MICRO-CIRRUS 3 INTERFACE

micromeritics®

USER GUIDE

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(Rev A)

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ABOUT THIS MANUAL

All references to FR-series Flow Reactors in this document encompass the FR-100 and FR-200 unless otherwise noted.

Parts and accessories are located on the Micromeritics web page.

The following symbols or icons indicate safety precautions and/or supplemental information and may appear in this manual:

NOTE — Notes contain important information applicable to the topic.

CAUTION — Cautions contain information to help prevent actions that may damage the analyzer or components.

WARNING — Warnings contain information to help prevent actions that may cause personal injury.

GENERAL SAFETY

Do not modify this instrument without the authorization of Micromeritics Service Personnel.

Any piece of laboratory equipment can become dangerous to personnel when improperly operated or poorly maintained. All employees operating and maintaining Micromeritics instruments should be familiar with its operation and should be thoroughly trained and instructed on safety.

- Read the operator manual for any special operational instructions for the instrument.
- Know how the instrument functions and understand the operating processes.

- Wear the appropriate personal protective equipment when operating this instrument — such as eye protection, lab coat, protective gloves, etc.
- When lifting or relocating the instrument, use proper lifting and transporting devices for heavy instruments. Ensure that sufficient personnel are available to assist in moving the instrument.
- Always pay attention to the safety instructions provided on each label affixed to the instrument and do not alter or remove the labels. When inspecting the instrument, ensure that the safety labels have not become worn or damaged.
- Proper maintenance is critical to personnel safety and smooth instrument operation and performance. Instruments require regular maintenance to help promote safety, provide an optimum end test result, and to prevent costly down time. Failure to practice proper maintenance procedures can lead to unsafe conditions and shorten the life of the instrument.
- Improper handling, disposing of, or transporting potentially hazardous materials can cause serious bodily harm or damage to the instrument. Always refer to the MSDS when handling hazardous materials. Safe operation and handling of the instrument, supplies, and accessories is the responsibility of the operator.

INTENDED USE

The instrument is intended to be operated by trained personnel familiar with the proper operation of the equipment recommended by the manufacturer and as well as relevant hazards involved and prevention methods. Other than what is described in this manual, all use is seen as unintended use and can cause a safety hazard.

The instrument is intended to be used as per applicable local and national regulations.

TRAINING

It is the customer's responsibility to ensure that all personnel operating or maintaining the equipment participate in training and instruction sessions. All personnel operating, inspecting, servicing, or cleaning this instrument must be properly trained in operation and machine safety before operating this instrument.

SYMBOLS THAT MAY APPEAR ON YOUR INSTRUMENT

The following symbols or icons indicate safety precautions and/or supplemental information and may appear on your instrument:

Use extreme caution when working on the instrument where one of these symbols may be displayed. These symbols indicate the part may be hot and cause serious burns.

Use the cotton gloves provided in the accessory when handling heated surfaces. These cotton gloves are not intended to protect hands when heated surfaces are above 60 °C.

When working on the instrument where this symbol is displayed, refer to your Micromeritics' instruction manual for additional information.

When this symbol is displayed, toxic or flammable gases require proper venting of exhaust.

This symbol can also indicate the instrument uses mercury which is an extremely toxic substance. Read the Material Safety Data Sheet (MSDS) and be aware of the hazards of mercury and know what to do in the event of a spill or an exposure incident

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1 ABOUT THE SOFTWARE

The MKS Process Eye Professional software must be installed prior to using the Micromeritics-Cirrus Interface Software and the MKS Cirrus mass spectrometer. Refer to the Micro-Cirrus 3 Interface Installation Instructions for more information [*part number 004-42805-00*].

The *Micromeritics-Cirrus Interface Software* is used with select Micromeritics analyzers integrated with an MKS Cirrus 3 mass spectrometer. This software runs within the *MKS Process Eye Professional* application. The Micromeritics-Cirrus Interface Software will perform the following tasks:

- Automatically collect data with the Cirrus 3 mass spectrometer via trigger signal from the Micromeritics analyzer
- Customize *Peak Jump* recipes
- Access standard operations such as analog scan, barchart scan, peak jump scan, leak check, and data storage
- Calibrate detectors
- Scale the Micromeritics analyzer sample thermocouple signal

RECIPES

The interface software controls the analyzer via recipes. A recipe is a list of mass fragments, or mass-to-change ratios (m/z), to be analyzed, along with detector selection and resolution setting. Micromeritics has included preset parameters in the software, enabling users to create *Peak Jump* recipes quickly. Recipes can be saved for future use.

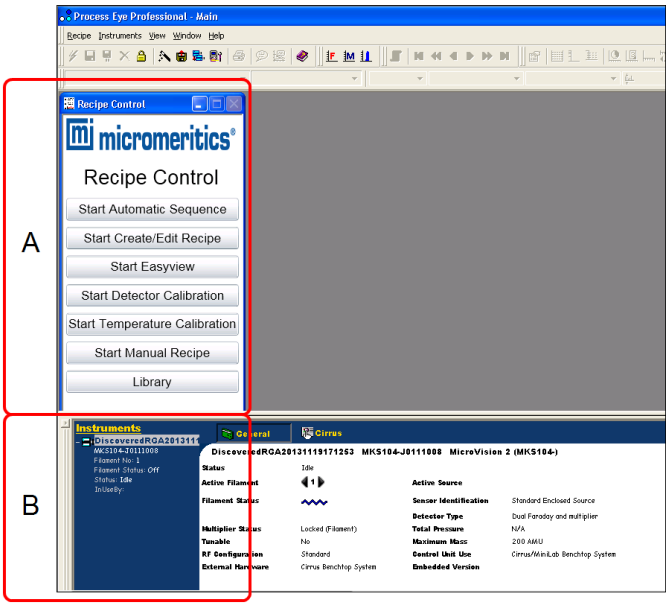
DOCUMENTATION

This user guide provides a description of the commands included in the *Micromeritics-Cirrus Interface Software* and instructions on how to use them. It does not include descriptions for all commands related to the Cirrus software. Refer to the MKS documentation supplied with the MKS Cirrus 3.

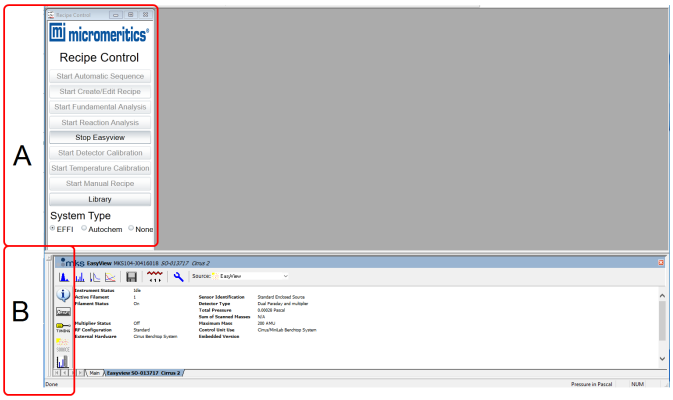
START THE INTERFACE SOFTWARE

Double click the *Process Eye Professional* desktop shortcut, or go to ***Windows > Start*** command to open the *Process Eye Professional* software to display:

- Recipe Control
- Recipe Information



A. Recipe Control window
B. Instrument dashboard



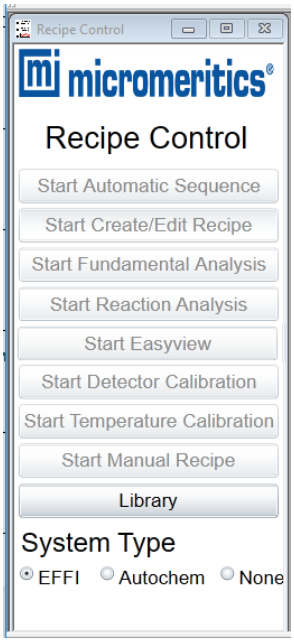
A. Recipe Control window
B. Instrument dashboard

EXIT THE INTERFACE SOFTWARE

Go to ***Recipe > Exit*** on the *Process Eye Professional* main menu.

2 RECIPE CONTROL

All functions for the *Micromeritics-Cirrus Interface Software* are accessed via the *Recipe Control* window.



Recipe Control Window Options Table

Option	Description
Start Automatic Sequence	Collect data automatically via the Micromeritics analyzer digital trigger.
Start Create/Edit Recipe	Create *Peak Jump* recipes for the *Automatic Sequence* mode.
Start Easyview	Access a number of operations, including different scan modes and diagnostics.
Start Detector Calibration	Calibrate the Faraday and SEM detectors. Refer to the Cirrus 3 Operator Manual for additional details.
Start Temperature Calibration	Calculate the scaling equation for the Micromeritics analyzer sample thermocouple signal. Refer to the Cirrus 3 Operator Manual for additional details.
Start Manual Recipe	Start and stop recipes created using a recipe wizard.
Library	View mass spectral data for a variety of compounds.
System Type	Sets the trigger behavior for the selected instrument.

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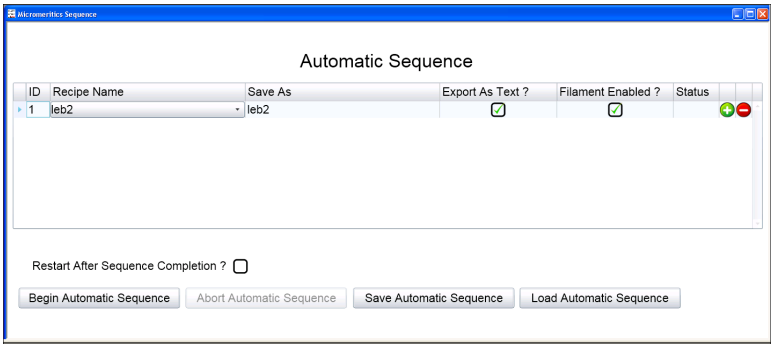
3 AUTOMATIC SEQUENCE

Click **Start Automatic Sequence** in the *Recipe Control* window to display the *Automatic Sequence*.



Use the *Automatic Sequence* window to:

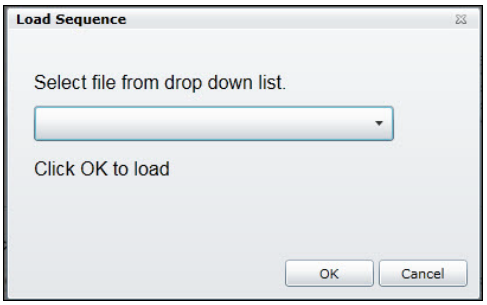
- Automatically collect data via the Micromeritics analyzer digital trigger.
- Automatically save data files, which can be imported into the Micromeritics analyzer application.
- Enable the filament for each recipe.

This window displays the current sequence configuration, which includes a list of recipes and associated run conditions.

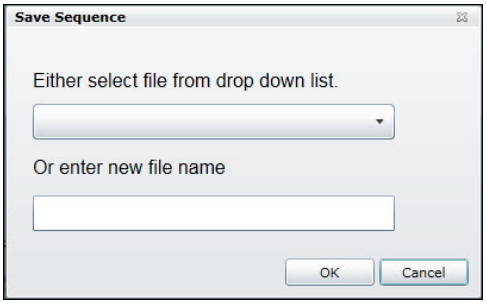
Automatic Sequence Options Table

Options	Description
(–) Sign	Click to remove recipes.
(+) Sign	Click to add recipes below the current recipe.
Abort Automatic Sequence [*button*]	Stops the sequence run. Enabled when the *Automatic Sequence* has started.
Begin Automatic Sequence [*button*]	Starts the recipe.
Export As Text [*check box*]	Select for each recipe to be exported to a text file. Text files are saved to the default *Process Eye* data directory with a .TXT file extension.
Filament Enabled [*check box*]	Select to have the filament turned on during recipe initialization. The filament must be turned on for data collection.

Automatic Sequence Options Table (continued)

Options	Description
ID	Unique number given for each recipe selected.
Load Automatic Sequence [*button*]	Enabled when a sequence is not running. Click to display the *Load Sequence* window to load another sequence. Click **OK** to save the options. 
Recipe Name [*selection*]	Select from the list of recipes.
Restart After Sequence Completion [*check box*]	Select to have the sequence restart upon completion of the last recipe in the list. Use this option when collecting data in more than one experiment.
Save As [*button*]	Unique file name for each analysis. Enter a name for each *Process Eye* data file in this column. If a name is not specified, the data file will default to the option selected in the *Recipe Name* column. A sequential 6-digit tag is appended to each file name. For example, *Air* will be saved as *Air_000001.SI-d* if no previous versions exist. The file is saved to the default *Process Eye* Data directory.

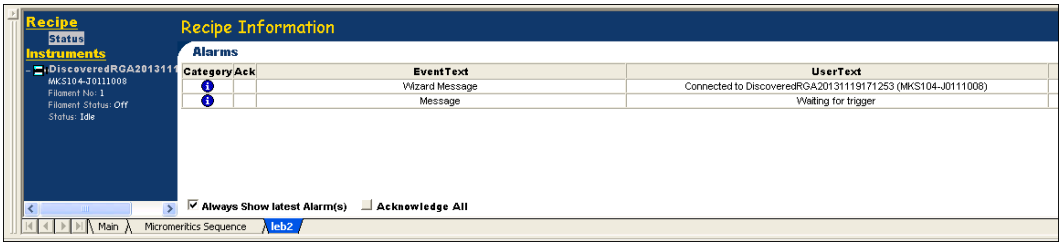
Automatic Sequence Options Table (continued)

Options	Description
Save Automatic Sequence [*button*]	Enabled when a sequence is not running. Displays the *Save Sequence* window to save the current sequence. Select a file from the drop-down list (the file will be overwritten with the current data) or enter a new file name. Click **OK** to save the options. 
Status	Displays recipe run statuses during an automatic sequence.

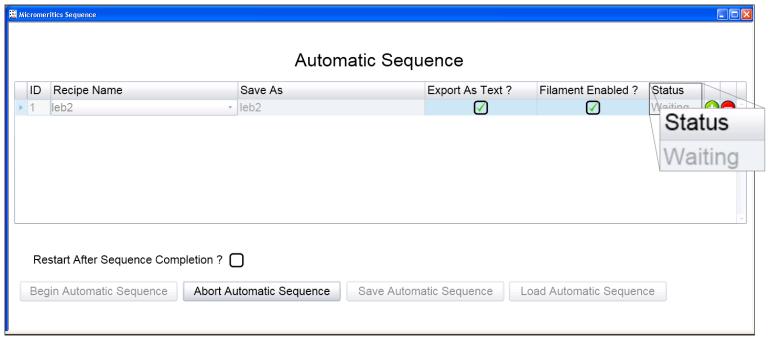
To begin an automatic sequence after recipes have been selected and configured:

1. Select *Restart After Sequence Completion* to have the sequence restart upon completion of the last recipe in the list (if applicable).
2. Click **Begin Automatic Sequence**.

If prompted to select an instrument, select one from the list, then click **Connect**.



- The first recipe is initialized and a tab with the recipe name is added to the *Recipe Information* pane.
- A status of *Waiting* displays in the *Status* column until the Micromeritics analyzer *Digital Trigger 1* is set to the high (1) state.


Micromeritics Sequence
Automatic Sequence
ID
Recipe Name
Save As
Export As Text ?
Filament Enabled ?
Status
1
leb2
leb2
Waiting
Status
Waiting
Restart After Sequence Completion ?
Begin Automatic Sequence
Abort Automatic Sequence
Save Automatic Sequence
Load Automatic Sequence

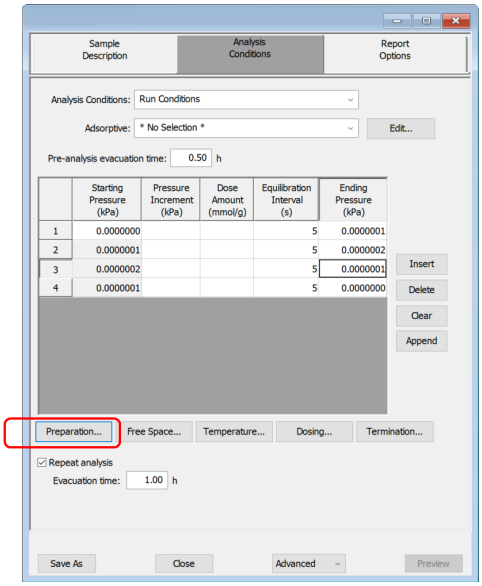
ENABLE THE TRIGGER FOR FLEX 3500 CHEMISORPTION

The trigger can be enabled using one of two methods:

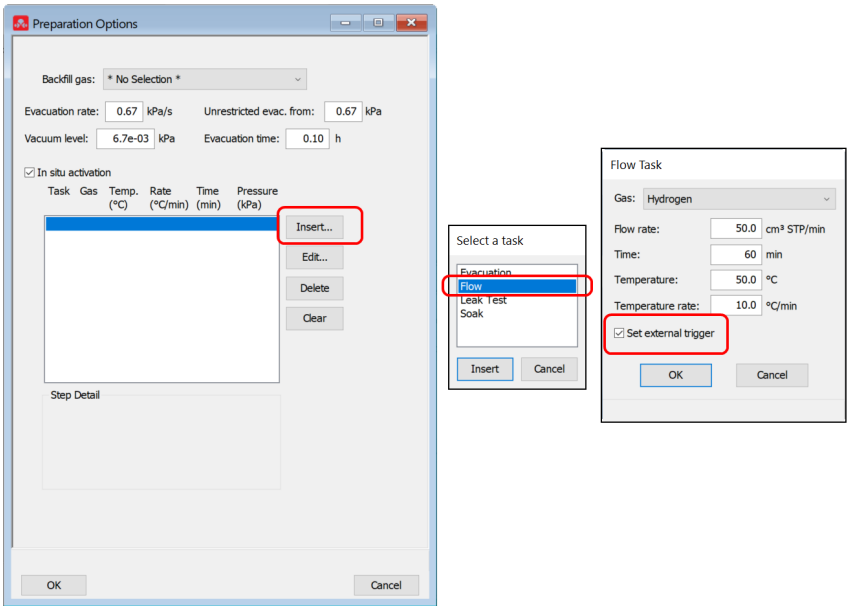
- Preset in the analysis conditions parameter file, or
- Set manually using the analyzer schematic

PRESET IN ANALYSIS CONDITIONS

1. Go to ***File > New Sample***.
2. On the *New Sample Type* window, select **Chemical Adsorption**.
3. On the *Analysis Conditions* tab, click **Preparation**.



4. On the *Preparation Options* window, click **Insert**.



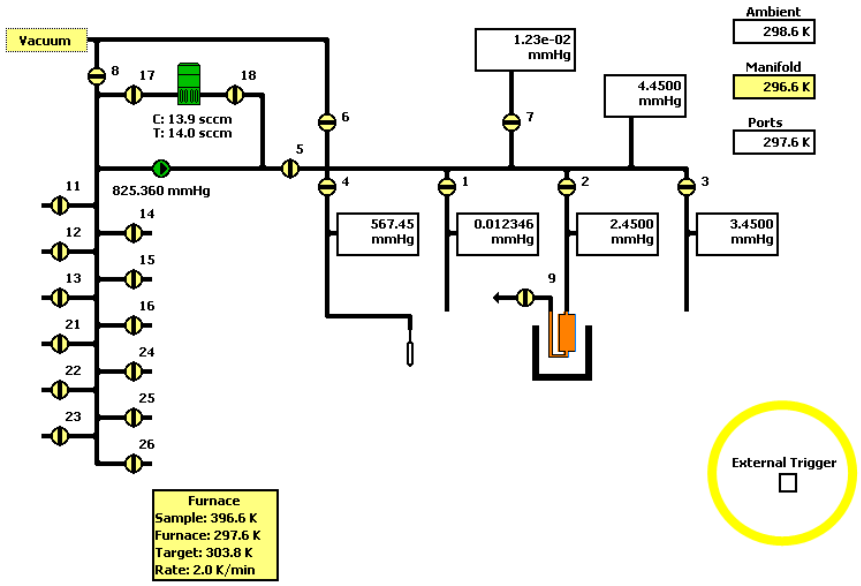
5. On the *Select a task* window, select *Flow*, then click **Insert**.
6. On the *Flow Task* window, ensure *Set external trigger* is selected, then click **OK**.

Minimum recommended flow rate is 50 sccm.

7. On the *Preparation Options* window, click **OK**.
8. On the *Analysis Conditions* window, click **Save As** to save the sample file parameter changes.

SET MANUALLY IN THE ANALYZER SCHEMATIC

1. Go to ***Unit [n] > Chemical Adsorption*** (if not already selected).
2. Use *Enable Manual Control* to enable the manual control of certain system valves and pump components on the analyzer schematic. When this option is enabled, a checkmark appears to the left of ***Unit [n] > Enable Manual Control***. If the analyzer schematic is not immediately visible, go to ***Unit [n] > Show Instrument Schematic***.
3. Right-click the *External Trigger* icon, then select *On*. Alternately, double click the *External Trigger* icon.



External Trigger icon circled in yellow

The *External Trigger* icon changes to a solid black box to indicate the external trigger is enabled.

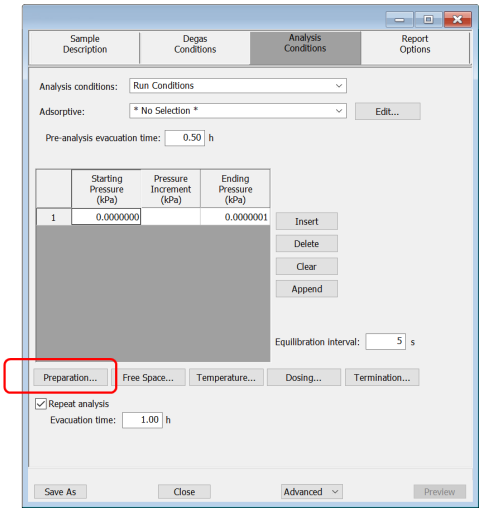
ENABLE THE TRIGGER FOR ASAP 2020 PLUS CHEMISORPTION

The trigger can be enabled using one of two methods:

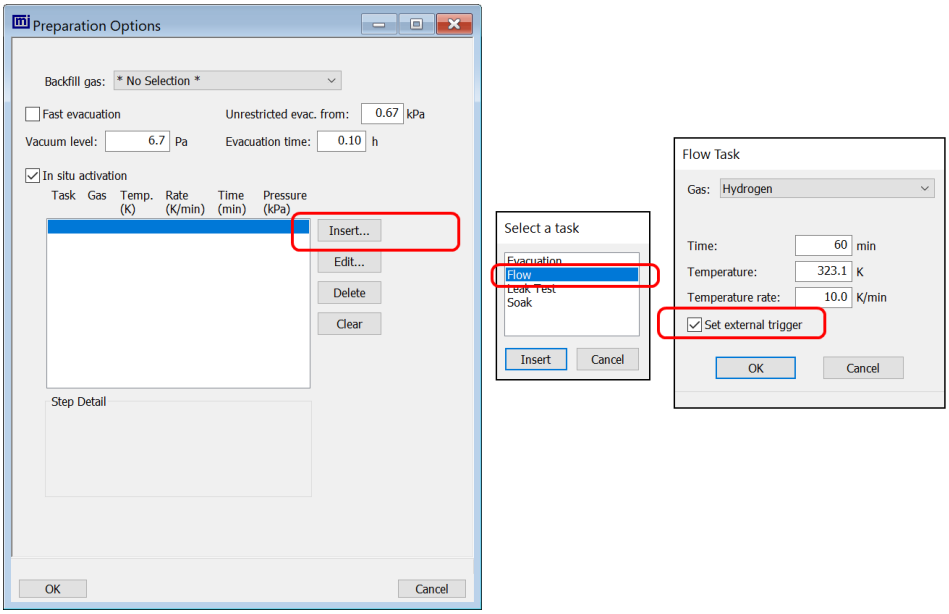
- Preset in the analysis conditions parameter file, or
- Set manually using the analyzer schematic

PRESET IN ANALYSIS CONDITIONS

1. Go to ***File > New Sample***.
2. On the *Analysis Conditions* tab, click **Preparation**.



3. On the *Preparation Options* window, click **Insert**.

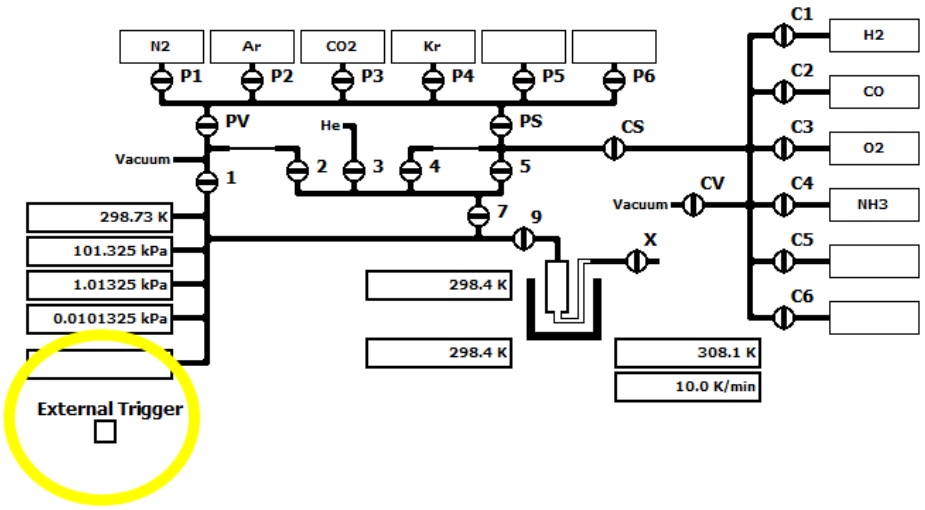


4. On the *Select a task* window, select *Flow*, then click **Insert**.
5. On the *Flow Task* window, ensure *Set external trigger* is selected, then click **OK**.

Minimum recommended flow rate is 50 sccm.

6. On the *Preparation Options* window, click **OK**.
7. On the *Analysis Conditions* window, click **Save As** to save the sample file parameter changes.
8. Use *Enable Manual Control* to enable the manual control of certain system valves and pump components on the analyzer schematic. When this option is enabled, a checkmark appears to the left of ***Unit [n] > Enable Manual Control***. If the analyzer schematic is not immediately visible, go to ***Unit [n] > Show Instrument Schematic***.
9. Right-click the *External Trigger* icon, then select *On*. Alternately, double click the *External Trigger* icon.

SET MANUALLY IN THE ANALYZER SCHEMATIC



External Trigger icon shown in yellow circle

The *External Trigger* icon changes to a solid black box to indicate the external trigger is enabled.

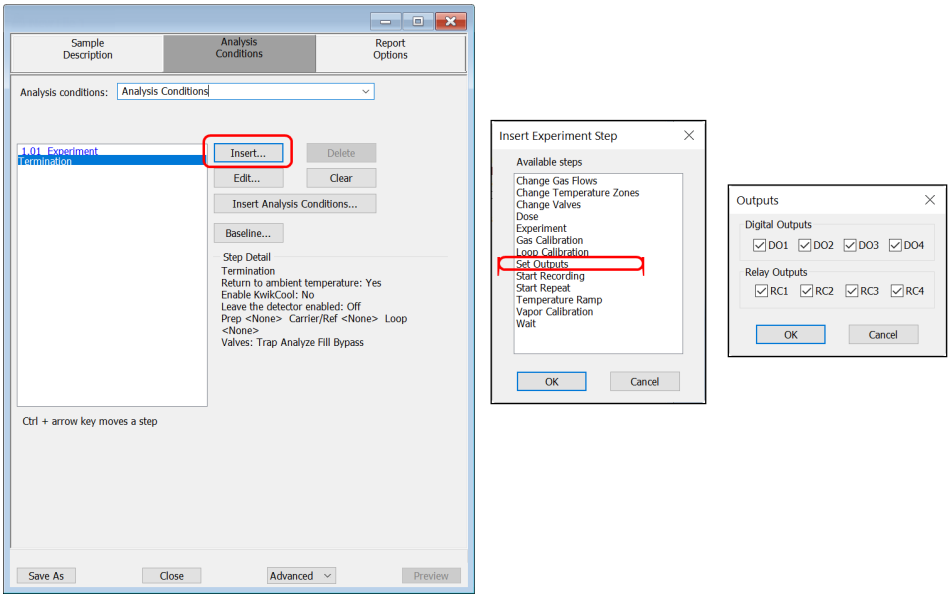
ENABLE THE TRIGGER FOR AUTOCHEM 2920 AND 2950

The trigger can be enabled on the AutoChem using one of two methods:

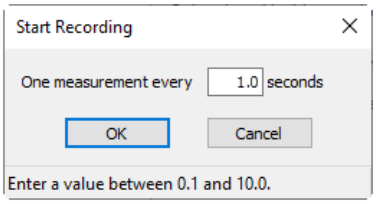
- Preset in the analysis conditions parameter file, or
- Set manually using the analyzer schematic

PRESET IN ANALYSIS CONDITIONS

1. On the *Analysis Conditions* tab, insert a *Set Outputs* step before the *Start Recording* step.
   a. Click **Insert**.
   b. Select *Set Outputs*.
   c. Click **OK**.
   d. Select digital output *D01* to be enable the trigger.
   e. Click **OK**.



2. Enter a *Start Recording* step.
   a. Click **Insert**.
   b. Select *Start Recording* and enter the recording interval.

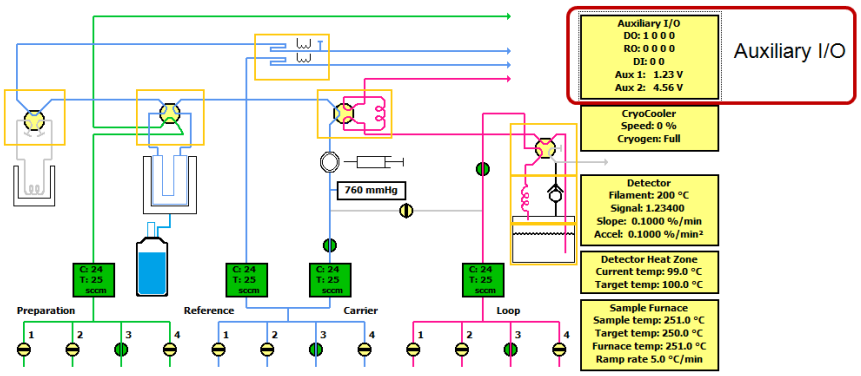


   c. Click **OK**.
3. A *Stop Recording* step is automatically entered after the *Start Recording* step. After the *Stop Recording* step, insert a *Set Outputs* step following the steps shown above.
4. Disable digital output *D01*.

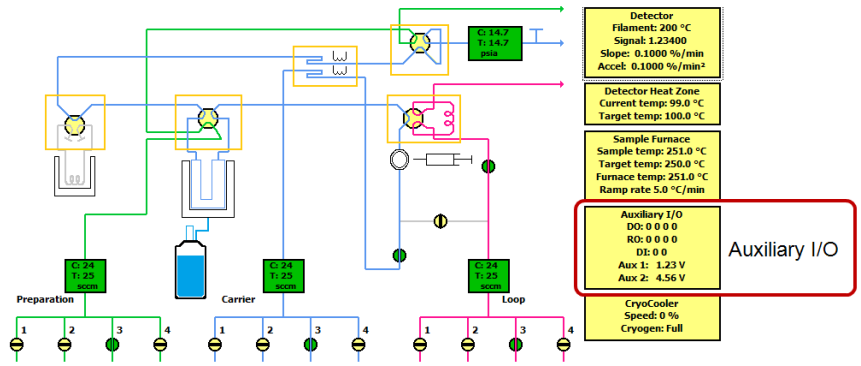
SET MANUALLY IN THE ANALYZER SCHEMATIC

The *External Trigger* icon changes to a solid black box to indicate the external trigger is enabled.

1. Enable manual control to display the schematic, then right-click in the *Auxiliary I/O display* window.
2. Select *Digital Output 1*.



AutoChem 2920



AutoChem HP 2950

ENABLE THE TRIGGER FOR AUTOCHEM III

The trigger can be enabled on the AutoChem III using one of two methods:

- Preset in the analysis conditions parameter file, or
- Set manually using the analyzer schematic

PRESET IN ANALYSIS CONDITIONS

The external trigger for the mass spectrometer can be enabled in either the:

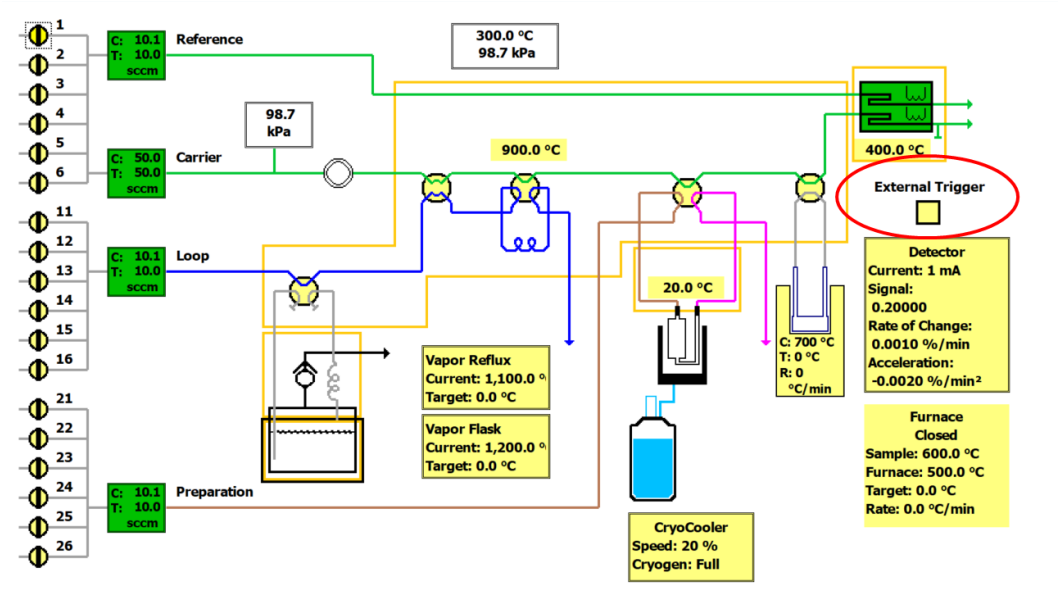
- Start Recording step
- Gas Calibration step
- Vapor Flow Calibration step, or
- Vapor Pulse Calibration step

On the *Analysis Conditions* tab, insert one of the above steps and enable the *Set external trigger* option and enter other settings as needed.

SET MANUALLY IN THE ANALYZER SCHEMATIC

The *External Trigger* icon changes to a solid black box to indicate the external trigger is enabled.

Enable manual control to display the schematic, then double-click (or right-click) the *External Trigger* checkbox on the schematic and select ON.



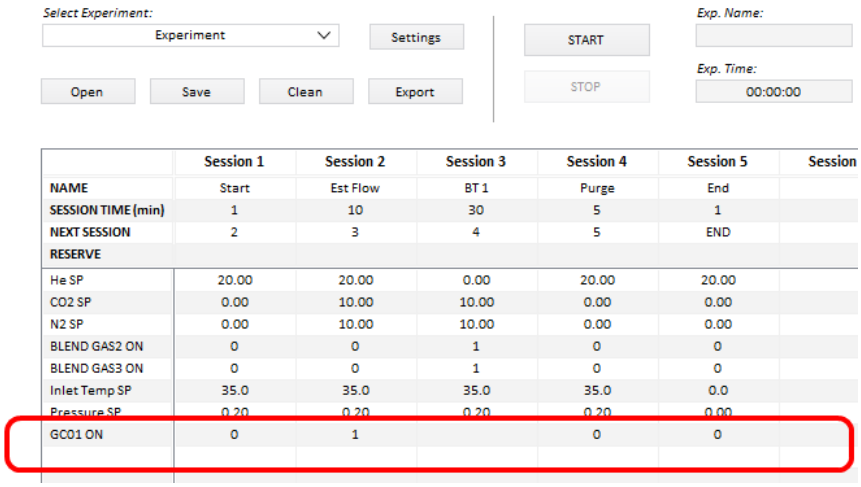
ENABLE THE TRIGGER FOR BTA OR FR-SERIES

The trigger can be enabled on the Breakthrough Analyzer or FR-series using one of two methods:

- Preset in the *Session* tab by enabling GC01
- Set manually in the *Panel* tab by enabling the GC01 signal

PRESET IN THE SESSION TAB

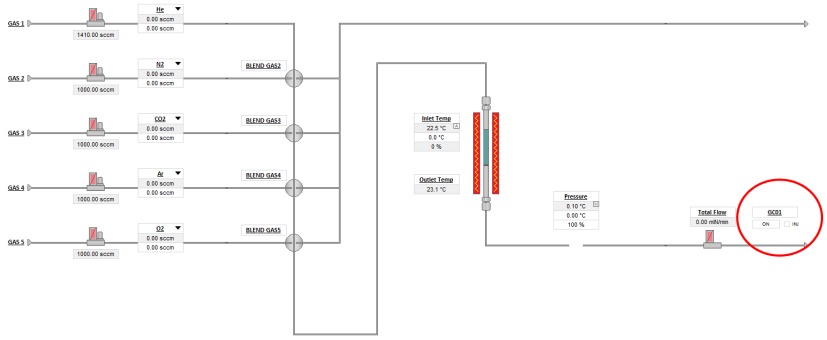
The external trigger to the mass spectrometer can be enabled by setting the parameter *GC01 ON* to *1* in a session file. Setting the value to *0* will disable the external trigger. By default, the GC01 external trigger will be set to the last value if the column is left blank during a session.



	Session 1	Session 2	Session 3	Session 4	Session 5	Session
NAME	Start	Est Flow	BT 1	Purge	End	
SESSION TIME (min)	1	10	30	5	1	
NEXT SESSION	2	3	4	5	END	
RESERVE						
He SP	20.00	20.00	0.00	20.00	20.00	
CO2 SP	0.00	10.00	10.00	0.00	0.00	
N2 SP	0.00	10.00	10.00	0.00	0.00	
BLEND GAS2 ON	0	0	1	0	0	
BLEND GAS3 ON	0	0	1	0	0	
Inlet Temp SP	35.0	35.0	35.0	35.0	0.0	
Pressure SP	0.20	0.20	0.20	0.20	0.00	
GC01 ON	0	1		0	0	

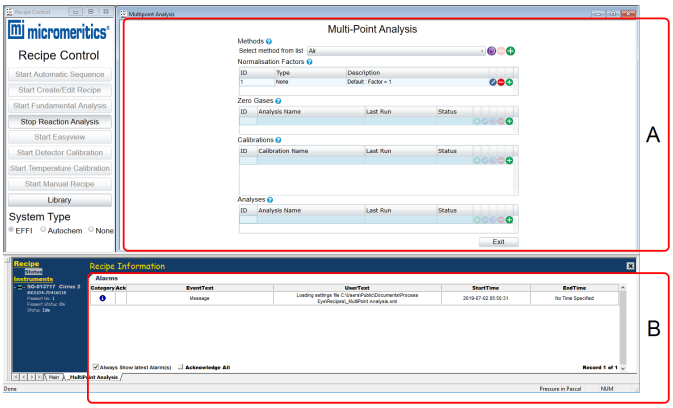
SET MANUALLY IN THE PANEL TAB

The external trigger to the mass spectrometer can be manually enabled by pressing the ON/OFF GC01 button on the main panel screen. The ON/OFF button will turn green when the external trigger is enabled.



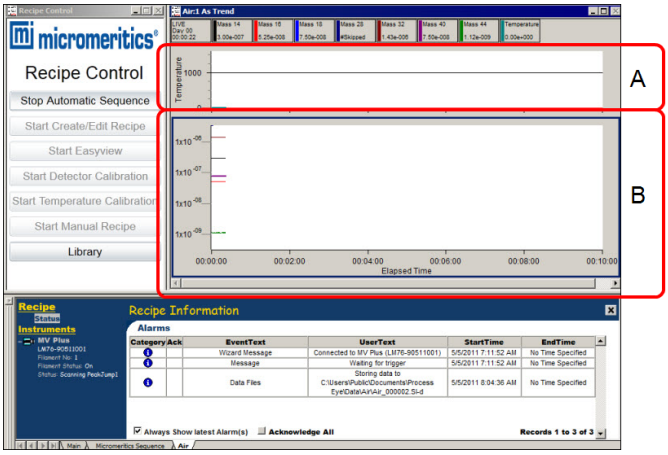
DATA COLLECTION

After the trigger is enabled, the recipe is launched and data collection begins. A message on the *Recipe Information* window states that data collection has started and displays the directory where the data file is saved.



A. Area showing where settings are loaded
B. Message area indicating stored data location

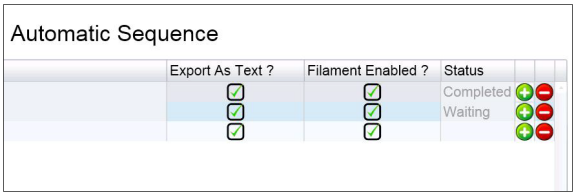
Two windows are opened, one with atomic mass trends and one with the scaled analyzer sample temperature, where mass spectra parameters and temperature axes can be edited. See ***Recipe Control on page 2 - 1***.

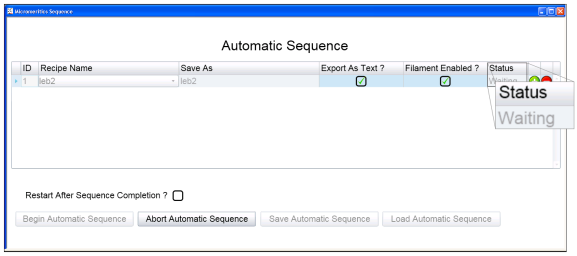


A. Sample temperature
B. Atomic mass trends

CLOSE THE DATA FILE

When the Micromeritics analyzer *Digital Output 1* is set to low (0), the recipe stops running and closes the data file. The *Trend View* windows are closed.



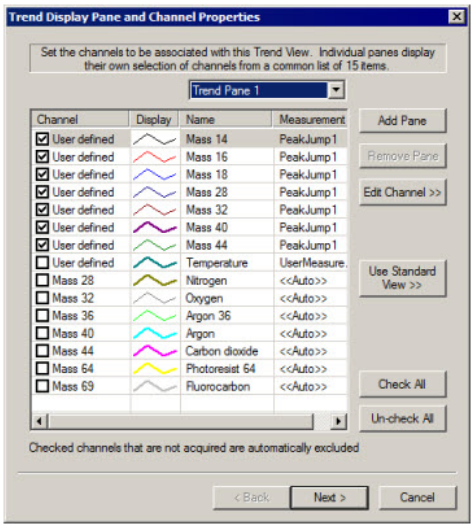


On the *Automatic Sequence* window, the *Status* column shows *Completed* for the completed recipe. If there is more than one recipe in the sequence list, the next recipe in the sequence initializes and *Waiting* displays. Once the digital trigger is enabled for the recipe with a *Waiting* status, the recipe launches and data collection begins.

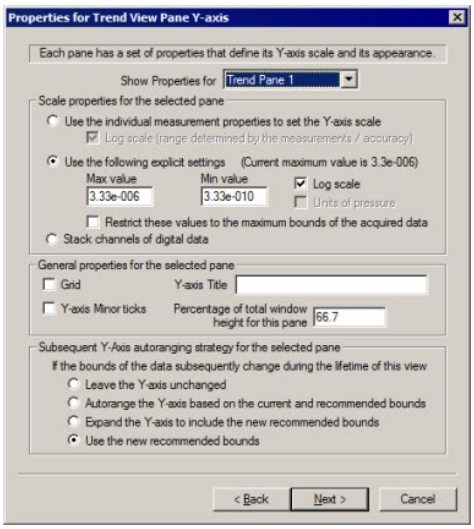
Axes and Legend Properties

To change the axes and legend properties:

1. Right-click on the graph where changes are to be made.



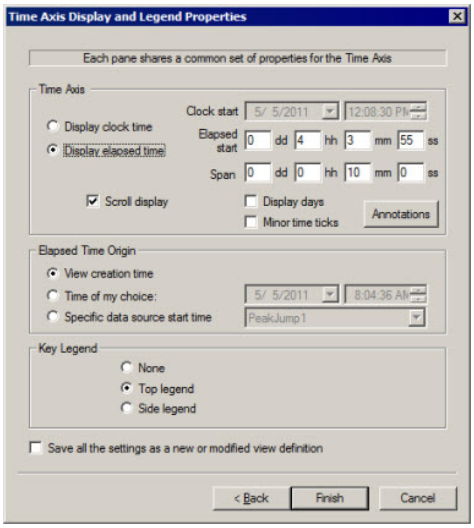
2. On the *Trend Display Pane and Channel Properties* windows, select the check-boxes in the *Channel* column to display or hide a mass.
3. From the *Trend Pane*, select:
   - **Trend Pane 1.** Select for the atomic mass spectra.
   - **Trend Pane 2**. Select for the Micromeritics analyzer sample temperature.
4. Click **Next**.



5. On the *Properties for Trend View Pane Y-axis* window, edit the values in the *Max value* and *Min value* fields.

These value changes apply limits to the partial pressure for the mass spectra (*Trend Pane 1*) and temperature scale for the Micromeritics analyzer (*Trend Pane 2*).

6. Select *Log scale* to display the y-axis in logarithmic form. Leave it unchecked to display the y-axis in linear form.
7. Edit other window parameters as needed, then click **Next**.

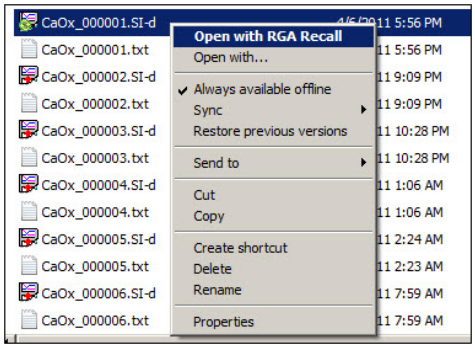


8. On the *Time Axis Display and Legend Properties* window, select *Display elapsed time* to change the elapsed time displayed on the time axis (x-axis).
9. Edit the *Elapsed start* and *Span* fields, if applicable. The default span time is 10 minutes.
10. Select *Save all the settings as a new or modified view definition* to have the modified settings saved as the defaults.
11. Click **Finish** to save changes and return to the graph display.

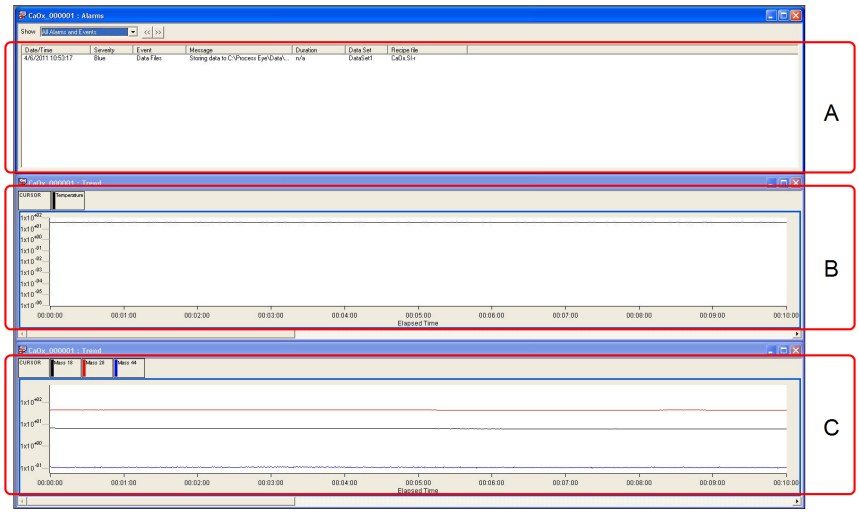
DATA AND RGA RECALL

Only a limited amount of spectra and temperature data is stored in the data buffer used for the live display; therefore, some data may appear truncated when viewing in *Process Eye*. An auxiliary program called *RGA Recall* has been included so that the entire set of data can be reviewed. To access *RGA Recall*:

1. In Windows Explorer, navigate to the applicable data directory, then locate the data file.
2. Right-click the data file, then select *Open with RGA Recall* from the menu.



The entire data set displays. Certain parameters of the mass spectra and temperature axes panes can then be edited.

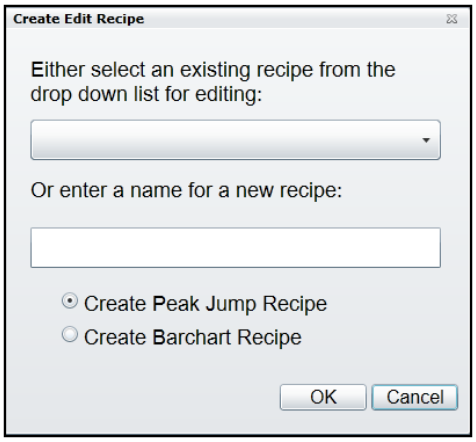


A. Recipe data set
B. Sample temperature
C. Atomic mass trends

If the data file being opened is currently running, the file will not open. A prompt displays indicating either to open a copy of the file or wait until the file is inactive before trying *RGA Recall* again.

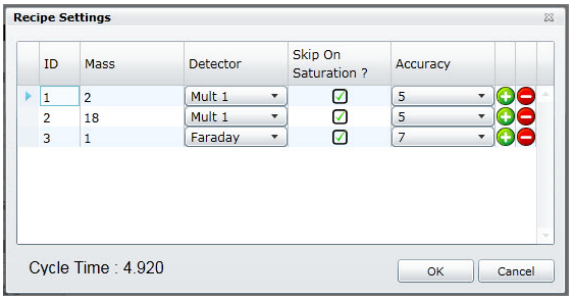
4 CREATE / EDIT RECIPE

Use to create, configure, or edit *Peak Jump Recipes* for the *Automatic Sequence* mode. A recipe specifies the actions that the Cirrus software is to take during data acquisition.



The Micromeritics analyzer sample thermocouple signal is included in each *Peak Jump* recipe created.

1. Select a recipe from the drop-down list, or enter the name for a new one. Click **OK**.



2. On the *Recipe Settings* window, use **(+)** and **(–)** buttons to add or delete a mass to the *Peak Jump* scan.
   - **(+) sign.** Adds a mass below the selected one. The ID is added automatically. All fields contain program defaults.
   - **(-) sign.** Deletes the selected mass.
3. Select and edit the *Mass* field as needed. See *Library on page 9 - 1* if mass spectra information is needed.

4. Click the down arrow in the *Detector* column to choose the detector to be used for the selected mass. Select the *Faraday* detector or any of the three gain configurations from the detector options.

Scanning for concentrated species (> 20% of mixture composition) on the Mult1, Mult2, or Mult3 detector is not recommended. For recipes where a short cycle time is crucial, use only one detector (Faraday, Mult1, Mult2, or Mult3). Switching between detectors involves changing settings for internal electrical components, resulting in a high cycle time.

5. Select the *Skip On Saturation* checkboxes (recommended). When selected, masses that saturate the SEM detector (Mult1, Mult2, or Mult3) will be removed from the recipe after five consecutive saturated measurements.

Saturating the SEM detector with a high concentration of ions may damage the detector.

6. Click the down arrow on options in the *Accuracy* column, then select the accuracy level. This parameter controls the time for converting the signal in the analog to digital electronics. The range is *0* (fastest scan and cycle rate with the lowest noise filtering) to *8* (longest scan with the highest noise filtering).

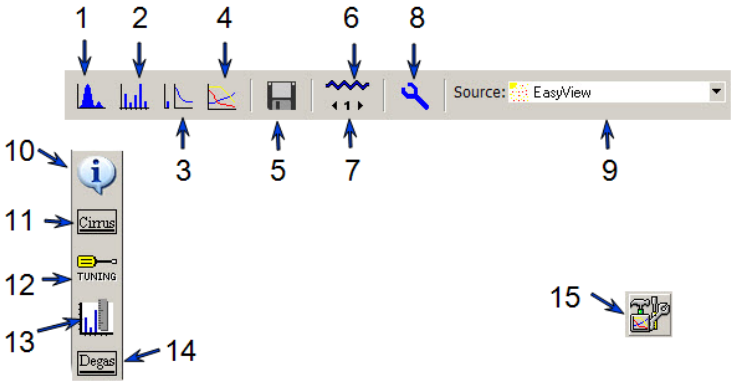
For recipes where a short cycle time is crucial, select the same accuracy level for all masses.

7. Click **OK** to save changes and close the window. Click **Cancel** to discard all changes.

5 EASYVIEW

To launch the *Easyview* software suite, click **Start Easyview** on the *Recipe Control* window to access typical RGA operations. When *EasyView* is activated, the **Start Easyview** button on the *Recipe Control* window changes to **Stop Easyview**. Click **Stop Easyview** to exit the *Easyview* application.

For additional information on the following toolbars, refer to the *Easyview* and *Process Eye User Guides.*



Easyview Functions Table

Legend	Description
1	Starts or stops Analog scan mode.
2	Starts or stops Barchart scan mode.
3	Starts or stops Leak Check scan mode.
4	Starts or stops Peak Jump scan mode. When started, adds the **Peak Settings** button to the *Easyview* window. See legend item 15 for **Peak Settings** button information.
5	Disk store. Allows data to be saved.
6	Filament status. Turns active filament off (blue) and on (red).
7	Filament number. Toggles between filaments.
8	Performs a diagnostic on the Cirrus electronics and associated sensor.
9	Select an option to change the electron energy for ionization (generally, 40eV or 70 eV).
10	Returns the status window to the *General Information* page.
11	Displays the status of the turbo pump, manifold heater, and capillary heater.

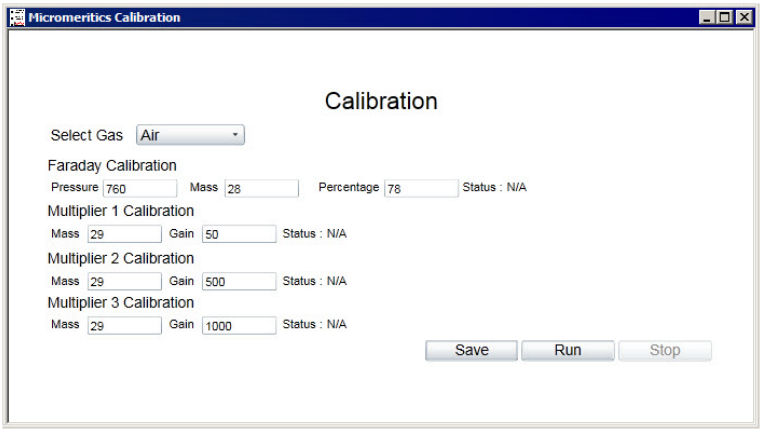
Easyview Functions Table (continued)

Legend	Description
12	Activates *Tuning* mode. Use to adjust the tuning of the RGA (peak alignment and resolution).
13	Calibration mode. Provides a calibration factor calculated for converting the measured ion-current to the gas being used. It is recommended to use the **Start Detector Calibration** on the *Recipe Control* pane for this calibration.
14	Opens the *Degas Settings* page for degassing the ion source of selected MKS RGAs.
15	Visible when *Peak Jump* mode is activated. Use to edit recipe parameters.

6 DETECTOR CALIBRATION

Use *Start Detector Calibration* to calibrate the Faraday and SEM detectors. Detector calibration is recommended at the beginning of each workday when the Cirrus analyzer is used on a daily basis.

- **Faraday calibration.** Compares the Faraday detector ion current associated with a particular mass against the known partial pressure of that mass. A Faraday calibration defines the basic ion source sensitivity in amps per pressure unit for a given set of ion source conditions.
- **Multiplier calibration**. Uses the basic ion source sensitivity to find a voltage that amplifies the ion source signal by a given gain setting. The multiplier calibration uses mass fragments with signals less intense than the fragment used for the Faraday calibration.



The calibration may be performed with a variety of gas species, both pure and mixtures. The composition of the gas must be well known.

CONFIGURATION SETTINGS

Before specifying calibration parameters, review the recommended calibration configuration settings.

Calibration Configuration Settings Table

Gas	Pressure	Faraday Mass	Percentage	Multiplier Mass
Air	760	28	78	29
Pure nitrogen	760	28	100	29
Pure argon	760	40	100	36
Pure helium	760	4	100	8

CALIBRATION PARAMETERS

1. Click the *Select Gas* down arrow, then choose the applicable gas.
2. Follow the instructions in the following *Calibrations Instructions Table* for each calibration entered.

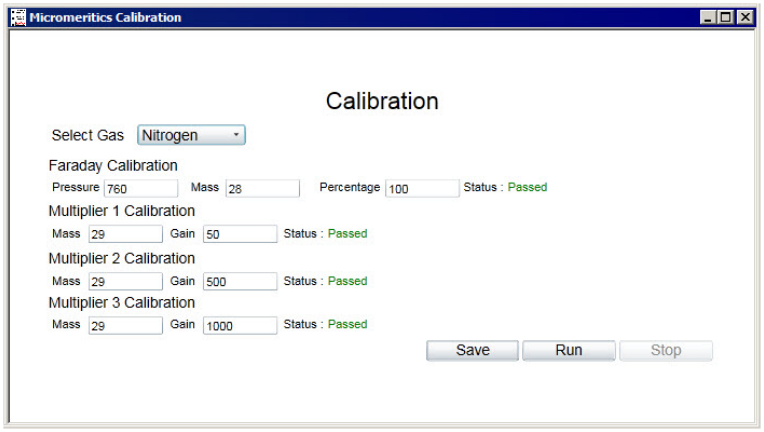
Calibration Instructions Table

Type	Instructions
Faraday	■ Enter the inlet pressure (in Torr) in the *Pressure* field. The typical atmospheric pressure entry of *760* is used. ■ Enter the mass in the *Mass* field. Typically, this is the mass of dominant species in the calibration gas. If using pure argon as the calibration gas, the entered mass would be *40*. If using air as the calibration gas, the entered mass would be *28* for nitrogen. ■ In the *Percentage* field, enter the percentage contribution of the partial pressure of the selected mass to the inlet pressure entered previously. If using pure argon as the calibration gas, enter *100* for the contribution of mass *40*. If using air as the calibration gas, enter *78* for the contribution of mass *28* (diatomic nitrogen).
Multiplier	■ Enter a mass that will not saturate the SEM detector. This is the mass used to perform a calibration against the comparative Faraday detector reading. If using pure argon as the calibration gas, enter mass *36* (has a 0.337% contribution to the partial pressure of pure argon). If using air as the calibration gas, enter mass *29* (has a 0.57% contribution to the partial pressure of air). ■ Use the same mass for all three multiplier detectors. ■ The values in the *Gain* fields are appropriate in most cases and should remain unchanged.

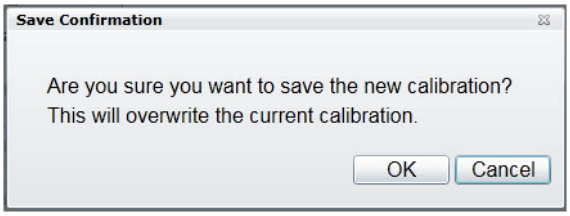
BEGIN CALIBRATION

1. Click **Run** to begin calibration. The Faraday calibration and SEM calibrations will run sequentially.

The *Status* field will change from *N/A* to *Passed* if the calibrations completed a successful run.



2. Click **Save**.



3. On the *Save Confirmation* window, click **OK** to overwrite the current calibration and close the window.
4. Click **Stop Detector Calibration** to close the *Calibration* window.

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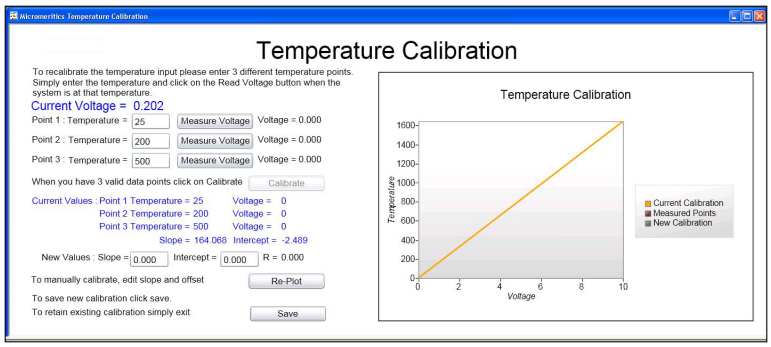
7 TEMPERATURE CALIBRATION

START TEMPERATURE CALIBRATION

Use *Start Temperature Calibration* to calibrate the Micromeritics analyzer sample thermocouple signal.



When using the standard Micromeritics analyzer-MKS Cirrus interface cable, the thermocouple signal is transmitted to the Cirrus on analog input channel 4. To collect temperature data, the analog input signal (in volts) must be scaled to temperature in degrees Celsius (ºC).

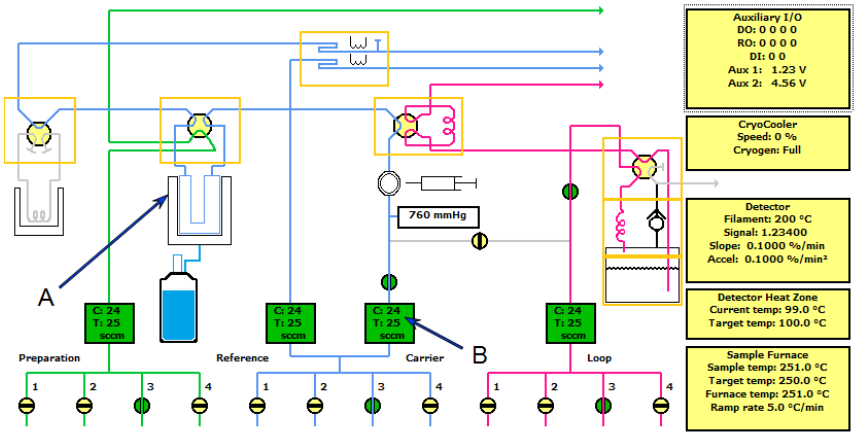
The Micromeritics analyzers only use a single Digital I/O interface cable to connect to the mass spectrometer.

FLEX AND ASAP 2020 PLUS CHEMISORPTION ANALYZERS

The Flex 3500 Chemisorption analyzer and the ASAP 2020 Plus Chemisorption analyzer do not provide an analog temperature signal to the mass spectrometer, so there is no corresponding temperature calibration necessary. These analyzers transmit elapsed time versus sample temperature data in ASCII format every half second while the mass spectrometer signal is active via Ethernet on TCP/IP port 54200. This data can be captured using any TCP/IP capable data capture software or with a Python script. The captured data must then be combined with the mass spectrometer data for use.

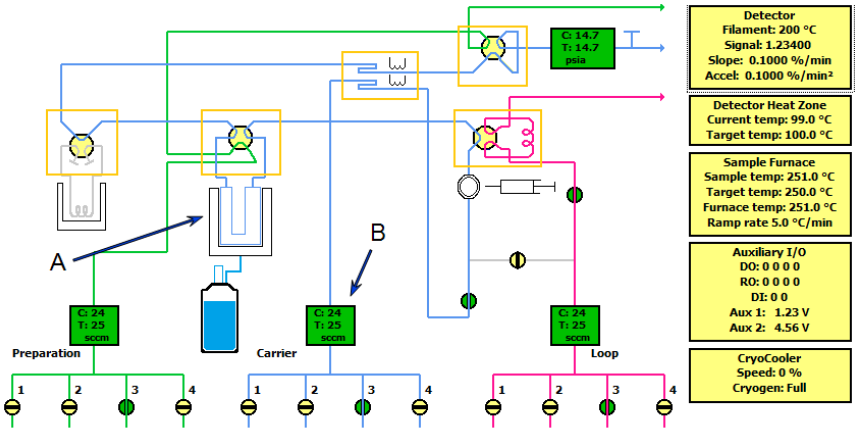
AUTOCHEM 2920 AND 2950 ANALYZERS

1. Ensure that the AutoChem analyzer furnace is closed and a sample tube is installed.
2. Start the AutoChem application.
3. Go to ***Unit [n] > Enable Manual Control***.
4. Set the analysis valve to *Analyze*.
5. Ensure gas is flowing at a rate similar to experimental conditions.



AutoChem 2920

A. Furnace
B. Gas flow (50 sccm)

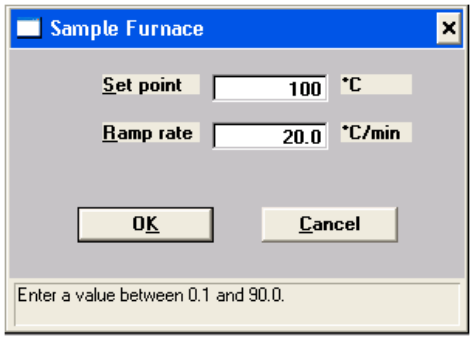


AutoChem 2950

A. Furnace
B. Gas flow (50 sccm)

POINT 1: TEMPERATURE CALIBRATION

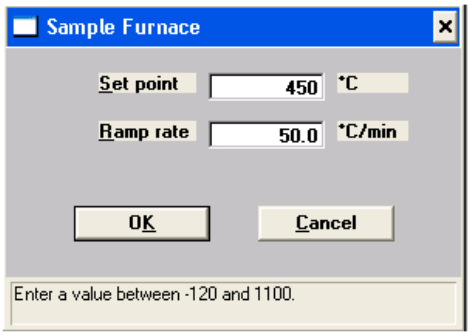
1. Right-click the furnace icon on the schematic, then select *Set Temperature*.
2. On the *Sample Furnace* window, enter *100* in the *Set point* field, then enter *20* in the *Ramp rate* field.



3. Click **OK**.
4. Observe the *Sample Temperature* reading on the schematic. When the sample temperature reaches approximately 100 ºC and stabilizes, enter the sample temperature in the *Point 1: Temperature* field on the *Process Eye Professional Temperature Calibration* window.
5. Click **Measure Voltage** for Point 1. The analog input voltage will be associated with the AutoChem analyzer sample temperature and the measured voltage displayed at the end of the row.

POINT 2: TEMPERATURE CALIBRATION

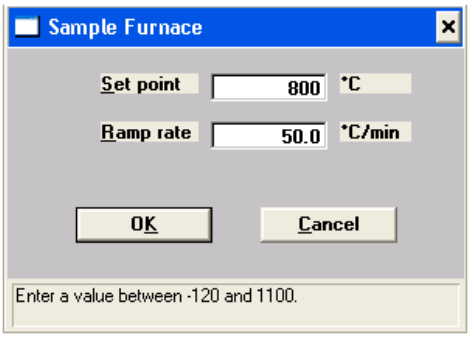
1. Right-click the furnace icon on the schematic, then select *Set Temperature*.
2. On the *Sample Furnace* window, enter *450* in the *Set point* field, then enter *50* in the *Ramp rate* field.



3. Click **OK**.
4. Observe the *Sample Temperature* reading on the schematic. When the sample temperature reaches approximately 450 ºC and stabilizes, enter the sample temperature in the *Point 2: Temperature* field on the *Process Eye Professional Temperature Calibration* window.
5. Click **Measure Voltage** for Point 2. The analog input voltage will be associated with the analyzer sample temperature and the measured voltage displayed at the end of the row.

POINT 3: TEMPERATURE CALIBRATION

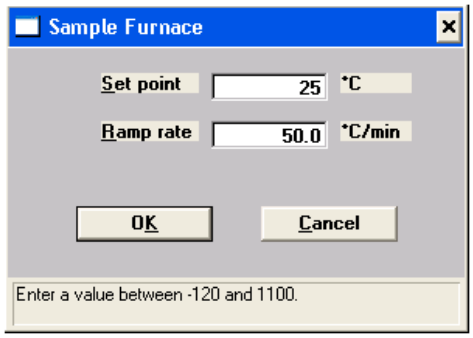
1. Right-click the furnace icon on the schematic, then select *Set Temperature*.
2. On the *Sample Furnace* window, enter *800* in the *Set point* field, then enter *50* in the Ramp rate field.



3. Click **OK**.
4. Observe the *Sample Temperature* reading on the schematic. When the sample temperature reaches approximately 800 ºC and stabilizes, enter the sample temperature in the *Point 3: Temperature* field on the *Process Eye Professional Temperature Calibration* window.
5. Click **Measure Voltage** for Point 3. The analog input voltage will be associated with the analyzer sample temperature and the measured voltage displayed at the end of the row.

RETURN THE FURNACE TO ROOM TEMPERATURE

1. Right-click the furnace icon, then select *Set Temperature*.
2. On the *Sample Furnace* window, enter *25* in the *Set point* field, then enter *50* in the *Ramp rate* field.

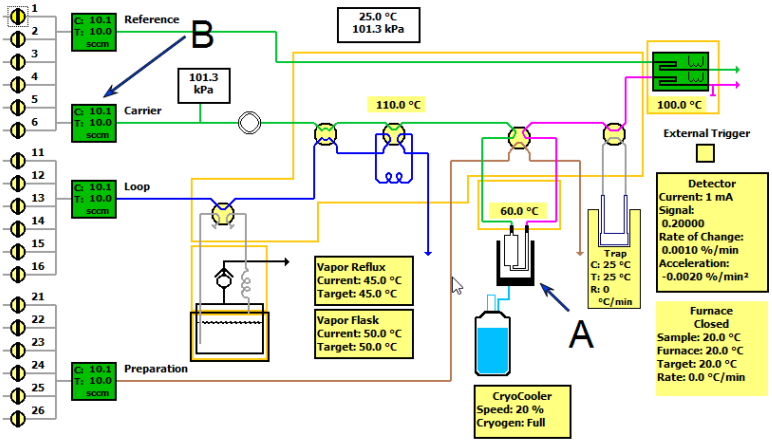


3. Click **OK**.

Attaching the CryoCooler will speed up this process.

AUTOCHEM III ANALYZERS

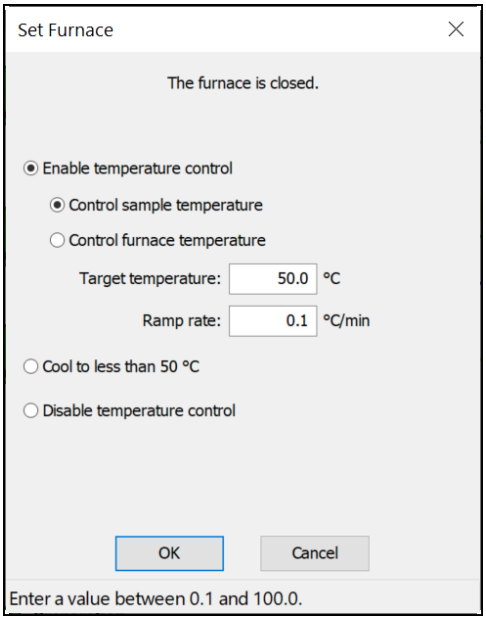
1. Ensure that the analyzer furnace is closed and a sample tube is installed.
2. Start the AutoChem application.
3. Go to ***Unit [n] > Enable Manual Control***.
4. Set the analysis valve to *Analyze*.
5. Ensure gas is flowing at a rate similar to experimental conditions.



A. Furnace
B. Gas flow (50 sccm)

POINT 1: TEMPERATURE CALIBRATION

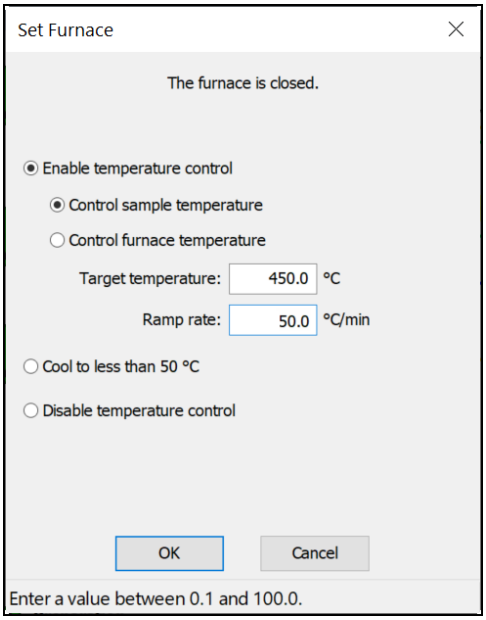
1. Right-click the furnace box on the schematic, then select *Set*.
2. On the *Set Furnace* window, select *Enable temperature control and change the Target temperature to 50 °C.*



3. Click **OK**.
4. Observe the *Sample Temperature* reading on the schematic. When the sample temperature reaches approximately 100 ºC and stabilizes, enter the sample temperature in the *Point 1: Temperature* field on the *Process Eye Professional Temperature Calibration* window.
5. Click **Measure Voltage** for Point 1. The analog input voltage will be associated with the analyzer sample temperature and the measured voltage displayed at the end of the row.

POINT 2: TEMPERATURE CALIBRATION

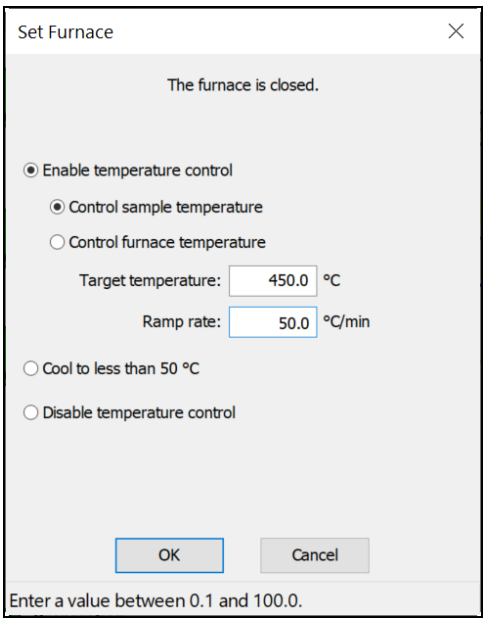
1. Right-click the furnace box on the schematic, then select *Set*.
2. On the *Set Furnace* window, select *Enable temperature control. and change the Target temperature* to 450 °C and 50 °C in the *Ramp rate* field.



3. Click **OK**.
4. Observe the *Sample Temperature* reading on the schematic. When the sample temperature reaches approximately 400 ºC and stabilizes, enter the sample temperature in the *Point 2: Temperature* field on the *Process Eye Professional Temperature Calibration* window.
5. Click **Measure Voltage** for Point 2. The analog input voltage will be associated with the analyzer sample temperature and the measured voltage displayed at the end of the row.

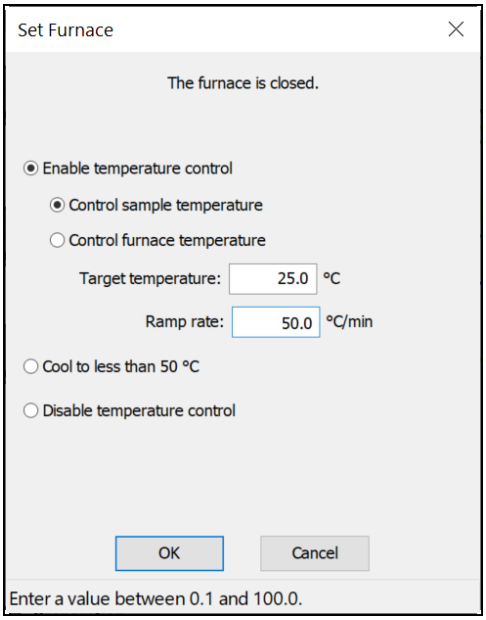
POINT 3: TEMPERATURE CALIBRATION

1. Right-click the furnace box on the schematic, then select *Set*.
2. On the *Set Furnace* window, select *Enable temperature control. and change the Target temperature* to 450 °C and 50 °C in the *Ramp rate* field.
3. Click **OK**.
4. Observe the *Sample Temperature* reading on the AutoChem instrument schematic. When the sample temperature reaches approximately 800 ºC and stabilizes, enter the sample temperature in the *Point 3: Temperature* field on the *Process Eye Professional Temperature Calibration* window.
5. Click **Measure Voltage** for Point 3. The analog input voltage will be associated with the AutoChem analyzer sample temperature and the measured voltage displayed at the end of the row.



RETURN THE FURNACE TO ROOM TEMPERATURE

1. Right-click the furnace box on the schematic, then select *Set*.
2. On the *Set Furnace* window, select *Enable temperature control. and change the Target temperature* to 25 °C and 50 °C in the *Ramp rate* field.



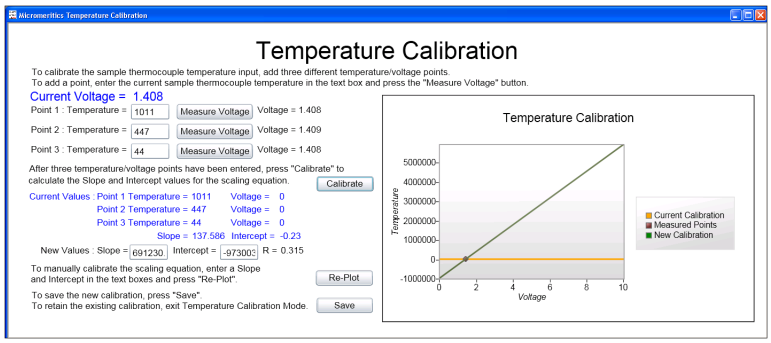
3. Click **OK**.

Attaching the CryoCooler will speed up this process.

ENABLE CALIBRATION

After all three temperatures have been entered and associated with input voltages, the **Calibrate** button on the *Process Eye Professional Temperature Calibration* window is enabled.

1. Click **Calibrate**. A linear fit is applied to the collected points to determine a scaling equation. The temperatures, associated analog voltage values, and the Slope and Intercept results are displayed.

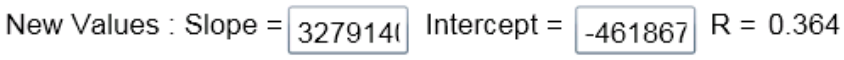


2. Click **Save**.
3. On the *Save Confirmation* window, click **OK** to overwrite the current calibration and close the window.
4. Click **Stop Temperature Calibration** on the *Recipe Control* pane to close the calibration window.

ENTER A SCALING EQUATION

As an alternative to fitting a line to three temperature and analog signal values, a scaling equation can be manually entered for the thermocouple signal.

1. Where *New Values* is displayed, enter the applicable values in the *Slope* and *Intercept* fields.



2. Click **Re-Plot** to display the new scaling function on the chart.
3. Click **Save**.
4. Click **OK** to overwrite the current calibration and close the window.
5. Click **Stop Temperature Calibration** on the *Recipe Control* window to close the calibration window.

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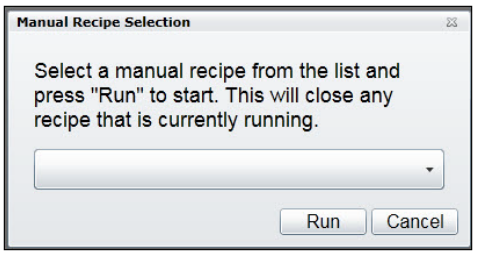
8 MANUAL RECIPE

The *Start Manual Recipe* option provides access to recipes created using the *Recipe Wizard*. For detailed instructions on using the *Recipe Wizard*, refer to the *Recipe Wizard User Guide* included with the Cirrus II documentation.



1. Click the down arrow to select the applicable recipe from the list.
2. Click **Run**.
3. Click **Stop Manual Recipe** on the *Recipe Control* window to close the window.

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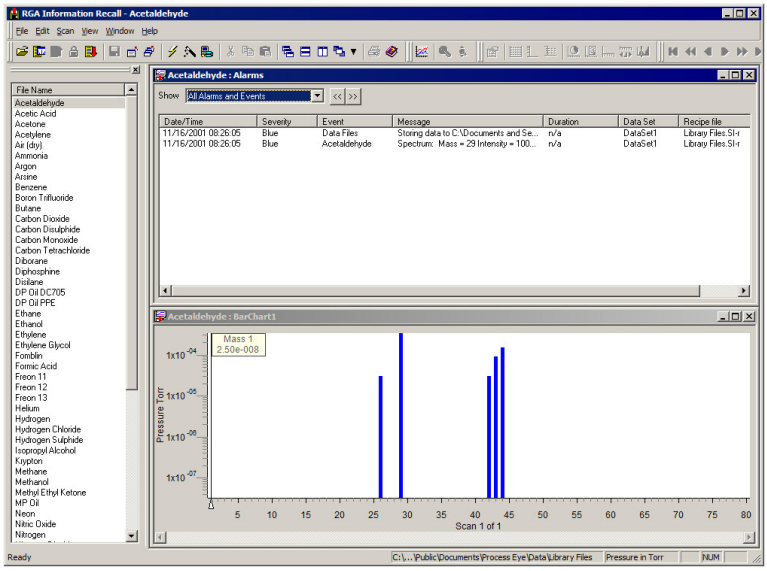
9 LIBRARY

The Library provides access to mass spectra for a variety of compounds.



1. In the *File Name* list, double click the applicable compound to display mass spectral data.
2. Click the **X** in the upper right corner to close the window.

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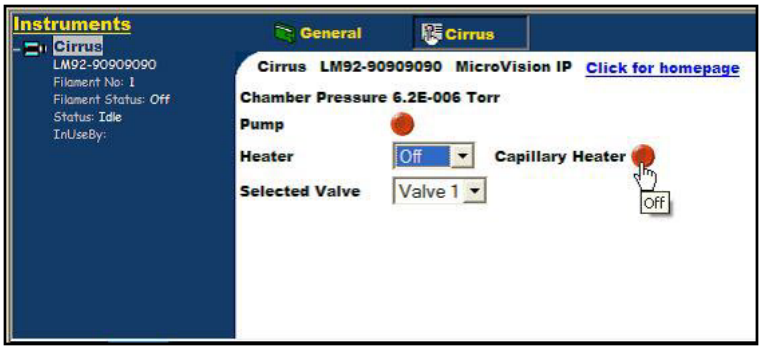
10 POWER OFF THE MASS SPECTROMETER

The mass spectrometer should be allowed to run continuously unless it will not be used for an extended period of time or needs to be powered down for maintenance.

1. Power OFF the capillary and system heaters.
2. Power OFF the filaments.



3. Wait 10 minutes to allow the filaments to cool.
4. Click the **Pump** button to stop the turbo. The pumps will stop and the vent valve will open after a few minutes.
5. Wait 5 minutes for the system to cool further and come to a full stop.

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