

# ***MICROMERITICS-CIRRUS***

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*INTERFACE*



***USER GUIDE***

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292-42804-00  
Sep 2016  
(Rev D)

## ***TRADEMARKS***

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## ***ABOUT THIS MANUAL***

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The following icons may be found in this document:



**NOTE** - Notes contain important information pertinent to the subject matter.



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

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## ABOUT THE SOFTWARE

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The *Micromeritics-Cirrus Interface Software* is used with select Micromeritics analyzers integrated with an MKS Cirrus 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 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



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 Micromeritics-Cirrus Interface Installation Instructions for more information (P/N 292-42805-00).

## RECIPES

The interface software controls the analyzer via recipes. A recipe is a list of mass fragments, or mass-to-charge 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

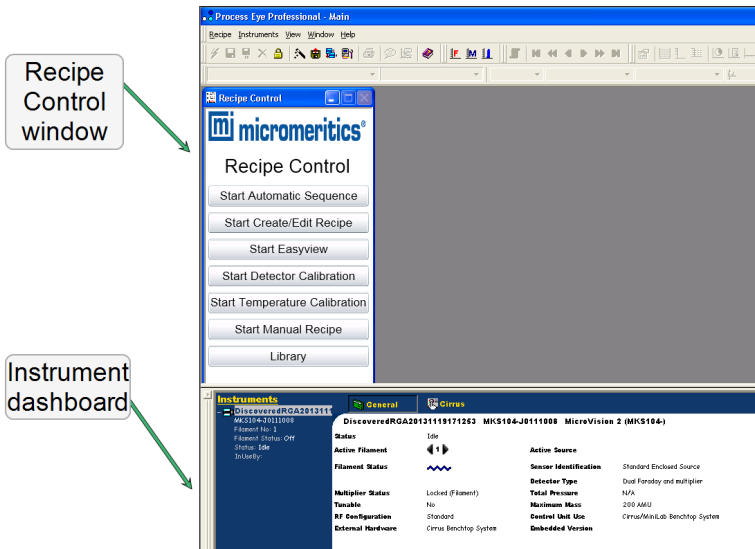
This user guide provides a description of the commands included in the *Micromeritics-Cirrus Interface Software* and instructions on how to use them. It does not include descriptions for all commands related to the Cirrus software. To access the documentation for Cirrus specific software:

1. Navigate to the *C:/Micromeritics/MKS* directory where the Micromeritics-MKS application was installed.
2. Open the *Manual* sub-directory, then double click the applicable manual.

## 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



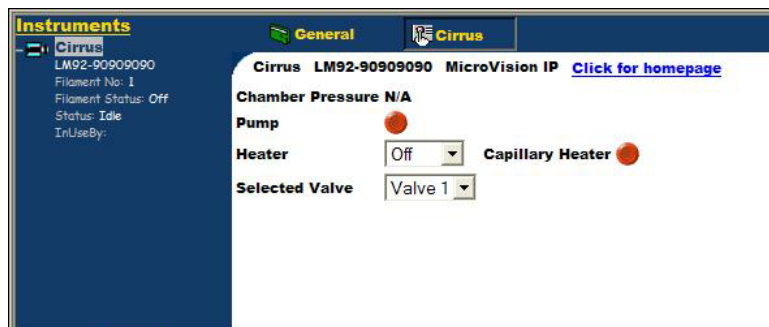


## SET UP AND PREPARE FOR OPERATION



The MKS Cirrus Mass Spectrometer pumps are powered on using the *MKS Process Eye Professional Software*. Powering on the pumps (to allow them to get to full performance) and powering on the heaters should be done before using the MKS Cirrus Mass Spectrometer.

1. Click the *Cirrus* tab on the *Status* window to access and begin component operation.



2. Use the instrument schematic to begin the flow of an inert gas (such as helium, argon, and nitrogen) to remove residual gases from the analyzer and for the next steps.
3. Click **Pump** to start the pump. The button changes to yellow to indicate that the internal molecular drag pump is accelerating. After the turbo is up to speed, the button will change to green and the chamber pressure will display.



To avoid damaging the pump, do not move the unit after the turbo pump has begun operating.

The pressure should be less than  $2\text{E}-5$  Torr before switching on a filament or the quadrupole. The trip contact on the cold cathode gauge is connected to the external trip on the RGA control unit to prevent filament operation until the pressure falls below this level. This level should be attained within 20 minutes after starting the vacuum system.

4. Use the *Warm/Bake* options in the *Heater* drop-down list. Do not operate the secondary electron multiplier (SEM) detector with the *Bake* option.



Before the system can be started properly, it will need to run a sufficient amount of time to allow the partial pressure peaks of moisture and other airborne gasses to drop to an acceptable level. This time can be significantly reduced by baking the system. This should be done after the system has run for at least an hour to allow the pressure in the system to drop below  $2\text{E-}5$  Torr. The gas flow rate should be at least 20 sccm.

A dry, inert gas should be flushed continuously through the capillary inlet during bake out. The quadrupole should be running with the filament on but only using the Faraday detector. The multiplier detector will be unavailable during baking.

The total pressure may gradually start to rise during the *Bake* cycle as the system outgases; bake the system until the pressure starts to fall. In normal operation, an initial bake of at least 24 hours is recommended, although, in general, the longer the system is baked the better. If the Cirrus is powered off, it will vent to atmosphere introducing water vapor and should be baked again.

The *Warm/Bake* option should be set to *Bake* for the initial bake-out to reduce the water background. After this period, running at the lower temperature of *Warm* is adequate in preventing the condensation of vapors in the vacuum chamber which could lead to memory effects.

Often it is not necessary to have the system heater on at all, but this will depend on the application. The Cirrus is designed to allow continuous operation with the system heater set to *Warm*.

Refer to the Cirrus 2 Hardware manual for additional information on the *Warm/Bake* option.

5. Use the *Capillary Heater* to lessen the chance of vapor condensing in the capillary leading to memory effects or blockage. Whether or not the capillary needs to be heated depends on the application and the nature of the gases being sampled. The Cirrus is designed to allow the capillary heater to be run continuously.

## ***EXIT THE INTERFACE SOFTWARE***

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

## RECIPE CONTROL

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

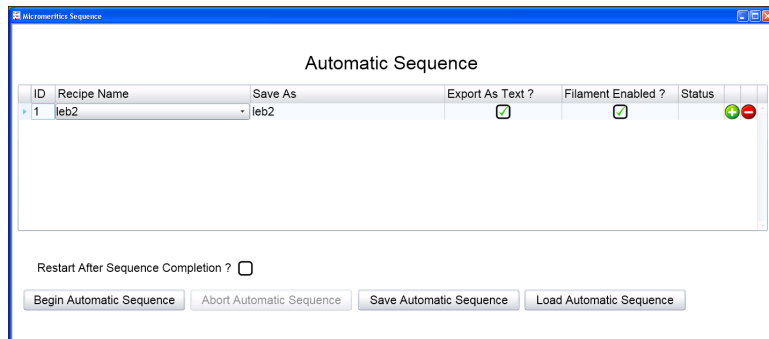


Recipe Control Window Options Table

Option	Description
<b>Start Automatic Sequence</b>	Collect data automatically via the Micromeritics analyzer digital trigger.
<b>Start Create/Edit Recipe</b>	Create <i>Peak Jump</i> recipes for the <i>Automatic Sequence</i> mode.
<b>Start Easyview</b>	Access a number of operations, including different scan modes and diagnostics.
<b>Start Detector Calibration</b>	Calibrate the Faraday and SEM detectors.
<b>Start Temperature Calibration</b>	Calculate the scaling equation for the Micromeritics analyzer sample thermocouple signal.
<b>Start Manual Recipe</b>	Start and stop recipes created using a recipe wizard.
<b>Library</b>	View mass spectral data for a variety of compounds.

## AUTOMATIC SEQUENCE

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




Use the *Automatic Sequence* window to:

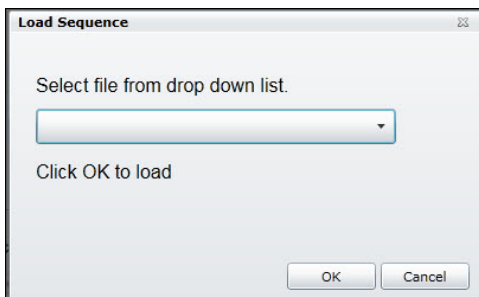

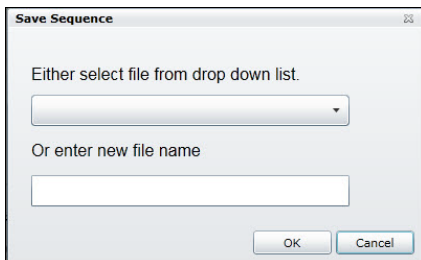
- Automatically collect data via the Micromeritics analyzer digital trigger.
- Automatically save data files, which can be imported into the Micromeritics analyzer application.
- Enable the filament for each recipe.

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.
<b>Abort Automatic Sequence</b> [button]	Stops the sequence run. Enabled when the <i>Automatic Sequence</i> has started.
<b>Begin Automatic Sequence</b> [button]	Starts the recipe.
<b>Export As Text</b> [check box]	Select for each recipe to be exported to a text file. Text files are saved to the default <i>Process Eye</i> data directory with a .TXT file extension.
<b>Filament Enabled</b> [check box]	Select to have the filament turned on during recipe initialization.  <div style="display: flex; align-items: center;">  <div style="border: 1px solid #ccc; padding: 5px; background-color: #f9f9f9;"> The filament must be turned on for data collection. </div> </div>

Automatic Sequence Options Table (continued)

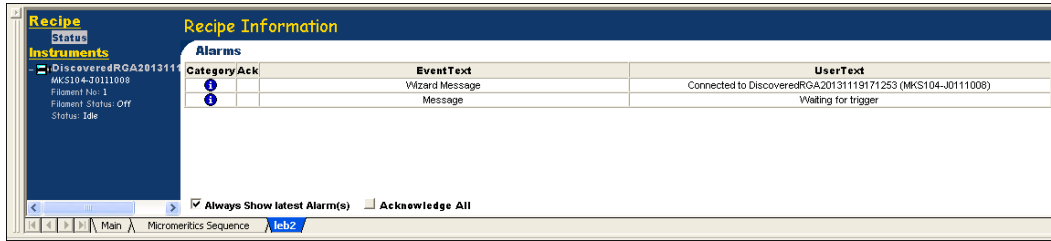
Options	Description
<b>ID</b>	Unique number given for each recipe selected.
<b>Load Automatic Sequence</b> [button]	<p>Enabled when a sequence is not running. Click to display the <i>Load Sequence</i> window to load another sequence. Click <b>OK</b> to save the options.</p> 
<b>Recipe Name</b> [selection]	Select from the list of recipes.
<b>Restart After Sequence Completion</b> [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.
<b>Save As</b> [button]	<p>Unique file name for each analysis. Enter a name for each <i>Process Eye</i> data file in this column.</p> <hr/>  <p>If a name is not specified, the data file will default to the option selected in the <i>Recipe Name</i> column. A sequential 6-digit tag is appended to each file name. For example, <i>Air</i> will be saved as <i>Air_000001.SI-d</i> if no previous versions exist. The file is saved to the default <i>Process Eye Data</i> directory.</p> <hr/>
<b>Save Automatic Sequence</b> [button]	<p>Enabled when a sequence is not running. Displays the <i>Save Sequence</i> 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 <b>OK</b> to save the options.</p> 
<b>Status</b>	Displays recipe run statuses during an automatic sequence.

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

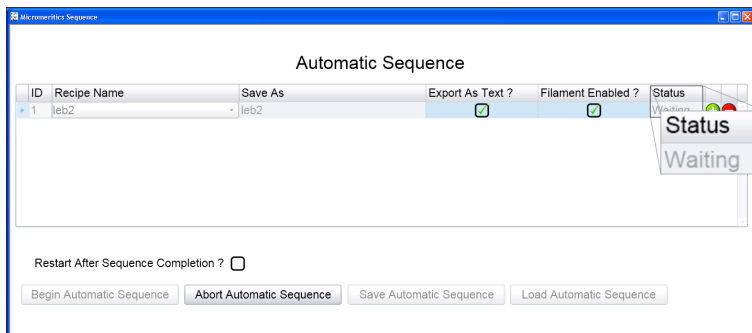
1. Select *Restart After Sequence Completion* to have the sequence restart upon completion of the last recipe in the list (if applicable).
2. Click **Begin Automatic Sequence**.



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.



## ***ENABLE THE TRIGGER FOR AUTOCHEM***

The trigger can be enabled using one of two methods:

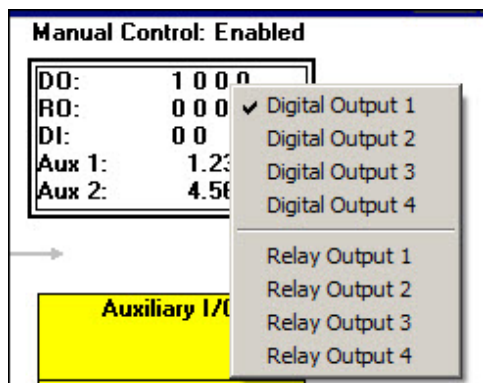
- Preset in the analysis conditions parameter file, or
- Set manually using the analyzer schematic

### ***PRESET IN THE ANALYSIS CONDITIONS PARAMETER FILE***

1. Before the *Start Recording* step, insert a *Set Outputs* step.
2. Enable digital output *D01* to be used as the trigger.
3. After the *Stop Recording* step, insert a *Set Outputs* step.
4. Disable digital output *D01*.

### ***SET MANUALLY USING THE ANALYZER SCHEMATIC***

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



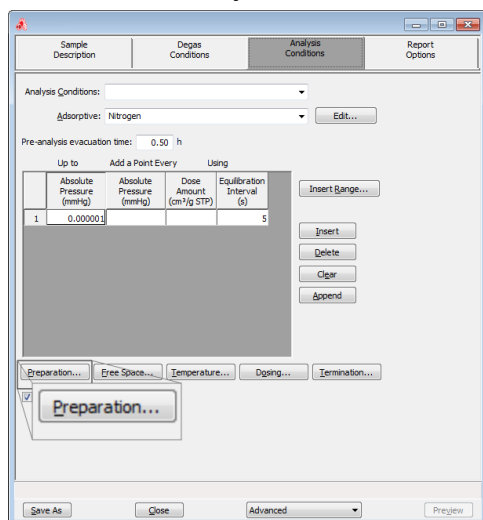
## ENABLE THE TRIGGER FOR 3FLEX 3500

The trigger can be enabled using one of two methods:

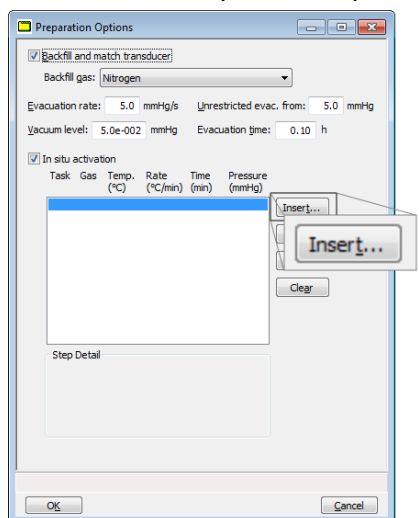
- Preset in the analysis conditions parameter file, or
- Set manually using the analyzer schematic

### PRESET IN THE ANALYSIS CONDITIONS PARAMETER FILE

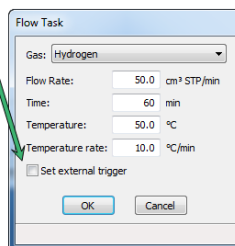
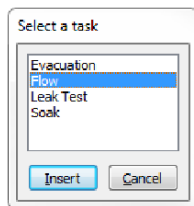
1. Go to **File > New Sample**.
2. On the *New Sample Type* window, select **Chemical Adsorption**.
3. On the *Analysis Conditions* tab, click **Preparation**.



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



Enable  
Set external trigger





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**.

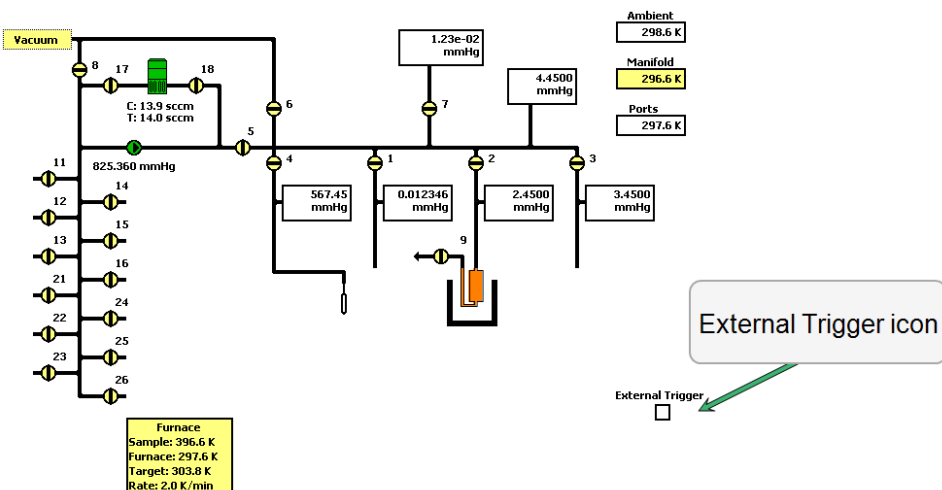


Minimum recommended flow rate is 50 sccm.

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 USING THE ANALYZER SCHEMATIC

1. Go to **Unit [n] > Chemical Adsorption** (if not already selected).
2. Go to **Unit [n] > Enable Manual Control**. Ensure a checkmark displays to the left of the menu item. If the instrument schematic does not display, go to **Unit [n] > Show Instrument Schematic**.
3. Right click the *External Trigger* icon, then select *On*. Alternately, double click the *External Trigger* icon.



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

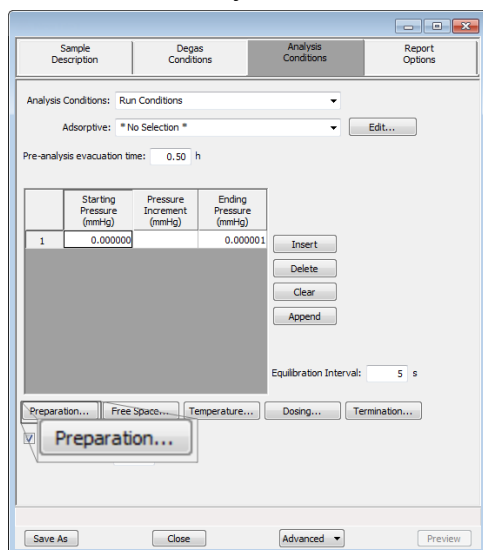
## ENABLE THE TRIGGER FOR ASAP 2020 PLUS

The trigger can be enabled using one of two methods:

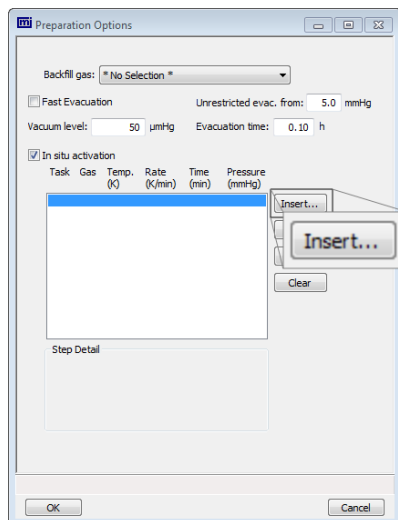
- Preset in the analysis conditions parameter file, or
- Set manually using the analyzer schematic

### PRESET IN THE ANALYSIS CONDITIONS PARAMETER FILE

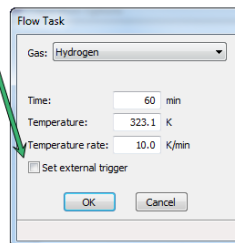
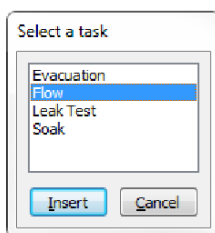
1. Go to **File > New Sample**.
2. On the **Analysis Conditions** tab, click **Preparation**.



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



Enable  
Set external trigger



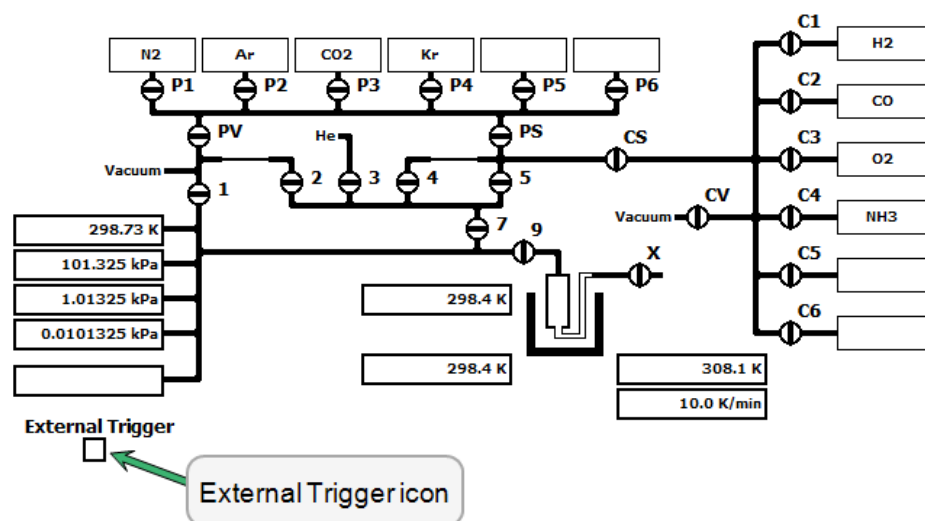
4. On the *Select a task* window, select *Flow*, then click **Insert**.
5. On the *Flow Task* window, ensure *Set external trigger* is selected, then click **OK**.



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.
1. Go to **Unit [n] > Enable Manual Control**. Ensure a checkmark displays to the left of the menu item. If the instrument schematic does not display, go to **Unit [n] > Show Instrument Schematic**.
2. Right click the *External Trigger* icon, then select *On*. Alternately, double click the *External Trigger* icon.

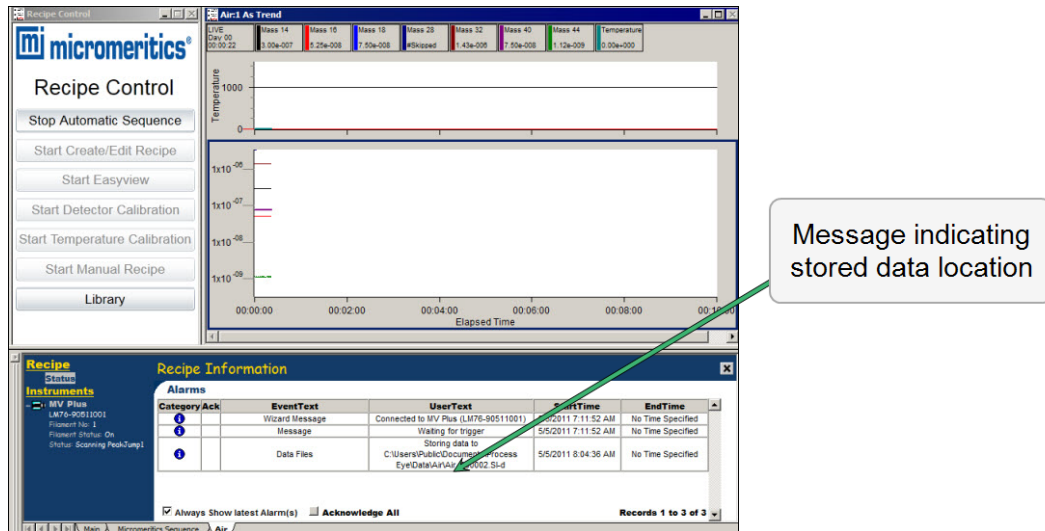
## SET MANUALLY USING THE ANALYZER SCHEMATIC



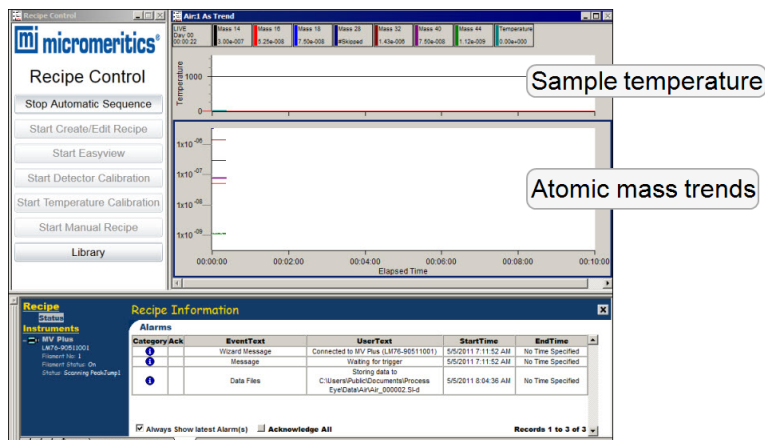
The *External Trigger* icon changes to a solid black box to indicate the external trigger is enabled.

## 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.






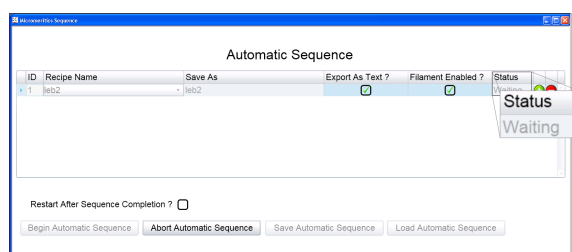
Two windows are opened, one with atomic mass trends and one with the scaled analyzer sample temperature, where mass spectra parameters and temperature axes can be edited. See [Recipe Control on page 5](#).



## 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.

Automatic Sequence				
	Export As Text ?	Filament Enabled ?	Status	
	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	Completed	
	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	Waiting	
	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>		

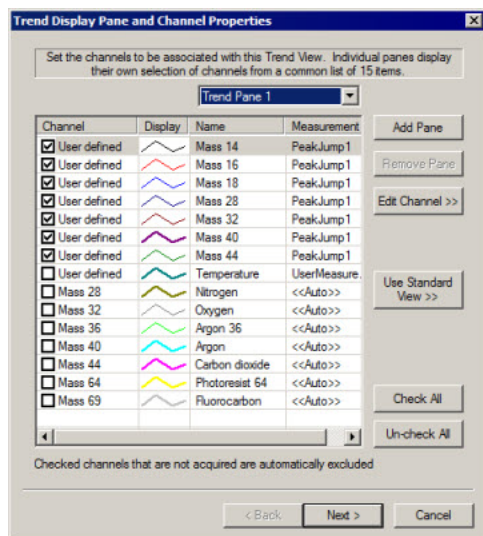


On the *Automatic Sequence* window, the *Status* column shows *Completed* for the completed recipe. If there is more than one recipe in the sequence list, the next recipe in the sequence initializes and *Waiting* displays. Once the digital trigger is enabled for the recipe with a *Waiting* status, the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

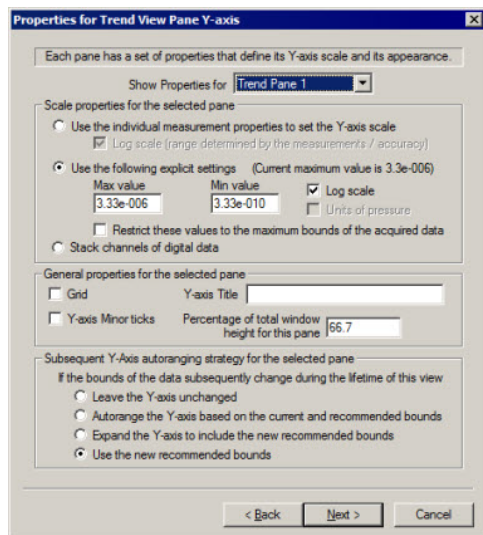
## AXES AND LEGEND PROPERTIES

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](#).

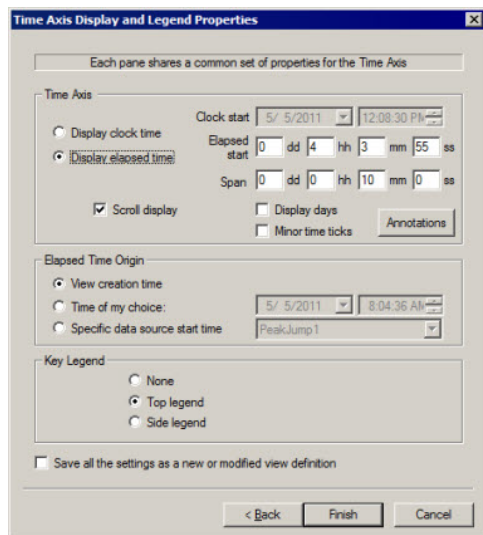


5. On the *Properties for Trend View Pane Y-axis* window, edit the values in the *Max value* and *Min value* fields.



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**.



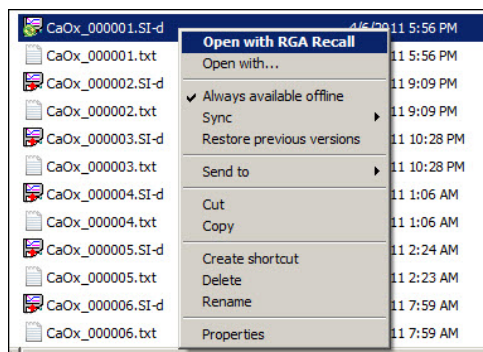
The screenshot shows the 'Time Axis Display and Legend Properties' dialog box. It has a title bar with a close button. Below the title bar is a subtitle 'Each pane shares a common set of properties for the Time Axis'. The dialog is divided into several sections: 'Time Axis' with radio buttons for 'Display clock time' and 'Display elapsed time' (selected), and checkboxes for 'Scroll display', 'Display days', and 'Minor time ticks'; 'Elapsed Time Origin' with radio buttons for 'View creation time', 'Time of my choice', and 'Specific data source start time'; and 'Key Legend' with radio buttons for 'None', 'Top legend' (selected), and 'Side legend'. At the bottom, there is a checkbox 'Save all the settings as a new or modified view definition' and three buttons: '< Back', 'Finish', and 'Cancel'.

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.

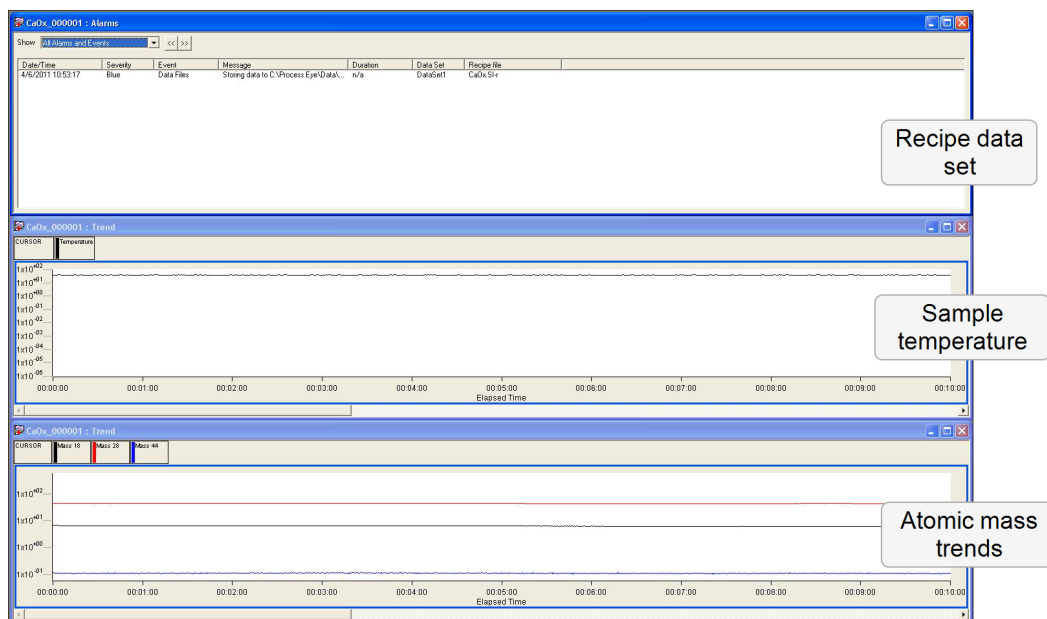
## DATA AND RGA RECALL

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.

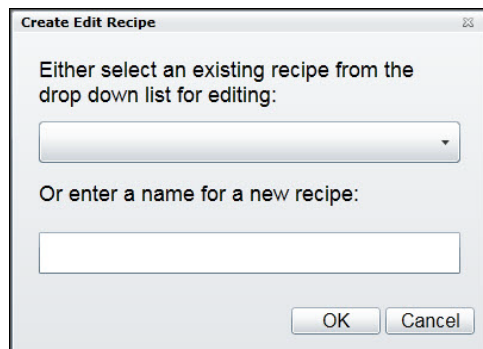


If the data file being opened is currently running, the file will not open. A prompt displays indicating either to open a copy of the file or wait until the file is inactive before trying *RGA Recall* again.



## 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.



Either select an existing recipe from the drop down list for editing:

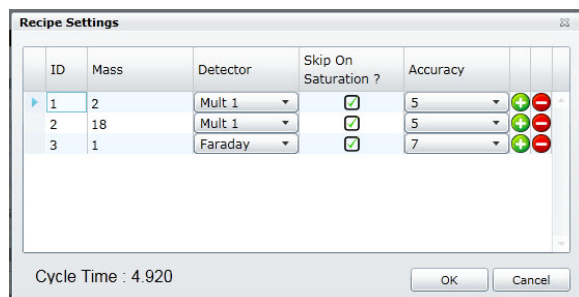
Or enter a name for a new recipe:

OK Cancel



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**.



ID	Mass	Detector	Skip On Saturation ?	Accuracy		
1	2	Mult 1	<input checked="" type="checkbox"/>	5	+	-
2	18	Mult 1	<input checked="" type="checkbox"/>	5	+	-
3	1	Faraday	<input checked="" type="checkbox"/>	7	+	-

Cycle Time : 4.920

OK Cancel

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. **NOTE:** See [Library on page 33](#) 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).



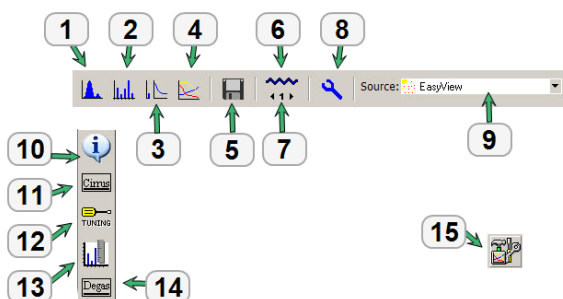
For recipes where a short cycle time is crucial, select the same accuracy level for all masses.

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

## 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 <b>Peak Settings</b> button to the <i>EasyView</i> window. See legend item 15 for <b>Peak Settings</b> 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 <i>General Information</i> page.
11	Displays the status of the turbo pump, manifold heater, and capillary heater.
12	Activates <i>Tuning</i> mode. Use to adjust the tuning of the RGA (peak alignment and resolution).

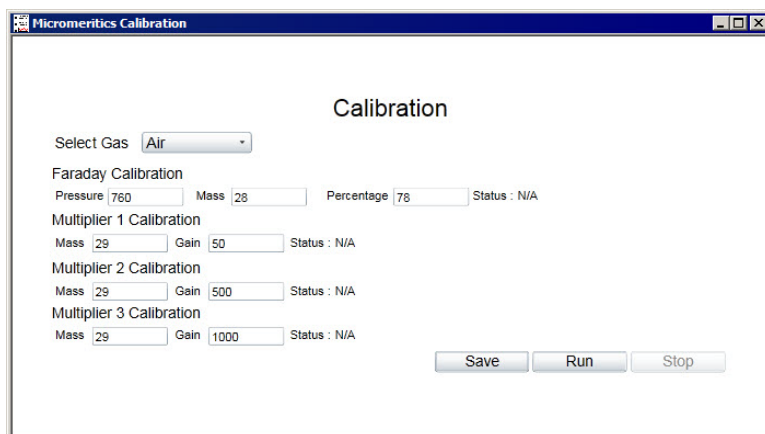
## EasyView Functions Table (continued)

Legend	Description
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 <a href="#">Start Detector Calibration</a> on the <i>Recipe Control</i> pane for this calibration.
14	Opens the <i>Degas Settings</i> page for degassing the ion source of selected MKS RGAs.
15	Visible when <i>Peak Jump</i> mode is activated. Use to edit recipe parameters.

## 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 next table for each calibration entered:

**Calibration Instructions Table**

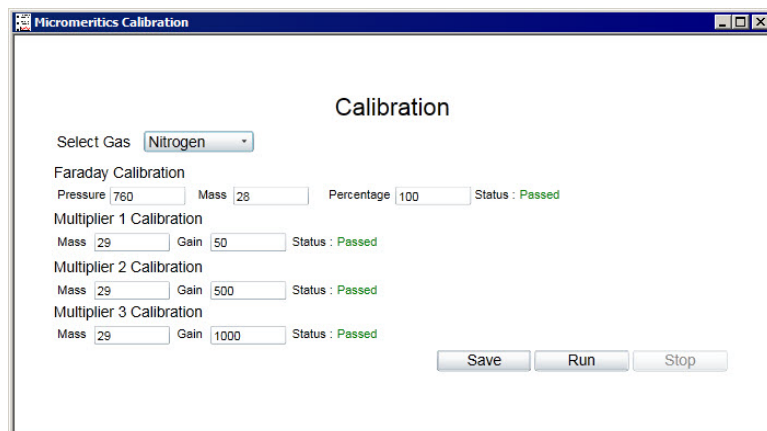
Type	Instructions
<b>Faraday</b>	<ul style="list-style-type: none"> <li>• Enter the inlet pressure (in Torr) in the <i>Pressure</i> field. The typical atmospheric pressure entry of 760 is used.</li> <li>• Enter the mass in the <i>Mass</i> field. Typically, this is the mass of dominant species in the 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.</li> <li>• In the <i>Percentage</i> 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).</li> </ul>
<b>Multiplier</b>	<ul style="list-style-type: none"> <li>• 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).</li> <li>• Use the same mass for all three multiplier detectors.</li> <li>• The values in the <i>Gain</i> fields are appropriate in most cases and should remain unchanged.</li> </ul>

## BEGIN CALIBRATION

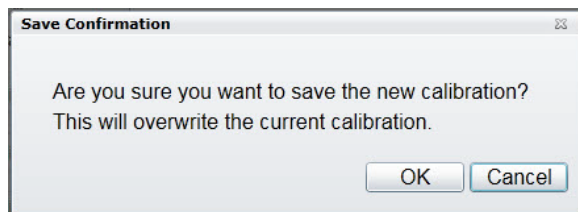
1. Click **Run** to begin calibration. The Faraday calibration and SEM calibrations will run sequentially.



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



2. Click **Save**.

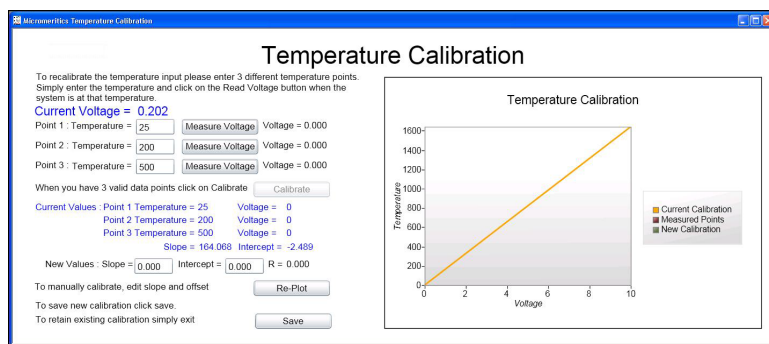


3. On the *Save Confirmation* window, click **OK** to overwrite the current calibration and close the window.
4. Click **Stop Detector Calibration** to close the *Calibration* window.

## 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 (°C).



The 3Flex 3500 Chemisorption and ASAP 2020 Plus analyzers only use a single Digital I/O interface cable to connect to the Mass Spectrometer.

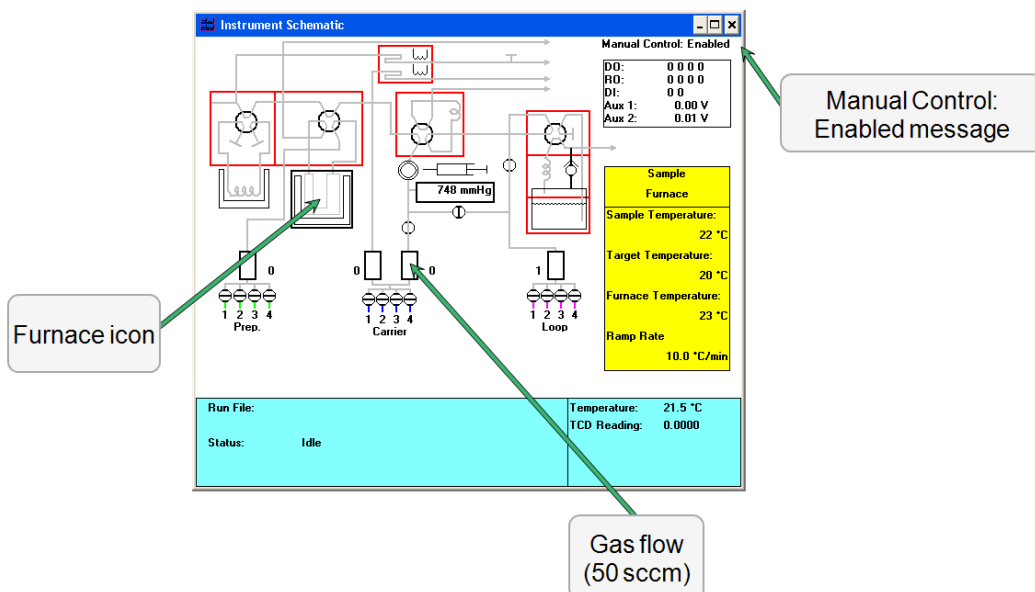


## AUTOCHEM ANALYZERS

1. Ensure that the AutoChem analyzer furnace is closed and a sample tube is installed.
2. Start the AutoChem analyzer application.
3. Open the instrument schematic, then enable manual control.
4. Ensure gas is flowing at a rate similar to experimental conditions.

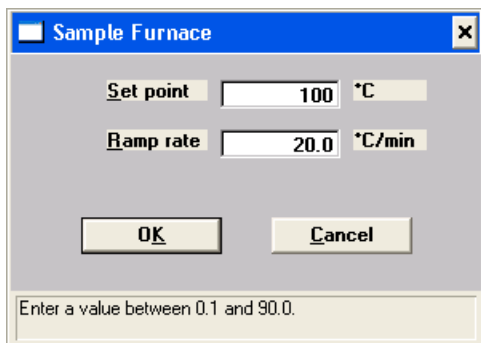


The instrument schematic illustrated here may differ slightly from yours; however, the process is the same.



## POINT 1: TEMPERATURE CALIBRATION

1. Right click the furnace icon on the AutoChem instrument 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.

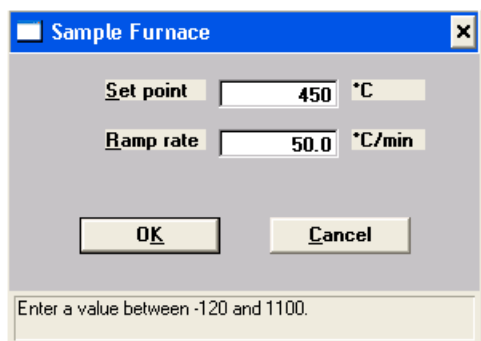


The **Sample Furnace** dialog box is shown. It has a title bar with a close button (X). Inside, there are two input fields: **Set point** with the value 100 and a unit of °C, and **Ramp rate** with the value 20.0 and a unit of °C/min. Below these are **OK** and **Cancel** buttons. At the bottom, a message box says "Enter a value between 0.1 and 90.0."

3. Click **OK**.
4. Observe the *Sample Temperature* reading on the AutoChem instrument schematic. When the sample temperature reaches approximately 100 °C 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 AutoChem instrument 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.

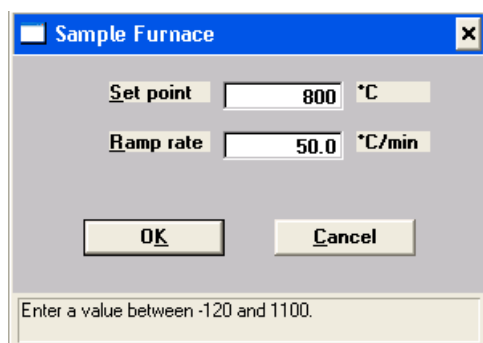


The **Sample Furnace** dialog box is shown. It has a title bar with a close button (X). Inside, there are two input fields: **Set point** with the value 450 and a unit of °C, and **Ramp rate** with the value 50.0 and a unit of °C/min. Below these are **OK** and **Cancel** buttons. At the bottom, a message box says "Enter a value between -120 and 1100."

3. Click **OK**.
4. Observe the *Sample Temperature* reading on the AutoChem instrument schematic. When the sample temperature reaches approximately 450 °C 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 AutoChem 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 AutoChem instrument 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 AutoChem instrument schematic. When the sample temperature reaches approximately 800 °C 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 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.

**Sample Furnace**

Set point  °C

Ramp rate  °C/min

Enter a value between -120 and 1100.

- Click **OK**.

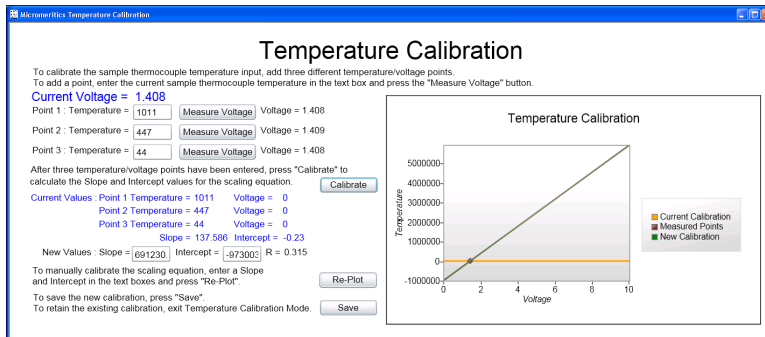


Attaching the Kwikcool or Cryocooler will speed up this process.

## 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.

- 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.



- Click **Save**.
- On the *Save Confirmation* window, click **OK** to overwrite the current calibration and close the window.
- 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.

New Values : Slope =  Intercept =  R = 0.364

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.

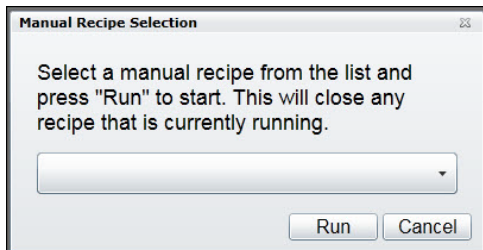
## 3FLEX AND ASAP 2020 PLUS CHEMISORPTION ANALYZERS

The 3Flex 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.

## MANUAL RECIPE

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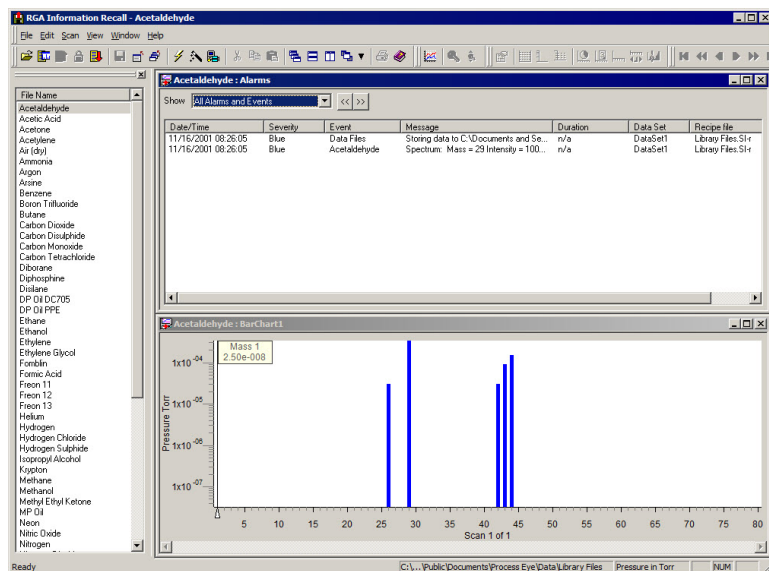
The *Start Manual Recipe* option provides access to recipes created using the *Recipe Wizard*. For detailed instructions on using the *Recipe Wizard*, refer to the *Recipe Wizard User Guide* included with the Cirrus II documentation.



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.

## LIBRARY

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



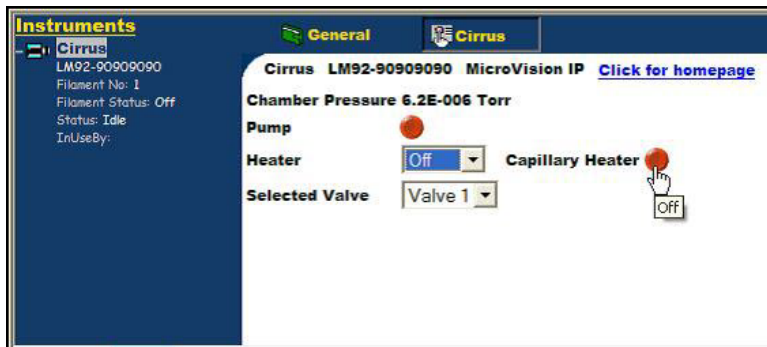
1. In the *File Name* list, double click the applicable compound to display mass spectral data.
2. Click the **X** in the upper right corner to close the window.

## POWER OFF THE MASS SPECTROMETER

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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.