

Thermocouple Calibration for the AutoChem – Thermostar Interface

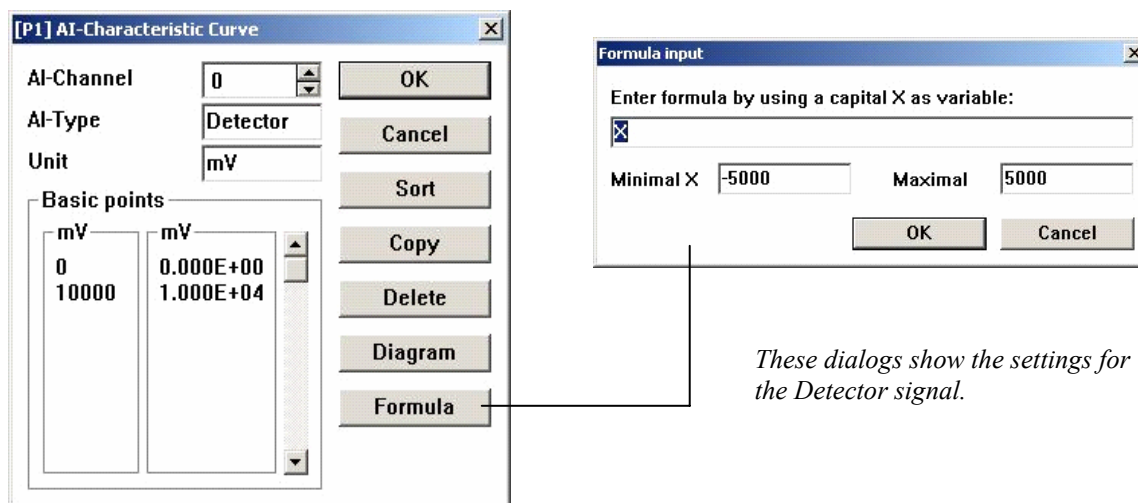
Micromeritics' AutoChem series of instruments is equipped with both digital and analog I/O (inputs/outputs), which may be used to control a Pfeiffer Vacuum Thermostar mass spectrometer via a single interface (*Micromeritics' part number 291/60811/00*). This cable has been specifically designed to provide the hardware interface between the two instruments. The single-connector cable end is attached to the mass spectrometer*. The double-connector cable end (consisting of small and large connectors) is connected to the AutoChem. The small connector is attached to the Analog I/O and the large connector attached to the Digital I/O.

The Quadstar software package from Pfeiffer Vacuum consists of several applications which are primarily used for the configuration of the Thermostar mass spectrometer and collection of mass spectrometer data. Pfeiffer Vacuum has also developed the TG-MS software macros for use with the Quadstar software. This suite of macros used with the Quadstar software and Micromeritics' cable interface allows AutoChem users full control of their thermostar mass spectrometer.

Several parameters must be established to utilize the Quadstar software to the fullest. A simple thermocouple calibration is the most significant

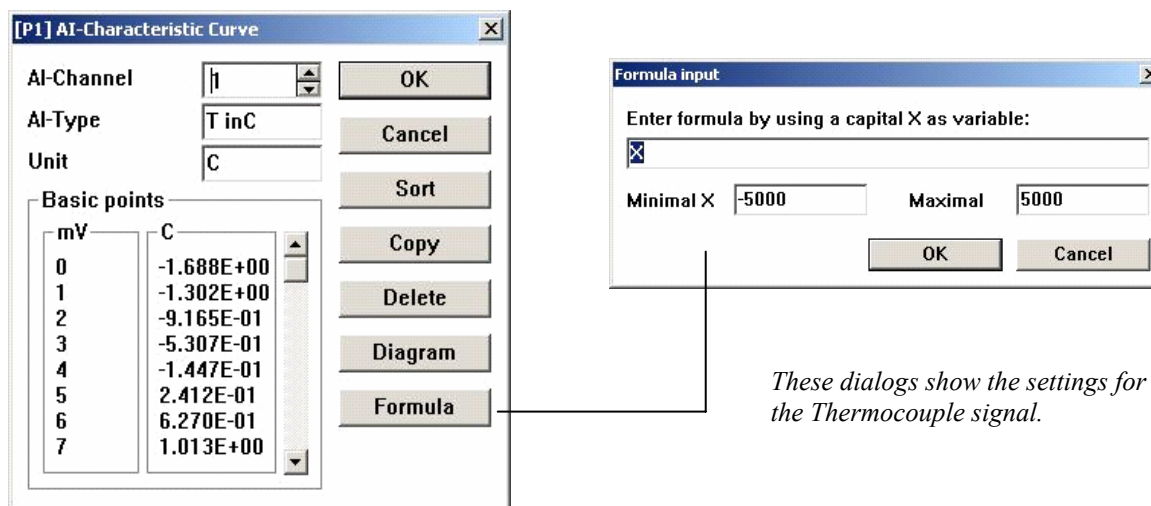
1. Launch the Quadstar *Parset* program; use one of the following methods:
 - Select **Start > Programs > Quadstar > Parset**
 - Double-click the Quadstar icon on the Windows desktop, then double-click the **Parset** icon.
2. Select **Config > AI Characteristic Curve**; the AI Characteristic Curve dialog is displayed. Use this dialog to define parameters for the Detector and Thermocouple signals.
3. Configure the Detector signal as follows:
 - a. Click the up/down arrow in the **AI-Channel** field until **0** is displayed.
 - b. Enter **Detector** in the **AI-Type** field.
 - c. Enter **mV** in the **Unit** field.
 - d. Click **Formula**; enter **X** as the formula, **-5000** in the **Minimal X** field and **5000** in the **Maximal** field.

*The cover must be removed from the Thermostar to make this connection; refer to the Thermostar manual for instructions on removing the cover.



These dialogs show the settings for the Detector signal.

4. Configure the Thermocouple signal:
 - a. Click the up/down arrow in the **AI-Channel** field until **1** is displayed. All parameters defined for **0** (Detector) are saved automatically when **1** displays.
 - b. Enter **T inC** in the **AI-Type** field. Be sure to space between **T** and **inC**; otherwise, the AutoChem software cannot properly identify the temperature signals.
 - c. Enter **C** in the **Unit** field.
 - d. Click **Formula**; enter **X** as the formula, **-5000** in the **Minimal X** field and **5000** in the **Maximal** field.

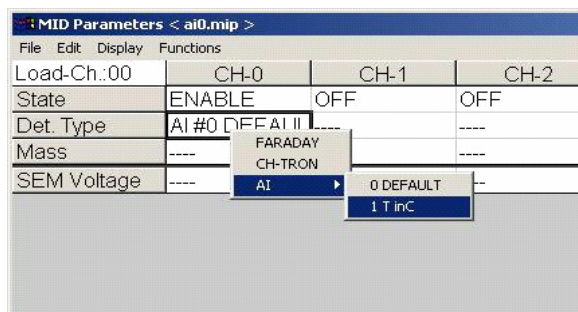


These dialogs show the settings for the Thermocouple signal.

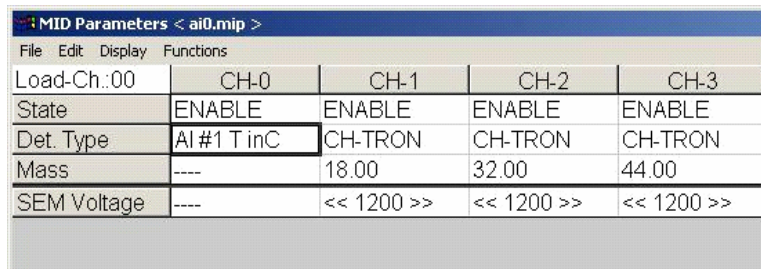
5. Click **OK** to close the AI-Characteristic Curve dialog.

With these parameters set, you can now develop a thermocouple calibration.

1. Select **Measure > MID** from the main menu of the **Parset** application; the MID Parameters dialog is displayed. This dialog is used to specify the signals to be recorded by the Thermostar.
2. Choose parameters for the calibration:
 - a. Right-click in the **State > CH-0** field; select **Enable** from the pop-up menu.
 - b. Right-click in the **Det. Type > CH-0** field; select **AI > T inC**.



3. Specify mass signals (required for the Thermostar to collect data); an example is given here. Save this file as **mstcal.mip**. Refer to your Thermostar instruction manual if assistance is needed for this step.

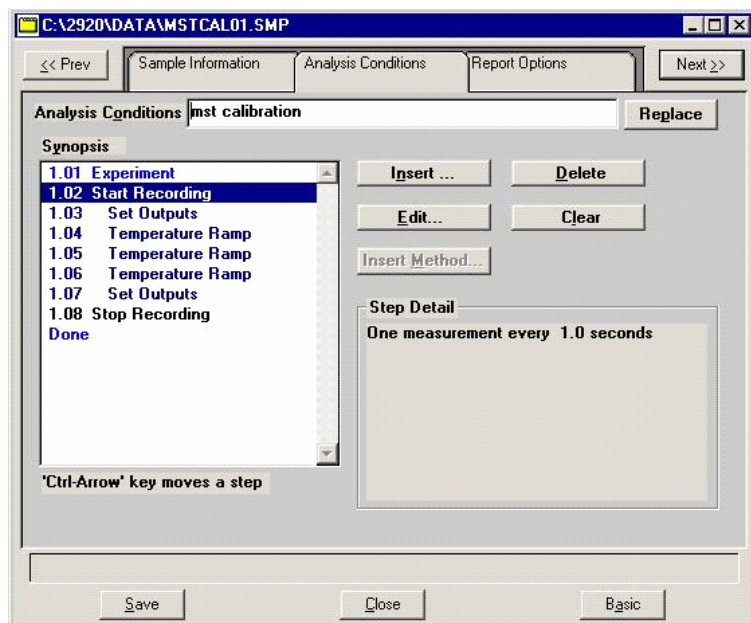


4. Create a sample file (AutoChem software) for the thermocouple calibration; in this example we use **mstcal01.smp**. On the Analysis Conditions dialog, define the parameters as follows:

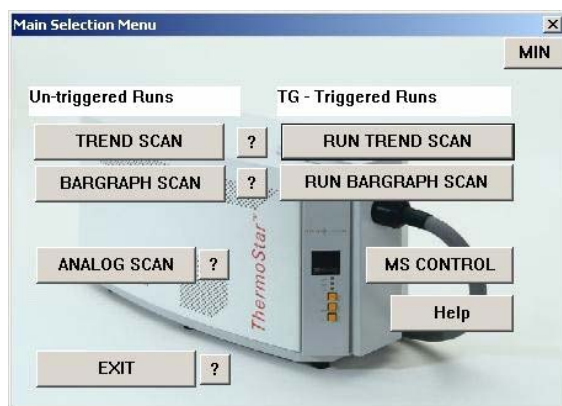
Insert	Action
Experiment	Click Gas Flows ; choose Helium as Carrier gas at 10 mL/min .
Start Recording	One measurement every 1.0 second
Set Outputs	Select Enable DO 1 (this relay is used as a trigger that allows you to start and end data collection on the Thermostar)
Temperature Ramp	End Temperature: 100 °C Ramp Rate: 25 °C/min Hold time: 10 min
Temperature Ramp	End Temperature: 300 °C Ramp Rate: 25 °C/min Hold time: 10 min
Insert	Action

Temperature Ramp	End Temperature: 500 °C Ramp Rate: 25 °C/min Hold time: 10 min
Set Outputs	Disable DO 1 .
Stop Recording	Automatically inserted when Recording step created.
Done	Permanent step; no editing required in this example

The synopsis in your sample file should look similar to the following:



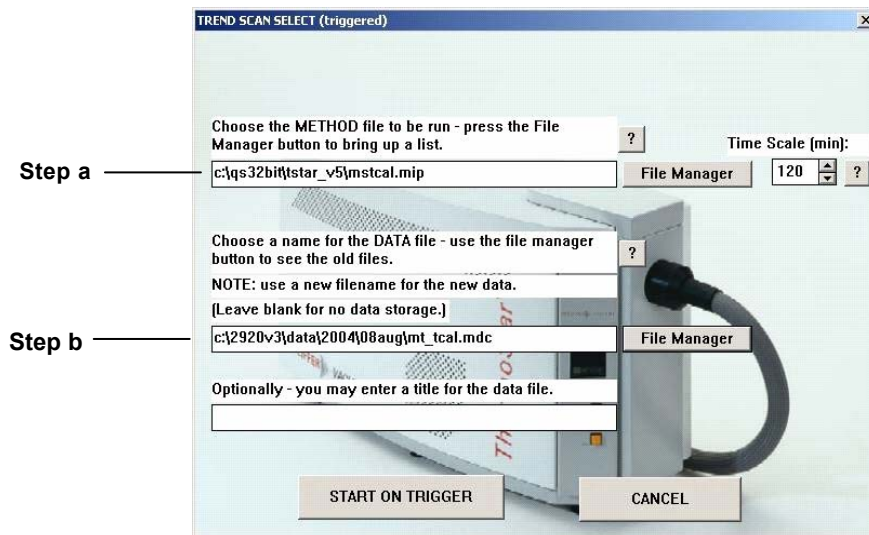
1. Click the **TG-MS V5** icon (on your desktop) to start the program



Untriggered Runs are for manual operation and **TG-Triggered Runs** are for operation using the AutoChem.

2. Click **RUN TREND SCAN** to display the Trend Scan Select (triggered) dialog.
 - a. Click **File Manager** (upper one) and choose **mstcal.mip** for the method to run.

- b. Click **File Manager** (lower one); enter a file name for the calibration data, this example uses **mt_tcal.mdc**.
- c. Click **START ON TRIGGER** to close the dialog; the Thermostar is now being controlled by the AutoChem software.



3. From the AutoChem software, select **Unit 1 > Start Analysis**; choose **mstcal01.smp** as the sample file.
4. Click **Next** to start the analysis.

Data will be recorded by both the AutoChem and the Thermostar. Example data are shown in Figures 1 and 2.

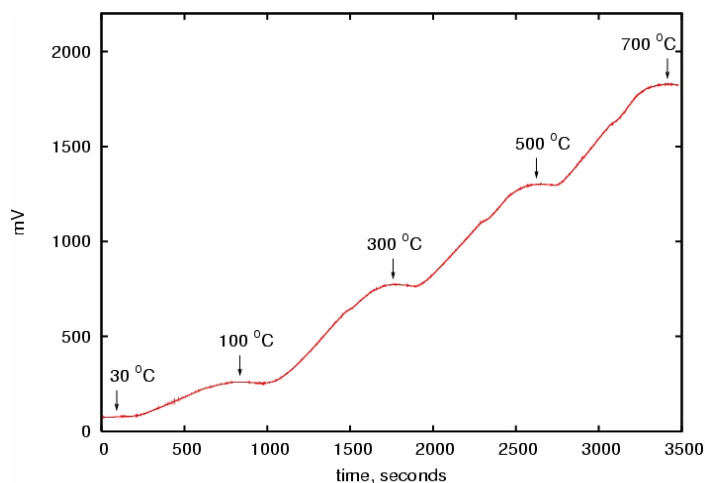


Figure 1. Typical mV versus time collected using the Thermostar.

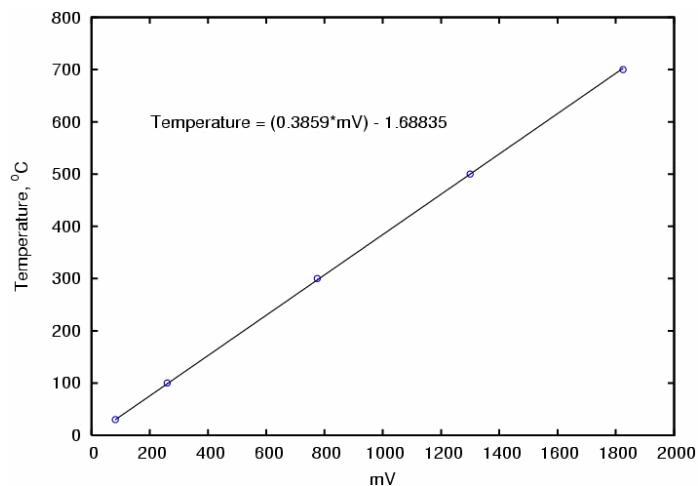


Figure 2. Regression results from AutoChem temperature versus AI-1 mV data collected using the Thermostar.

The Thermostar signal versus AutoChem temperature plot (Figure 2) yields a linear relationship. To update the **AI-1 Characteristic** curve:

1. Select **Config > AI Characteristic Curve** from the main menu of the **Parset** application.
2. Click the up/down arrow in the **AI-Channel** field until **1** is displayed.
3. Enter the value displayed for the temperature.

Formula input

Enter formula by using a capital X as variable:

Minimal X Maximal

Future analyses will now contain a temperature signal in addition to any mass signals that are recorded.