or after the analysis.

The following example assumes that the loop calibration will be performed after the analysis. See the instructions later in this appendix for performing a loop calibration. You can also include a Loop Calibration step in your pulse chemisorption analysis.

## Preparation

---

<b>Pretreatment</b>	Degas by flowing inert gas (such as helium, argon, or nitrogen) over the sample while ramping the temperature.
<b>Analysis</b>	Pulse the loop (analysis) gas over the sample until the peak area remains constant.
<b>Cold Trap</b>	<p>No cold trap is needed during pretreatment</p> <p>During the analysis, use the cold trap fixture with a Dewar of water in place as a short delay path. This delay compensates for the flow disturbance caused by each loop injection.</p>
<b>Pressure regulator</b>	For Pulse Chemisorption, gas cylinders should be set to a level of at least 40 psig (276 kPag).
<b>Furnace temperature</b>	Select a temperature high enough to remove any contaminants or moisture, but not so high as to cause sintering or fusing of the sample. Make sure the Termination step is set to return the sample temperature to ambient.



**In the following example, Pulse Chemisorption is performed on a sample of Pt/Al<sub>2</sub>O<sub>3</sub> with CO. You should alter the analysis conditions to accommodate the analysis you are conducting.**

## Procedure



**Before performing an analysis, make sure you have adequately prepared the analyzer using the instructions in Chapter 3.**

1. Obtain the sample mass, then install the loaded sample tube and thermocouple on the analyzer. Close the furnace around the sample tube.
2. Create a sample file for this analysis.
  - a. Select **File > Open > Sample Information**. Enter a file name or accept the default; click **OK**, then **Yes** to confirm.
  - b. Complete the Sample Information dialog using appropriate values.
  - c. Click the Analysis Conditions tab and insert the experiment steps listed below:

Step to Insert	Dialog or subdialog(s)	For this parameter:	Use this value:
Experiment	New Experiment	Experiment Description	Type an appropriate description.
		Type of analysis	Select <b>Pulse Chemisorption</b> from the drop-down list.
	Zones	Block	100
		Filament	175
		Cold trap	110
		Analysis	110
		Loop	110
	Gas Flows	Prep	Helium
		Carrier/Reference	Helium
		Loop	None
		Flow rate	50 cm <sup>3</sup> /min
		Valve states	Bypass, Prepare, Fill, Analyze
	Outputs	Use defaults	
	Peaks	Use defaults	
Wait	Wait	Select: <b>Wait</b>	Type: 5 minutes

Step to Insert	Dialog or subdialog(s)	For this parameter:	Use this value:
Change Gas Flows	Gas Flows	Prep	Hydrogen
		Carrier	Helium
		Loop	None
		Valve states	Bypass, Prepare, Fill, Analyze
Temperature Ramp	Temperature Ramp	Type	Sample Ramp
		End Temperature	120
		Rate	10.0
		Hold Time	30
Temperature Ramp	Temperature Ramp	Type	Sample Ramp
		End Temperature	220
		Rate	10.0
		Hold Time	30
Temperature Ramp	Temperature Ramp	Type	Sample Ramp
		End Temperature	350
		Rate	10.0
		Hold Time	120.0
Change Gas Flows	Gas Flows	Prep	Helium
		Carrier	Helium
		Loop	None
		Valve states	Bypass, Prepare, Fill, Analyze
Wait	Wait	Select: <b>Wait</b>	Type: <b>90 minutes</b>
Temperature Ramp	Temperature Ramp	Type:	Return to Ambient
Wait	Wait	Select: <b>Wait</b>	Type: <b>30 minutes</b>
Temperature Ramp	Temperature Ramp	Type	Sample Ramp
		End Temperature	35
		Rate	5.0
		Hold Time	10.0
Change Gas Flows	Change Gas Flows	Prep	None
		Carrier	Helium
		Loop	Carbon Monoxide
		Valve states	Trap, Analyze, Fill, Prepare
Wait	Wait	Select: <b>Wait until baseline is stable.</b>	

Step to Insert	Dialog or subdialog(s)	For this parameter:	Use this value:
Recording	Start Recording	One measurement every:	0.2 second
Wait	Wait	Select: <b>Wait</b>	Type: <b>3.0 minutes</b>
Repeat	Start Repeat Sequence	Select: <b>Repeat until peaks are equal or 20 times.</b>	
Dose	Dose	Select: <b>Inject Loop Gas</b>	
		Select: <b>Wait for</b>	Type: <b>1.5 minutes</b>
The software automatically inserts a Stop Repeat step when a Start Repeat step is inserted. Make sure that you insert the Dose step within the Start/Stop Repeat loop.			
The software automatically inserts a Stop Record step when a Start Record step is inserted. Make sure that you insert the Start/Stop Repeat step and the Dose step within the Start/Stop Record loop.			
Termination	Termination (Click <b>Edit</b> , if necessary)	Return to ambient	Yes
		Detector	Off
		Zones	Not applicable
		Gas Flows	Not applicable

- d. Click the Report Options tab and set the values to use.
  - e. Click **Save**, then **Close**.
3. Start the analysis. (Descriptions of the Unit menu options are found in Chapter 6 **UNIT MENU**.)
    - a. Select **Unit > Start analysis**. From the Files list box, select the sample file you just created.
    - b. You can edit the sample file if needed. For our example, no editing is needed; click **Next** to continue.
    - c. The second dialog appears. From the drop-down list, select the calibrations associated with each experiment in the sample file (if any). For this example, select None.
    - d. Click **Next**; the next Analysis dialog appears: the reports dialog. For our example, no changes are needed.
    - e. Read the cautionary dialog and make any necessary changes, then click **Start** to start the analysis.



When the analysis ends, the furnace begins to lower the sample to room temperature. To speed cooling of the sample, use the KwikCool or the optional CryoCooler.



**Use the *cotton* gloves provided in the accessory kit to remove the sample tube before it has cooled. You may use latex gloves to handle the sample tube when it has cooled.**

When the displayed sample temperature reaches the ambient temperature, open the furnace. Using gloves, remove the sample tube.

### Generate the Report

---

Before proceeding with the steps below, open the sample file using **Peak Editor** on the Reports menu to ensure that the peaks are properly marked.

There are three possibilities for each dose of gas injected during Pulse Chemisorption: all of the gas is taken up by the sample, some of the gas is taken up by the sample, or none of the gas is taken up by the sample. When Pulse Chemisorption is properly performed, there will be some injections of each type. When you view the data using the Peak Editor, however, only those injections in which some or none of the gas is taken up will appear as peaks. When all of the gas is taken up by the sample, none of it reaches the detector and, therefore, the peak area is zero. These types of peaks are detected automatically by the software and do not require marking.

After verifying that the peaks are properly marked, perform the following steps to generate the report:

1. Select **File > Open > Sample Information**.
2. Select the sample file used with this analysis and click **OK**.
3. Click the Collected Data tab to display its associated dialog box.
4. Enter the percent of active gas in the **Active concentration** field.
5. Click **Active Metals**; the Active Metals Table is displayed. Verify that the **Stoichiometry Factor** and the **% of Sample Weight** values are correct.
6. Click **OK** to close the Active Metals dialog box.
7. Verify that the values for the physical injection volume, ambient temperature, and ambient pressure are accurate.
8. Select **Reports > Start Report** to generate the report.

## Loop Calibration

---

You must calibrate each injection loop before its first use to determine its precise volume under local conditions. Calibration consists of:

- Determining the average area of a series of peaks generated by injections of a known volume of gas through the analyzer septum using a syringe.
- Determining the average area of a series of peaks generated by injections of the same gas using the analyzer's internal loop.
- Calculating the volume of the loop by comparing the average peak area generated by the loop injections with that generated by the syringe injections.

## Update Ambient Pressure and Temperature

---

Ambient pressure and temperature at the time of the analysis affect the results of a loop calibration. In calculations, the analyzer uses the ambient pressure and temperature recorded in the Environmental Defaults dialog box (accessed from the Options menu). Check and update environmental defaults before beginning a loop calibration. If necessary, also update the ambient temperature and pressure on the Collected Data dialog of the sample information file after the analysis. (The sample information file must be opened in Advanced format in order to view or edit collected data.)

## Create a Sample File

---

First, you must create a sample file:

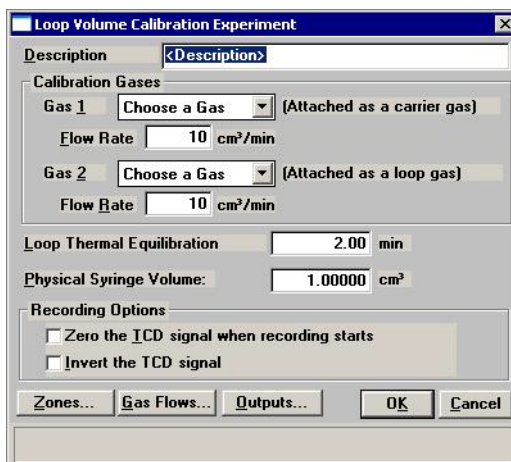
1. Select **File > Open > Sample information**; the Sample Information File selection dialog appears.
2. Enter the name to use for this calibration in the **File name** field. Click **OK**, then **Yes** to confirm.



**If the file is in Basic format, click Advanced to switch to the Advanced format.**

3. Complete the fields of the Sample Information dialog as needed.
4. Click the **Analysis Conditions** tab. Click **Insert**, then select **Loop Calibration** from the list of available steps.

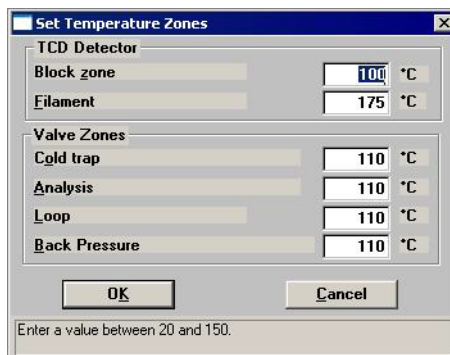
5. Complete the Loop Volume Calibration Experiment Step dialog using Helium for Gas 1 and Argon for Gas 2.



6. Click **Zones** to set the temperature for the analyzer's heated zones. Complete the fields of the Set Temperature Zones dialog box as shown below, using 175 °C for the filament and 100 °C for the block zone. Use the defaults for all other zones. Click **OK** to close the dialog.



**You must create a Loop Calibration for each loop you use, using the temperatures used in the analyses that will be associated with the calibration file.**



7. Click **Save**, then **Close**.

## Perform the Analysis

1. Make sure the injection loop to calibrate is installed on the analyzer. Refer to **“Changing the Injection Loop” on page 9-44** for the steps to install or replace an injection loop.
2. Select **Unit > Start Analysis**; select the sample file you just created.
3. Click **Next** three times to accept the values contained in this sample information file. (Although you can edit these values, no changes are needed for this example). Read the cautionary statement, then click **Start** to start the analysis.
4. As the test progresses, you are prompted to make three manual injections.

For this example, use argon for your injections (because argon is specified as the loop gas in the sample information file). Use a volume that is close to the volume of the loop you are calibrating. For example, if you are calibrating the 1-cm<sup>3</sup> loop, use a 1-cm<sup>3</sup> syringe and inject as close to 1 cm<sup>3</sup> of gas as possible.



**Pay close attention to the instructions given in prompts that appear on the screen. Perform the prompted steps in the order given. For example, if you are prompted “Click OK, then perform manual injection,” you should perform these actions in the order given. Otherwise, data may not be recorded properly.**



**Most accurate data results from keeping injection size as consistent as possible. Injection errors may be evident in the data, and may make it necessary to repeat the experiment.**

After the third manual injection, the analyzer automatically makes three injections using the loop.

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