

In This Issue

Micromeritics Announces New Laboratory

New business unit offers laboratory services..... 1

TPR, TPO and TPD Examination of Cu_{0.15}Ce_{0.85} O_{2-y} Mixed Oxide Catalyst Prepared by Co-precipitation Synthesis

This study utilizes the AutoChem II 3

Surface, Colloids, and Nanoscience short course

..... 2

Thermocouple Calibration for The AutoChem-Thermostar Interface..... 8

Micromeritics Provides More Than Instruments

Micromeritics Sales Engineers help to solve problems 7

Also...

Training Courses..... 11

Events..... 11



www.micromeritics.com

Micromeritics Analytical Services - The Particle Testing Authority

Micromeritics is pleased to announce the formation of a new business unit, Micromeritics Analytical Services. Micromeritics has always supported a Material Analysis Lab where customer samples were analyzed on a contract basis. Now, to provide our customers with improved support and superior service, we have formed the new business unit that will have its own image and direction for the future.



Several key changes have already taken place and more are planned. We have introduced a brand new web site (www.particletesting.com). We have also completed updating laboratory policies and procedures to comply with CGMP guidelines, and we are now a registered laboratory with the U.S. Food and Drug Administration.

Micromeritics Analytical Services has also applied for a U.S. Drug Enforcement Agency license to perform laboratory tests on controlled substances.

Our lab is backed by a highly qualified staff of scientists, analysts, and engineers who are committed to providing superior service and support before, during, and after sample analysis. Senior research and application scientists with extensive experience in their respective areas are available to discuss applications and provide useful data interpretation. Regional sales engineers are available around the world with technical knowledge and experience. Mechanical, software, and electrical engineers are available to assist in instrument or software customization for unique sample analyses. Service personnel are on-site ensuring

that analytical instruments are at optimum performance so there is no delay in sample turnaround.

Micromeritics Analytical Services operates a state-of-the-art laboratory, equipped with the most current instruments available. The laboratory has access to new technology and new Micromeritics instruments as soon as they become available. In today's market, major advances in instrument technology occur frequently. If the lab you currently use does not purchase new instrumentation every few years, you may not be receiving the most complete analysis possible.

Micromeritics Analytical Services offers rapid sample turnaround with the highest quality results. Most samples are completed within 5-10 days. We pride ourselves on providing results that are of the highest quality. All results undergo a thorough review process. This means laboratory management, senior scientists, and product specialists must approve the results before they are released.

For more information or to submit a sample for analysis, visit the recently introduced Micromeritics **Analytical Services** website at www.particletesting.com or call 770-662-3630.

New Online Catalog for Micromeritics Products

Micromeritics has recently redesigned and updated its online product catalog. This catalog allows you to request a quotation for our instruments, replacement parts, manuals, software, and accessories. Graphics help you identify the parts you are looking for. You may browse through the catalog, select the items you wish to purchase, and submit the quote request form. You will receive an email that includes the cost of your order as well as purchasing instructions.

You can access the catalog at www.micromeritics.com/catalog



Surfaces, Colloids, and Nanoscience

The University of Washington – Seattle is offering
a 5-day intensive course
July 11 – 15, 2005

The science underlying the present and emerging technologies in Surfaces, Colloids, and Nanoscience is presented, and the consequences are traced to applied research and practice. Areas impacted include fluid-solid systems handling (coating, spinning, spraying, foaming, wicking, printing, etc.), drug development and delivery, food technology, imaging technology, and much more. The most up-to-date techniques and experimen-

tal instrumentation for property measurement and system characterization are discussed and demonstrated.

More information and forms for online registration (or pre-registration to hold a place) are available at:

http://faculty.washington.edu/spc/Short_Course.html

TPR, TPO and TPD Examination of $\text{Cu}_{0.15}\text{Ce}_{0.85}\text{O}_{2-y}$ Mixed Oxide Catalyst Prepared by Co-precipitation Synthesis

Albin Pintar*, Jurka Batista, Stanko Hoever

Laboratory for Catalysis and Chemical Reaction Engineering, National Institute of Chemistry, Hajdrihova 19, P.O. Box 660, SI-1001 Ljubljana, Slovenia

This is a condensed article submitted to Micromeritics. The full version of this article can be found on our website at www.micromeritics.com/applications/articles.aspx

Introduction

The use of CeO_2 and CeO_2 -containing materials as a component of heterogeneous industrial catalysts or as a support for transition metals is based on superior chemical and physical stability, high oxygen mobility, and high oxygen vacancy concentrations, which are characteristic for the fluorite-type oxides (economically and technologically most important application is the use of ceria in the three-way automotive exhaust catalysts as a thermal stabilizer and oxygen storage medium [1]). In this work, we report on the characterization of a $\text{Cu}_{0.15}\text{Ce}_{0.85}\text{O}_{2-y}$ mixed oxide catalyst (prepared by a co-precipitation method and will be referred to as CuCe-1) TPR, TPO, and TPD. These techniques will be demonstrated as an efficient set of tools to obtain information about the redox behavior of this solid. This information is important for designing CuO-CeO_2 catalysts that can replace the expensive noble metal catalysts in a number of down-stream processes, including the production of H_2 -rich gas streams from fossil and renewable fuels for use as a fuel for the proton exchange membrane fuel cells (PEMFC).

BET, TPR, TPO, and TPD Measurements

Single-point BET surface area, temperature-programmed reduction (TPR) with hydrogen, temperature-programmed oxidation (TPO) with oxygen, temperature-programmed desorption (TPD) of hydrogen, temperature-programmed desorption of oxygen, and hydrogen pulse chemisorption measurements of CuCe-1 sample were performed by means of an automated catalyst characterization system (Micromeritics' AutoChem II 2920), which incorporates a thermal conductivity detector (TCD).

The TPR measurements were carried out following activation and after cooling the sample in helium flow to 0 °C. Then the TPR experiments were performed up to a temperature 400 °C at which the sample was maintained for 30 min. In order to verify that mass transfer limitations do not affect the TPR measurements, we have carried out TPR experiments at different sample loadings (*i.e.*, 0.10 and 0.25 g).

The TPO experiments were performed following TPR after cooling the samples in H_2 (5 vol. %)/Ar flow to 0 °C. After that O_2 (10 vol. %)/He gas mixture

was passed over the samples which were heated to 400 °C and then held at 400 °C for 1 h. To examine the reproducibility of TPR profiles after the reoxidation, the sample was cooled in helium flow to 0 °C at the end of the TPR-1/TPO cycle and then the second reduction run (TPR-2) was carried out under the same reaction conditions as the first run.

The TPD- H_2 was performed following the TPR experiments after cooling the reduced sample in H_2 (5 vol. %)/Ar gas mixture flow to -20 °C. After that the TPD- H_2 experiment was carried out up to a temperature of 650 °C, at which the sample was held for 30 min.

The TPD- O_2 was carried out on the activated sample, which was cooled in O_2 (10 vol. %)/He gas mixture flow to -20 °C. The TPD- O_2 measurement was performed up to a temperature of 400 °C, at which the sample was held for 6 h. The hydrogen pulse chemisorption measurements of CuCe-1 sample were applied after TPD- O_2 measurements to find out whether a chemisorption of hydrogen occurred at temperature close to 0 °C (*i.e.*, during TPR runs). The sample was degassed and cooled under

flowing argon to $-5\text{ }^{\circ}\text{C}$, at which pulses of H_2 (5 vol. %)/argon were injected into a stream of Ar flowing through the sample bed.

Results and Discussion

Interaction of hydrogen with $\text{Cu}_x\text{Ce}_{1-x}\text{O}_{2-y}$ mixed oxides during TPR involves the adsorption of hydrogen on all active sites of the cerium oxide surface, storage of hydrogen in the host oxide, and reduction of the CuO component [2]. The qualitative and quantitative characterization of reducibility and reoxidability of different types (*i.e.*, well-dispersed, bulk-like) of copper ions present in the prepared CuCe-1 mixed oxide are determined by TPR and TPO measurements carried out in the temperature range from 0 to $400\text{ }^{\circ}\text{C}$ (Figure 1). Fig. 1a confirms that highly dispersed copper ions in nanocrystalline $\text{Cu}_{0.15}\text{Ce}_{0.85}\text{O}_{2-y}$ can be readily reduced and oxidized at temperatures as low as $200\text{ }^{\circ}\text{C}$ [3]. The extent of further reoxidation of Cu^0 to Cu^{2+} , partial consumption of hydrogen stored in reduced samples, and storage of oxygen during the TPO run are illustrated in Fig. 1b.

Evidently, the total hydrogen

consumption of CuCe-1 solid is larger than the value expected for a complete reduction of the CuO component to Cu^0 (Table 1). The additional hydrogen consumption may be due to surface reduction of CeO_2 [2]. During the reduction process, storage of hydrogen in the oxide, mainly in the bulk (*i.e.*, formation of bronze-like species) and further reaction of these activated hydrogen species with the lattice oxygen ions at $T > 230\text{ }^{\circ}\text{C}$ can take place [4].

Fig. 1a shows that the CuCe-1 sample starts to be reduced at temperatures below $100\text{ }^{\circ}\text{C}$. The reduction steps as well as the simultaneous incorporation of hydrogen into the catalyst structure is completed at temperatures below $250\text{ }^{\circ}\text{C}$. Data shown in Table 1 confirm that in the performed TPR analysis complete reduction of CuO phases was obtained. At least two peaks could be distinguished on the TPR profile of CuCe-1: a very small peak with a maximum at $T = 90\text{ }^{\circ}\text{C}$ and a large reduction peak with a maximum at $155\text{ }^{\circ}\text{C}$ (Fig. 1a). The total hydrogen uptake obtained from the integrated area is $36.2\text{ ml H}_2/\text{g}_{\text{solid}}$,

which is higher than the equivalent stoichiometric total oxygen consumption of $15.3\text{ ml O}_2/\text{g}_{\text{solid}}$ (Table 1).

Subsequently, deconvolution of TPR profile illustrated in Fig. 1a was performed. Three peaks were sufficient to accurately describe H_2 consumption during the reduction of CuCe-1 sample. This suggests that the reduction of intermediate Cu^+ phases to Cu^0 proceeds much faster in comparison to the $\text{Cu}^{2+} \rightarrow \text{Cu}^+$ reduction step. The mathematical analysis of TPR profile illustrated in Fig. 1a reveals that the storage of H_2 into the catalyst structure occurs in parallel to the reduction of CuO (or Cu_2O) phases. It is interesting to note that the amount of H_2 incorporated into the catalyst structure, calculated by means of the performed deconvolution analysis, is in good agreement with the results of TPR analysis (Table 1).

Fig. 1b shows a profile obtained during the TPO analysis of pre-reduced CuCe-1 sample. It should be noted that no oxygen was consumed at $T \leq 0\text{ }^{\circ}\text{C}$. It was also verified by means of pulse chemisorption measure-

Table 1. Results of TPR-1, TPO, TPR-2, TPD- H_2 and TPD- O_2 analyses of CuCe-1 mixed oxide sample.

Analyses	CuCe-1	Analyses	CuCe-1
<i>TPR-1</i>		<i>TPD-H_2 up to $650\text{ }^{\circ}\text{C}$</i>	
total H_2 uptake, $\text{ml}/\text{g}_{\text{solid}}$	36.2	desorbed H_2 , $\text{ml}/\text{g}_{\text{solid}}$	4.7
stored H_2 (HSC ₁), $\text{ml}/\text{g}_{\text{solid}}$	15.0 (41 %) ^a	rest of H_2 , $\text{ml}/\text{g}_{\text{solid}}$	10.3
<i>TPO</i>		<i>irreversibly captured H_2, %</i>	
total O_2 uptake, $\text{ml}/\text{g}_{\text{solid}}$	15.3		69
partial O_2 uptake, $\text{ml}/\text{g}_{\text{solid}}$	4.8 (31 %) ^b	<i>TPD-H_2 up to $400\text{ }^{\circ}\text{C}$</i>	
remained H_2 , $\text{ml}/\text{g}_{\text{solid}}$	5.4 (36 %) ^c	desorbed H_2 , $\text{ml}/\text{g}_{\text{solid}}$	3.9
<i>TPR-2</i>		rest of H_2 , $\text{ml}/\text{g}_{\text{solid}}$	11.1
total H_2 uptake, $\text{ml}/\text{g}_{\text{solid}}$	36.2	irreversibly captured H_2 , %	74
stored H_2 (HSC ₂), $\text{ml}/\text{g}_{\text{solid}}$	15.0	<i>TPD-O_2 up to $400\text{ }^{\circ}\text{C}$</i>	
HSCC, $\text{ml}/\text{g}_{\text{solid}}$	≥ 20.4	desorbed O_2 , $\text{ml}/\text{g}_{\text{solid}}$	0.8

^aPart of H_2 consumed for incorporation into the catalyst structure.

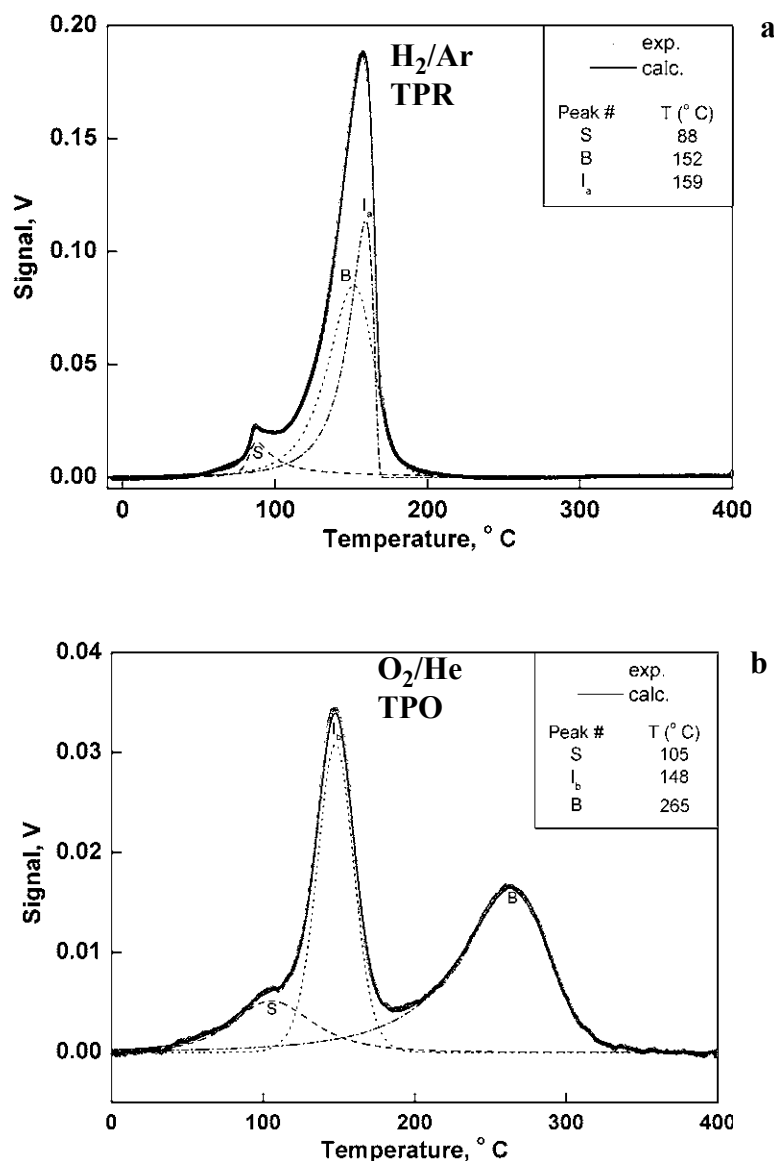
^bPart of O_2 consumed in a reaction with H_2 stored in the catalyst structure.

^cPart of H_2 stored in the catalyst structure after the completion of TPO analysis.

ments carried out at $T = 0\text{ }^{\circ}\text{C}$ that no O_2 was consumed during the preceding detector stabilization period (5 min), in which a catalyst sample was exposed to the oxygen stream. As Fig. 1b shows, the TPO profile measured during the reoxidation of CuCe-1 sample was satisfactorily simulated by assuming the following processes: (i) reoxidation of the well-dispersed Cu phase; (ii) reoxidation of the segregated Cu phase; (iii) consumption of H_2 stored in the catalyst structure. The calculated relative surface areas of peaks belonging to the reoxidation of well-dispersed (17 %) and segregated (83 %) Cu phases, are very close to the corresponding TPR-1 values. Furthermore, the calculated relative surface area of the I_b peak belonging to the consumption of H_2 stored in the catalyst structure, equals to 35 %, which is close to 31 %, calculated from the results of TPO analysis (Table 1). It is reported in Table 1 that significant amount of hydrogen (36 %) remained captured in CuCe-1 sample after the completion of TPO analysis conducted in the temperature range of 0-400 $^{\circ}\text{C}$.

The second reduction of catalyst sample examined in this study was performed and compared to those measured during the TPR-1 analysis. It was found that the TPR-2 profile is shifted towards higher temperatures. This shift is the consequence of substantial decrease of the sample volume (up to one third), which occurred during the TPR-1 analysis. Partially, the shift might be attributed also to the fact that the BET surface area drops considerably after TPR-1 analysis (from 44 to 21 m^2/g). However, this cumulative effect was less pronounced for the CuCe-1 sample (a temperature shift of only 5 $^{\circ}\text{C}$ was noted). Interestingly, the same number of peaks (3) was used to satisfactorily simulate

Figure 1. TPR-1 (a) and TPO (b) profiles of CuCe-1 sample measured in the temperature range of 0-400 $^{\circ}\text{C}$ and predicted by means of deconvolution method. Operating conditions: 50 ml/min (STP), H_2 (5 vol. %)/Ar (a), O_2 (10 vol. %)/He (b), 5 $^{\circ}\text{C}/\text{min}$. Sample weight, 250 mg. The initial state of (b) is fresh sample following TPR-1 run, cooling in H_2 (5 vol. %)/Ar to 0 $^{\circ}\text{C}$ and purging at 0 $^{\circ}\text{C}$ with pure Ar. Designation of peaks: S – well-dispersed CuO species; B – bulk-like CuO phase; I_a – H_2 incorporation in the catalyst structure; I_b – consumption of H_2 incorporated in the catalyst structure during the TPR-1 analysis.



the TPR-2 profile by means of the deconvolution method as was in the case of TPR-1 analysis. Although the BET surface area changes considerably during the TPR-1 analysis, these findings suggest that the interface between the CuO and CeO₂ phases is not modified significantly.

It is evident from Table 1 that significant quantities of hydrogen were stored in the structure of CuCe-1 sample during the TPR-1 analysis. It is also shown that hydrogen initially incorporated in CuCe-1 sample was only partially consumed in the subsequent reoxidation step conducted in the temperature range of 0-400 °C. Since the amounts of hydrogen consumed during the TPR-1 and TPR-2 analyses of CuCe-1 sample were found to be equal, this means that the complete hydrogen storage capacity (HSCC) of this solid was not achieved in the first reduction step. On the basis of the performed analysis, it is concluded that the HSCC for CuCe-1 sample is higher than 20.4 ml H₂/g_{solid}.

TPD-H₂ profiles of pre-reduced CuCe-1 sample were measured at temperatures up to 650 °C. This analysis reveals that very small amount of H₂ was physisorbed on the catalyst surface and that the majority of H₂

stored in the catalyst during the TPR analysis was chemisorbed. H₂ desorption occurred in a wide temperature range, and two wide peaks are noted. Although high temperature of desorption was applied, hydrogen was not completely removed from the structure of CuCe-1 sample, which implies that major part of incorporated H₂ is irreversibly chemisorbed in this solid. For illustration, only about 26 % of hydrogen was desorbed from the CuCe-1 sample in the temperature interval of 0-400 °C.

The TPD-O₂ profile of CuCe-1 sample pre-calcined at 400 °C lies in a very short range of TCD signals, which alludes that in the applied temperature range of 0-400 °C the examined solid exhibits, in comparison to measured TPD-H₂ values, lower ability for the exchange of oxygen. The amount of desorbed oxygen for given temperature range is listed in Table 1. It is seen that TPD-O₂ values are about 3 to 5 times lower compared with TPD-H₂ data obtained in the same temperature range.

In conclusion, the results of this study reveal that despite drastic drop in specific surface area of fresh CuCe-1 sample after the consecutive TPR-H₂/TPD-H₂ treatments, which implies severe morphological changes of the sample, the redox behavior

remains nearly unchanged as both the total hydrogen consumption and hydrogen storage capacity remain constant during successive TPR cycles (TPR-1/TPO/TPR-2). In the future, determination of specific surface area of CuO in calcined Cu_xCe_{1-x}O_{2-y} catalyst as well as dispersion of Cu⁰ in reduced solid will be investigated by means of selective NO and N₂O chemisorption, respectively [5,6].

References

- [1] A. Trovarelli, Catal. Rev. - Sci. Eng. 38(4) (1996) 439.
- [2] Krašovec, B. Orel, A.S. Aricó, H. Kim, Appl. Catal. B 28 (2000) 113.
- [3] C. Lamonier, A. Bennani, A. D'Huysser, A. Aboukaïs, G. Wrobel, J. Chem. Soc., Faraday Trans. 92(1) (1996) 131.
- [4] A. Tschöpe, M.L. Trudeau, J.Y. Ying, J. Phys. Chem. B 103 (1999) 8858.
- [5] J.L.G. Fierro, J. Soria, J. Sanz, J.M. Rojo, J. Solid State Chem. 66 (1987) 154.
- [6] R.M. Dell, F.S. Stone, P.F. Tiley, Trans. Faraday Soc. 49 (1953) 195.
- [6] J.C. Kevlin, M.G. White, J. Catal. 130 (1991) 447.

(Footnotes)

*Corresponding author.
Phone: +386-1-47-60-282; fax:
+386-1-47-60-300. E-mail:
albin.pintar@ki.si.

Micromeritics Provides More Than Instruments

The U.S. Sales department of Micromeritics is staffed with Sales Engineers who are charged with the primary responsibility of identifying and solving problems faced by scientists in the field of particle technology and materials research. In today's business climate, many companies are reducing the size and caliber of their customer support staff. Micromeritics however, remains dedicated to providing high-quality support to customers in pursuit of solutions.

The U.S. Sales staff is a key component of that support. The backgrounds of our staff include chemists, engineers, scientists, and technicians with a great deal of practical laboratory experience. U.S. Sales Engineers are rigorously trained when first joining Micromeritics, spending many weeks participating in "hands-on" exposure to our instruments. Likewise, they are versed in the theory of operation as well as data interpretation so that they can provide first-class support to our customers and prospects.

Each year the U.S. Sales staff participates in on-going training where at least two weeks are spent in training at our headquarters in Norcross, GA. Combining the intensive initial training with on-going training in addition to their vast experience makes them well prepared for the job.

In fact the eight Sales Engineers in our U.S. Staff have more than 85 years of experience with Micromeritics - an average of more than 10 years each! That is hard to find in the sales staff at any other particle technology company!

In terms of customer support, some companies have a handful of scientists who may provide support from a headquarters location. But standing behind our Sales staff, Micromeritics has a significant investment in a long-tenured scientific staff at our headquarters. This staff is comprised of many PhD- and Masters-level scientists and engineers with significant backgrounds in the various particle technology areas, and who are always available to respond to customer problems or questions. The support provided to our customers is many layers deep.

What does this mean to you? It means that we think enough of our customers and prospects to provide people who can help you solve problems, not waste your time. We can enter into discussions about your particle technology needs and identify key areas where Micromeritics can be of assistance. We pride ourselves on our customer satisfaction. A vast majority of our business is derived from repeat customers. Our sales staff understands that we must build lasting, beneficial relationships with our customers and prospects, and strive to be part of their solution team.

Each of our customers and prospects has a direct line to someone who will be an engaged assistant in the event that a question or problem should arise.

Given the investment Micromeritics makes in our direct sales staff, their training and support, and our commitment to continue on this course, we clearly strive to provide more value to our customers now and well into the future. Please let us know how we can assist you with your particle technology problems. We are ready, willing, and able to help you find a solution!

For more information contact, your local U.S. Sales Engineer or Micromeritics U.S. Sales department at 770-662-3633 or ussales@micromeritics.com

Thermocouple Calibration for the AutoChem - Thermostar Interface

This is an excerpt of the complete procedure which can be found at http://www.micromeritics.com/applications/application_notes.aspx (Application Note 135).

The AutoChem series analyzers may be used to trigger automatically the collection of mass spectrometer data via an interface cable, which is specifically designed to provide a hardware interface between the AutoChem and Pfeiffer Vacuum Thermostar (or Quadstar). The interface cable has a "Y" configuration. The single connector cable end is connected to the Thermostar. The double connector cable end is then connected to the AutoChem. With the small connector attached to the Analog I/O and the large connector attached to the Digital I/O.

Pfeiffer Vacuum supplies a suite of software macros to complement the AutoChem cable interface. These macros used with the Quadstar software allow AutoChem users full control of their Thermostar. The Quadstar software requires several parameters to be defined to fully utilize this interface and performing a simple thermocouple calibration is the most significant configuration change.

TCD Configuration

From the Config menu item select the AI Characteristic Curve ... A dialog displaying the characteristic curves for analog input to the Thermostar is displayed. The TCD signal is recorded by AI-Channel 0 and the following parameters should be updated:

- AI-Type field should be changed to TCD
- Unit field should be mV
- The formula should contain "X" - Figure 2

- The Minimal X should be set to -5000
- The Maximal X should be set to 5000

Thermocouple Configuration

The thermocouple signal (Figure 1) is recorded by AI-Channel 1 and the following parameters should be updated for the thermocouple calibration:

- AI-Type field should be changed to T in C
- Unit field should be C
- The formula should contain "X" - Figure 2
- The Minimal X should be set to -5000
- The Maximal X should be set to 5000

A thermocouple calibration curve can now be developed using the AutoChem and the Thermostar. From the main menu in the Parset application, select **Measure** → **MID**. The displayed dialog (Figure 3) is used to specify the signals to be recorded by the Thermostar. For the calibration, right-click on the CH-0 State field and select **ENABLE**; then right-click on the **Det Type** and select **AI** → **T in C** (Figure 3). An additional (mass) signal is also required by the Thermostar, an example is given in Figure 4.

The AutoChem can now be used to generate the thermocouple calibration data. Open a sample file - mstcal01.smp and define a simple experiment:

- Experiment
 - Carrier Flow - Helium 10 ml/min
- Start recording - 1 second



- Set Outputs - Enable DO1 ***
- Temperature ramp
 - Sample ramp - 25 °C/min
 - Temperature - 100 °C
 - Hold - 10 min
- Temperature ramp
 - Sample ramp - 25 °C/min
 - Temperature - 300 °C
 - Hold - 10 min
- Temperature ramp
 - Sample ramp - 25 °C/min
 - Temperature - 500 °C
 - Hold - 10 min
- Set Outputs - Disable DO 1
- Stop recording
- Done

Using the tg-ms software select Run Trend Scan from the Main Selection Menu Dialog (Figure 5). Select the file mstcal and also enter a filename mstcal to save the data (Figure 6). Then click on the "Start on Trigger" button, the Thermostar is waiting for control signals from the AutoChem. From the AutoChem software select Unit 1 → Start Analysis ... and select the mstcal01.smp file, then use the Next button to start the analysis. Data will be recorded by both the AutoChem and the Thermostar, example data are shown in Figures 7 and 8. The Thermostar signal vs. AutoChem tempera-

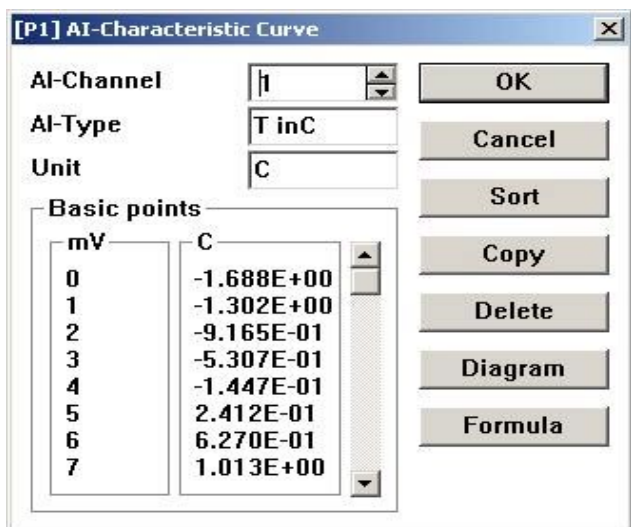


Figure 1: The AI-Characteristic Curve converts the voltage signal from the AutoChem to temperature, and the temperature is then associated with the mass spec ion signal.

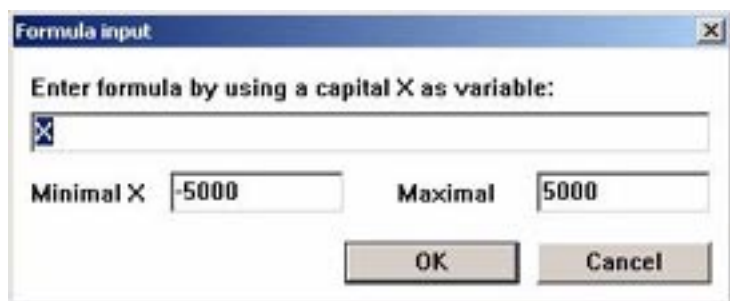


Figure 2: The formula for the AI-Characteristic Curve should be set to "X" for the TCD signal and also to "X" during the thermocouple calibration.

ture plot yields a linear relationship and this equation can then be used to update the AI-1 Characteristic curve (Figure 9). Future analyses will now contain a temperature signal in addition to any mass signals that are recorded.

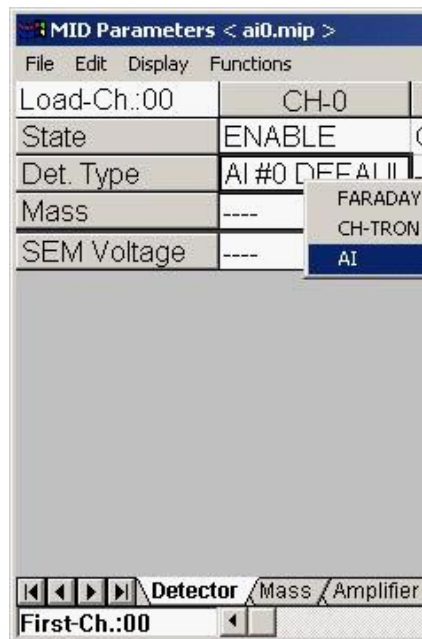


Figure 3: The Measure → MID dialog is used to define the signals to be monitored and recorded by the Thermostar.

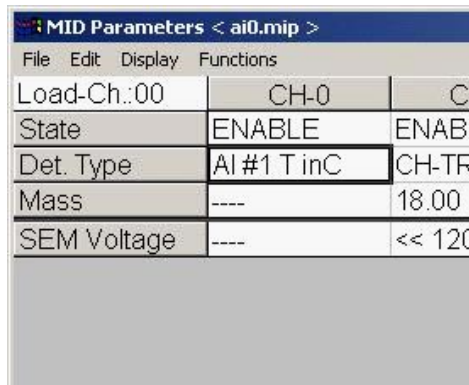


Figure 4: Mass signals are required for the Thermostar to collect data.

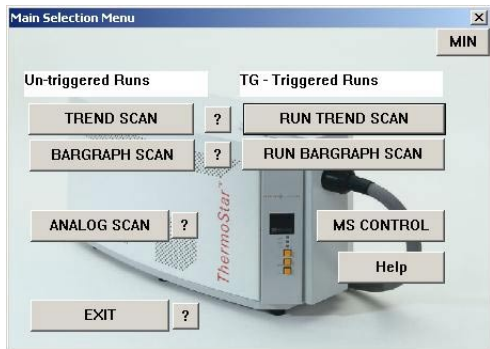


Figure 5: Main Selection Menu from the TG-MS V5 software provides Un-triggered Runs for manual operation and TG-Triggered Runs for the AutoChem.

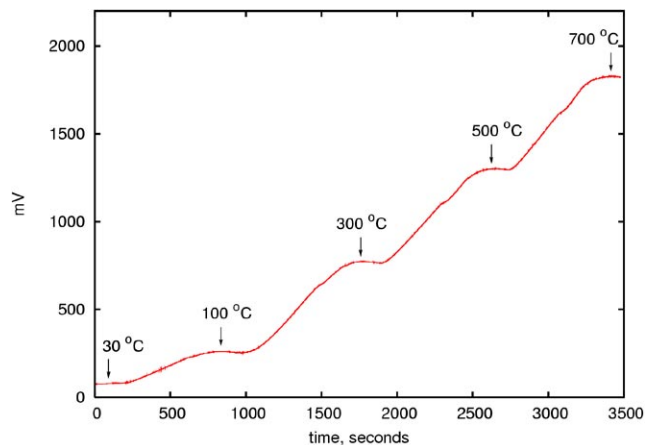


Figure 7: Typical mV versus time collected using the Thermostar.

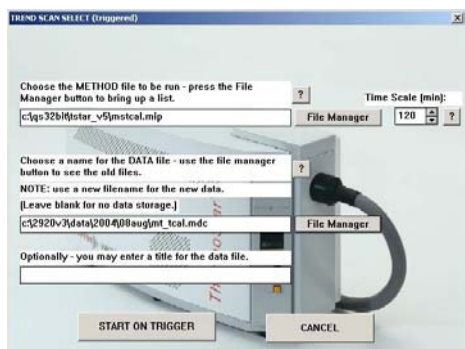


Figure 6: The trend scan select dialog allows users to specify methods and destination files for collected data.

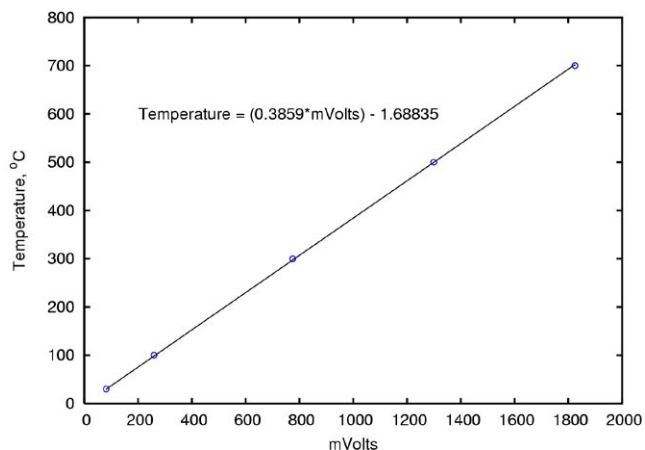


Figure 8: Regression results from AutoChem temperature vs AI-1 mV data collected using the Thermostar.

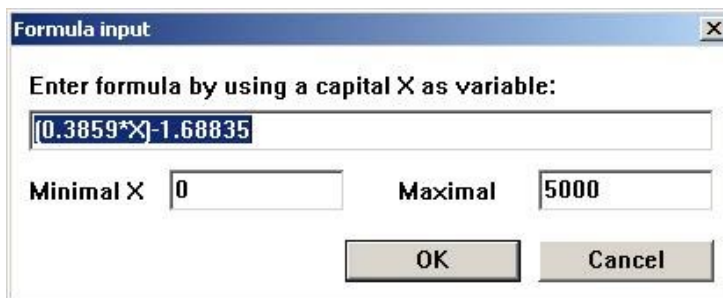


Figure 9: Updated AI-1 formula for the Thermostar temperature calibration.

Micromeritics Instrument Training Courses

Training is provided for most Micromeritics instrumentation at the time of installation. This training presents all the information required for a new operator to quickly become proficient operating the instrument. In cases where personnel changes occur or more advanced training is required, Micromeritics conducts a variety of classes for many of our instruments. These courses are held at our headquarters in suburban Atlanta, Georgia. The courses include:

Detailed Operational Procedures

Items covered are effective sample file creation, use of analysis parameters, and manual sample entry. You'll learn how to utilize the full power and flexibility of the operating software.

Automatic Analysis

Develop correct analysis procedures to optimize collection of accurate, reproducible data. Much of the class time is spent performing analyses in a controlled, tutorial environment.

Systems Utilities

Discover all of the instrument software utilities which help you manage sample information files and directories, protect data, and select system options.

Troubleshooting

Learn techniques that enable you to quickly locate and resolve instrument problems.

Report Generation and Comprehension

Learn to configure reports and obtain more useful information, as well as improve comprehension of the reports produced.

User Maintenance

Practice routine maintenance procedures which improve operation, reduce downtime, and increase data accuracy.

Theory Overview

Learn about the scientific theory upon which each instrument is based and how it applies to the critical factors relevant to successful sample preparation and analysis performance.

Enrollment

Training courses last from 2 to 3 days and are designed to provide hands-on, performance-based instrument knowledge. Small classes guarantee close individual attention. Included in the course materials are a Study Guide, an instrument Operator's Manual, and other handout materials. Certificates of Completion are also awarded to all trainees.

Training 2005

Saturn DigiSizer® 5200
1/4 - 1/6, 8/16 - 8/18

SediGraph 5120
1/11 - 1/13, 8/23 - 8/25

AutoPore IV
2/8 - 2/10, 8/30 - 9/1

TriStar 3000
3/15 - 3/17, 10/11 - 10/13

Gemini V
3/22 - 3/23, 10/4 - 10/5

ASAP 2020 Physi
4/5 - 4/7, 11/8 - 11/10

ASAP 2020 Chemi
4/12 - 4/14, 11/15 - 11/17

AutoChem II
5/3 - 5/5, 12/6 - 12/8

Elzone 5390
6/7 - 6/9, 12/13 - 12/15

For additional information or to register for the class of your choice, contact the Micromeritics Training Department at 770.662.3607. Early registration is recommended since class space is limited.

Events

American Association of Pharmaceutical Sciences

November 8 - 10, 2004
Baltimore Convention Center
Baltimore, MD

PowTex Tokyo

November 9 - 12, 2004
Makuhari Messe
Chiba, Japan

IFPAC - Process Analytical Technology

January 11 - 13, 2005
Crystal Gateway Marriott
Arlington, VA

International Exposition on Advanced Ceramics & Composites

January 25 - 26, 2005
Presented by The [American Ceramic Society](#)
Hilton Hotel
Cocoa Beach, Florida

Attention Authors

We welcome articles and information concerning particle technology applications performed with Micromeritics instrumentation. Everything from a single plot with operating conditions to an in-depth article on physisorption, chemisorption, etc. with supporting plots will be considered. If your material is published in The microReport, you will receive a copy of Analytical Methods in Fine Particle Technology by Paul A. Webb and Clyde Orr.

Send your article to:
Laurel Whitmire, Editor
The microReport
MICROMERITICS
One Micromeritics Drive
Norcross, GA 30093-1877
laurel.whitmire@micromeritics.com

Include your title, return address and phone number. Contributions cannot be returned, but each will be acknowledged.

How To Reach Us

Micromeritics offers over 50 sales, service, and distribution offices throughout the world. For additional information, a free product demonstration, or the location of the office nearest you, call or write:

HEADQUARTERS:

Micromeritics Instrument Corporation
One Micromeritics Drive
Norcross, GA 30093-1877
USA
Telephone:
U.S. Sales (770) 662-3633
International Sales (770) 662-3660
Fax (770) 662-3696
WEB: www.micromeritics.com

SUBSIDIARIES:

Micromeritics China
Apt. 5H, No. 1 Building
Hua-Ao (Epoch Center)
No. 31 Zi Zhu Yuan Road, Hai Dian District
Beijing 100089, P.R., CHINA
Tel: (+86) (0)10-6848-9371
Fax: (+86) (0)10-6848-9371

Micromeritics France
Zaet St. Maximin
181, rue Henri Bessemer
F-60100 Creil, FRANCE
Tel: (+33) (0)33-3-44-64-6080
Fax: (+33) (0)33-3-44-64-6089

Micromeritics GmbH
Erfststrasse 54
D-41238 Mönchengladbach, GERMANY
Tel: (+49) (0)2166-98708-0
Fax: (+49) (0)2166-98708-88

Micromeritics Ltd.
Unit 2, Chestnut House
178-182 High Street North
Dunstable, Bedfordshire LU6 1AT
ENGLAND
Tel: (+44) (0)1582-475248
Fax: (+44) (0)1582-475252

Micromeritics N.V./S.A.
Eugene Plaskylaan 140B
1030 Brussels, BELGIUM
Tel: (+32)2-743-39-74
Fax: (+32)2-743-39-79

Micromeritics SRL
Via W. Tobagi n. 26/7
20068 Peschiera Borromeo
Milano, ITALY
Tel: (+39) (0)2 553 02833
Fax: (+39) (0)2 553 02843

THE microReport

The microReport newsletter is published by Micromeritics Instrument Corp.
Editor: Laurel Whitmire,
(770) 662-3654.

Subscriptions are free to particle technology users and can be started simply by writing to The microReport editor.

Copyright 2004 Micromeritics Instrument Corporation. All rights reserved.

All trademarks noted herein are the property of their respective owners.



Micromeritics
One Micromeritics Drive
Norcross, GA 30093-1877, U.S.A.

