

## 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.



One Micromeritics Drive, Norcross, Georgia 30093 T. (770) 662-3620 www.micromeritics.com

[P1] AI-Characteristic Curve
Al-Channel 0 ✓ OK   Al-Type Detector Cancel Enter formula by using a capital X as variable:   Unit mV Sort Minimal X 5000   Basic points OK Cancel Minimal X 5000   MU mV O OK Cancel   Diagram These dialogs show the settings for the Detector signal. 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** in**C** in the **AI-Type** field. Be sure to space between **T** and in**C**; 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.

[P1] AI-Characteristic Curve	×	
Al-Channel 👔 🚔	ОК	Formula input
Al-Type T inC	Cancel	Enter formula by using a capital X as variable:
Unit C	Sort	Minimal X -5000 Maximal 5000
mV C	Сору	OK Cancel
1 -1.302E+00 2 -9.165E-01	Delete	
3 -5.307E-01 4 -1.447E-01	Diagram	These dialogs show the settings
5 2.412E-01 6 6.270E-01	Formula	the Thermocouple signal.
7 1.013E+00		

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 **A1** > **T** in**C**.

MID Parameters	i < ai0.mip >			
File Edit Display I	Functions			
Load-Ch.:00	CH-0	)	CH-1	CH-2
State	ENABLE	OF	E)	OFF
Det. Type	AI #0 DEP	AL II	1	
Mass	F			
SEM Voltage	4		0 DEFAULT	
	_		1 T inC	
		<u>,</u>		_

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.

Mit MID Parameters < ai0.mip >					
File Edit Display	Functions				
Load-Ch.:00	CH-0	CH-1	CH-2	CH-3	
State	ENABLE	ENABLE	ENABLE	ENABLE	
Det. Type	AI #1 T inC	CH-TRON	CH-TRON	CH-TRON	
Mass		18.00	32.00	44.00	
SEM Voltage		<< 1200 >>	<< 1200 >>	<< 1200 >>	

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 <b>Enable DO 1</b> (this relay is used as a trigger that allows you to start and end data collection on the Thermostar)		
Temperature Ramp	<b>End Temperature</b> : 100 °C <b>Ramp Rate</b> : 25 °C/min <b>Hold time</b> : 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 <b>DO 1.</b>
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:

Analysis C <u>o</u> Synonsis	nditions mst calibrat	ion			Replace
1.01 Exp 1.02 Star 1.03 S 1.04 T 1.05 T 1.06 T 1.07 S 1.08 Stop Done	eriment It Recording et Outputs emperature Ramp emperature Ramp emperature Ramp et Outputs p Recording	4	Insert Edit Insert Metho Step Detai One measu	 Delete Cjear very 1.0 seco	nds
'Ctrl-Arrow	' key moves a step				

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.

	TREND SCAN SELECT (triggered)	×
	Choose the METHOD file to be run - press the File Manager button to bring up a list.	? Time Scale (min):
Step a ——	c:\qs32bit\tstar_v5\mstcal.mip	File Manager 120 🔺 ?
	Choose a name for the DATA file - use the file manager button to see the old files. NOTE: use a new filename for the new data. [Leave blank for no data storage.]	2
Step b	c:\2920v3\data\2004\08aug\mt_tcal.mdc	File Manager
	Optionally - you may enter a title for the data file.	CANCEL

- 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 varia	ble:
Minimal X 0	Maximal	5000
	ОК	Cancel

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