

CryoCooler II

AutoChem
accessory

Subambient temperature controller

Features

- Provides subambient sample cooling to $-120\text{ }^{\circ}\text{C}$
- Permits ramping sample temperatures upward from $-120\text{ }^{\circ}\text{C}$ at user-specified rates from 5 to $50\text{ }^{\circ}\text{C}/\text{min}$
- Allows subambient temperatures to be seamlessly transitioned into above-ambient region
- Can be used for rapid furnace cooling to speed analysis



Micromeritics' CryoCooler II accessory for its AutoChem instruments provides sample temperature control from $-120\text{ }^{\circ}\text{C}$ to the instrument maximum of $+1110\text{ }^{\circ}\text{C}$. It permits ramping temperatures at rates from 5 to $50\text{ }^{\circ}\text{C}/\text{minute}$, selectable in $1\text{ }^{\circ}\text{C}/\text{minute}$ in-

crements, while transitioning seamlessly from subambient to above ambient as shown in figure 2. Analyses beginning at subambient temperatures are especially important when characterizing catalysts incorporating noble metals such as platinum and ruthenium.

The CryoCooler II attaches a source of LN_2 directly to the AutoChem furnace and plugs into the instrument control system. All operations thereafter are computer-controlled.

TCD Signal vs Temperature

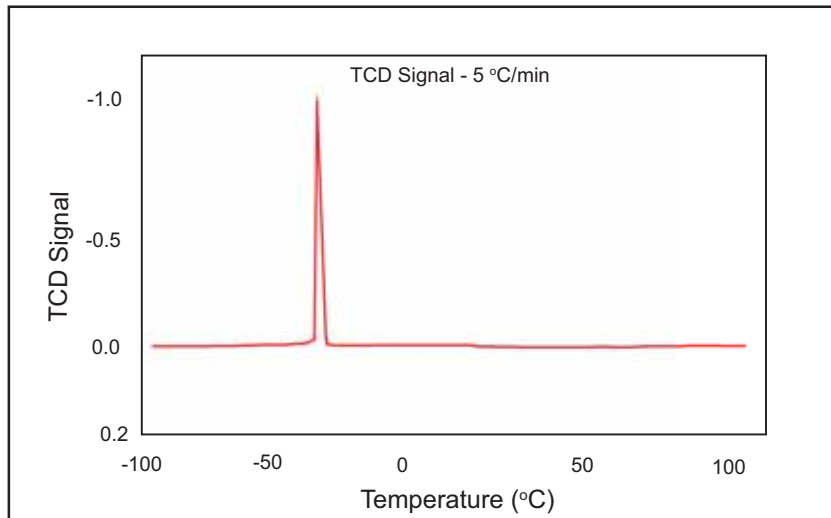


Figure 1

Temperature-programmed reduction of 50% platinum on carbon.

Temperature vs Time

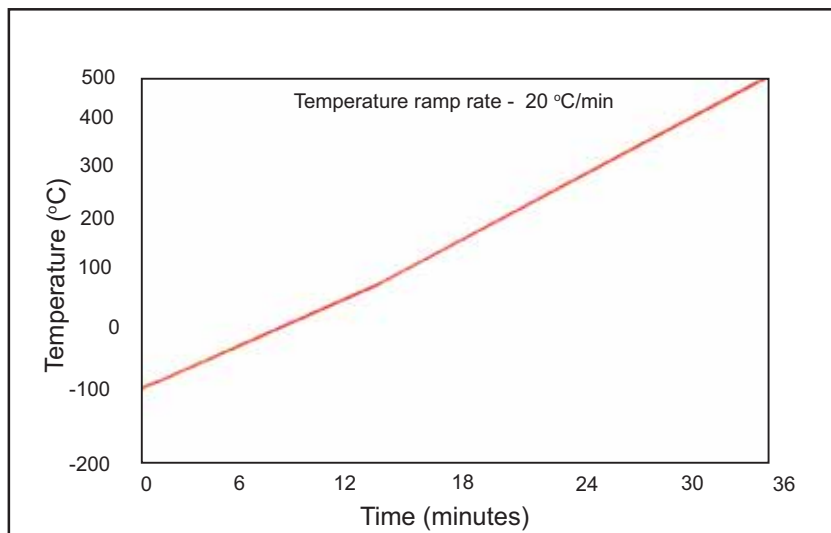


Figure 2

Typical linear temperature profile obtained using the CryoCooler II.

CryoCooler II

Platinum Catalyst Research Aided by New Subambient Temperature CryoCooler II

Subambient temperature reduction is critical in understanding the activity of platinum catalysts. Platinum-based catalysts are by far the most significant class of materials used in petrochemical and chemical reactors. Platinum supported on graphite and promoted by ruthenium, for example, is currently the focus of much research for use as the anode in fuel cell applications. Platinum undergoes a two-step reduction from Pt^{4+} to Pt^{2+} at subambient temperature and then to Pt^0 . This Pt^{4+} -to- Pt^{2+} transition is a strong indication of catalytic activity. Researchers who work with platinum, ruthenium, and nickel will find the CryoCooler II accessory an invaluable aid in their efforts.

Micromeritics' AutoChem fitted with the CryoCooler II accessory creates a powerful combination for characterizing platinum-based catalysts. The CryoCooler II now incorporates a new centrifugal system for pumping liquid nitrogen and new programming which result in sample and furnace cool-down to $-100\text{ }^{\circ}\text{C}$, smooth upward and downward temperature ramps, stable subambient temperature holding, and seamless transition into above-ambient temperature regions. Figure 1 is a typical plot of furnace temperatures with time starting at and above ambient, ramping down and up in temperature at rates from 5 to $50\text{ }^{\circ}\text{C}/\text{min}$, holding subambient temperature, and merging into elevated furnace temperatures. Figure 2 shows the temperature-programmed reduction of platinum supported on graphite. There are two significant features on this plot. The first is the very strong reduction peak at $-36\text{ }^{\circ}\text{C}$ which is characteristic of the transition from Pt^{4+} to Pt^{2+} , and the second is a desorption or slight release of surface hydrogen near $10\text{ }^{\circ}\text{C}$. By adding ruthenium to platinum, as shown in Figure 3, there is created a completely different reduction profile. The primary reduction peak has shifted to $18\text{ }^{\circ}\text{C}$ with several reduction peaks indicating oxidation states for the catalyst. There is, however, a similar release of hydrogen after the reduction. The addition of ruthenium to the platinum has created a new alloy that has very different properties from platinum alone. It is very easy to distinguish these effects using the AutoChem and CryoCooler II by overlaying these plots as in Figure 4.



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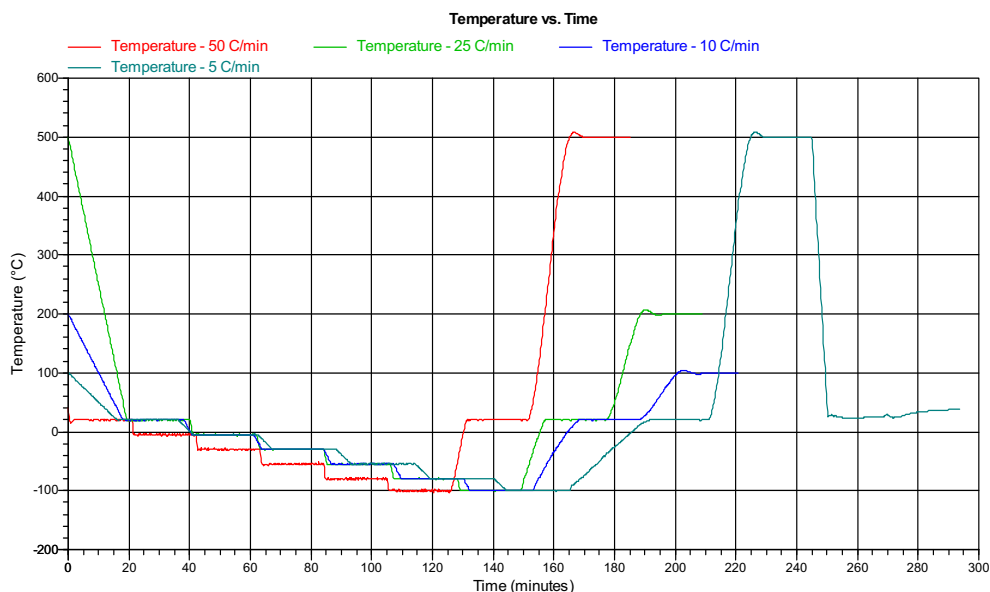


Figure 1. Examples of Furnace Temperature Control at Both Subambient and Elevated Temperatures

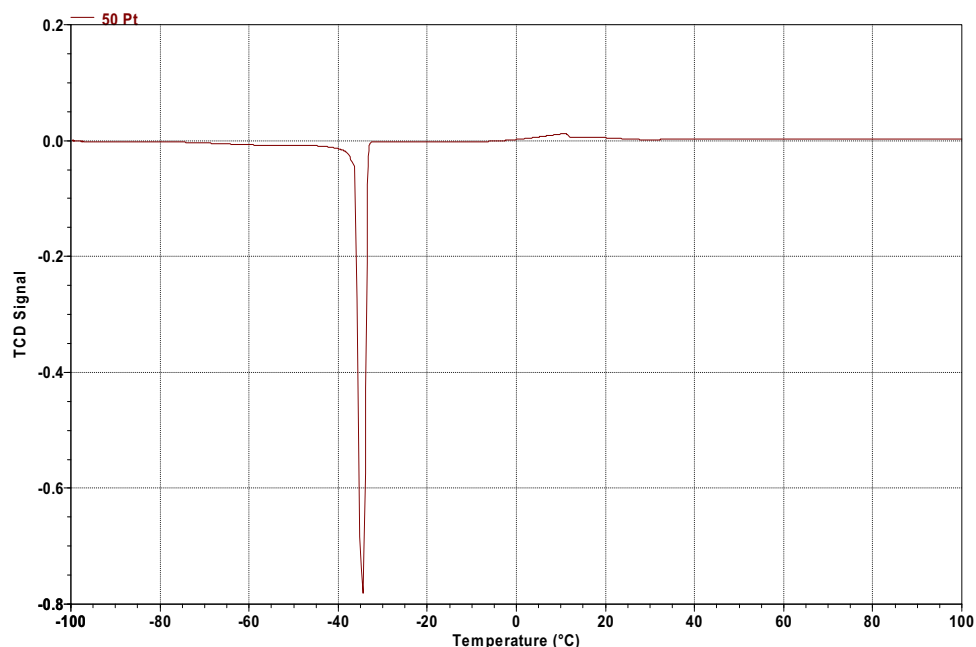


Figure 2. Temperature-Programmed Reduction of Platinum on Carbon Catalyst

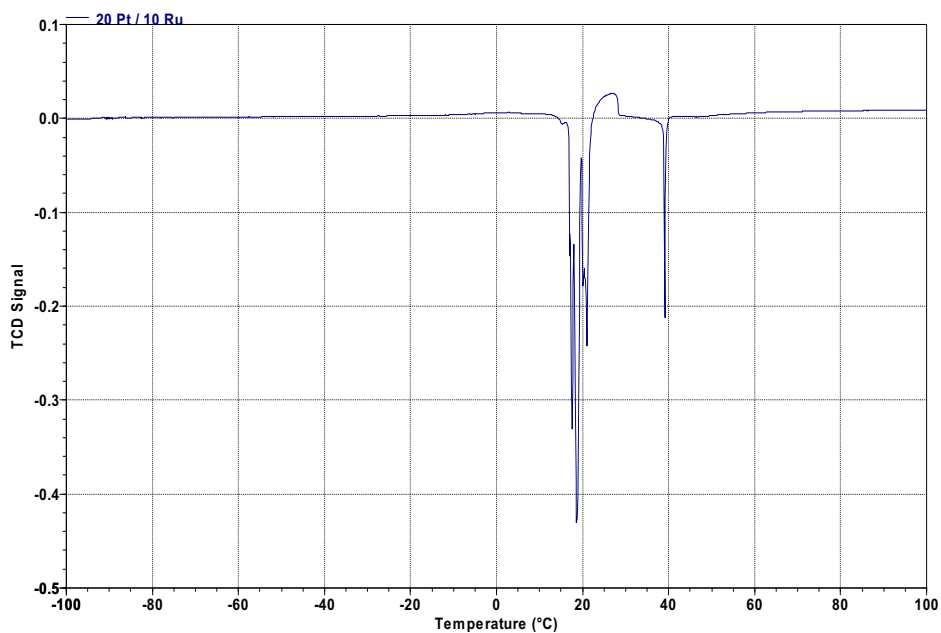


Figure 3. Temperature-Programmed Reduction of Platinum/Ruthenium on Carbon Catalyst

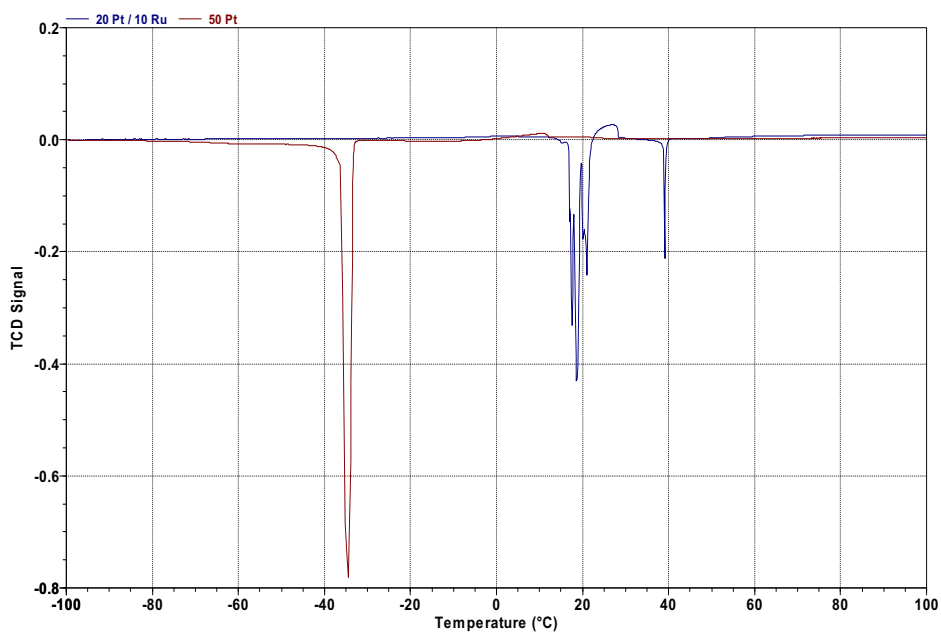


Figure 4. Overlay of Data from Figures 2 and 3