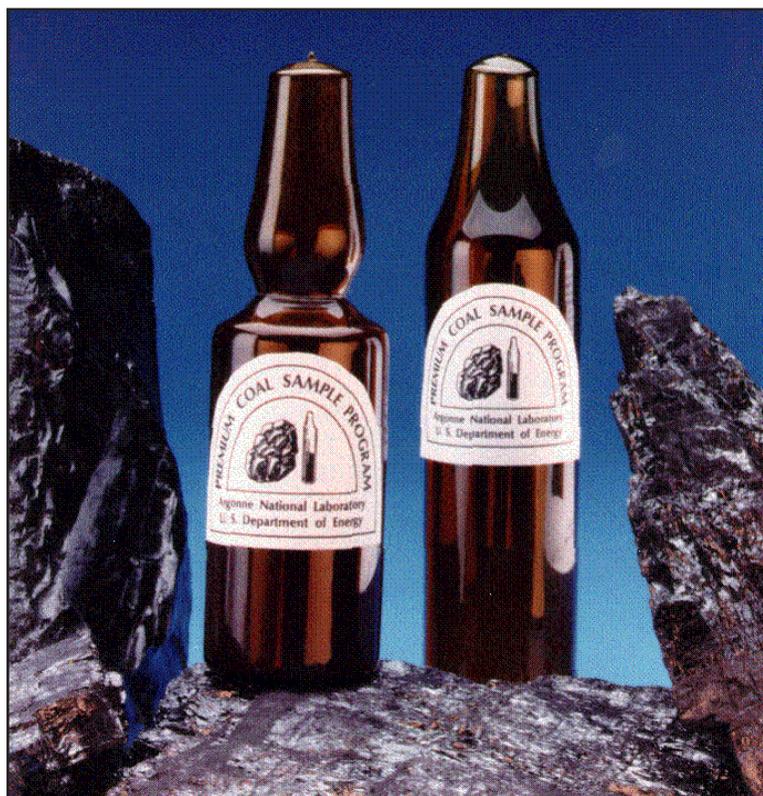


## Argonne Premium Coal Sample Program

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*Annual Technical Progress Report  
Reporting Period: 2/2006–2/2007*

**Chemistry Division**



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by  
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February 2007

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## **Abstract**

This project provides highly uniform, premium (unexposed to oxygen) coal samples to researchers investigating coal structure, properties and behavior, and maintains accessible databases of published reports describing work carried out on the Argonne Premium Coal Samples. The samples are made available to DOE researchers and others. The eight carefully selected samples have been kept in as pristine a condition as possible through careful control the conditions in all stages from sample collection throughout processing and packaging. The samples are available in glass ampoules to ensure sample uniformity and maintain premium quality to ensure sample integrity.

## **Executive Summary**

The goal of the Premium Coal Sample Program is to provide the best samples for basic coal science that can be obtained anywhere. These samples are used for comparison, correlation, and increasing the general understanding of the chemistry of coal. To date, more than 33,500 coal samples have been shipped to 1500 government, academic, and industrial researchers all over the world. A number of research laboratories have reported that they currently use only the Argonne Premium Coal Samples for basic research. Proposals continue to be written by university researchers around the world specifying the use of the samples in their proposed programs. The bibliographic database of reports describing work carried out with Argonne Premium Coal Samples currently contains more than 720 citations, and this number is continually increasing.

This report reflects the work statement submitted to DOE on March 15, 2007, continuing the project to distribute premium coal samples to DOE contractors and others performing coal research.

## **Introduction and Background**

The objective of the Argonne Premium Coal Sample (APCS) Program is to supply highly-uniform, premium (unexposed to oxygen) coal samples to researchers investigating coal structure, properties and behavior, and to maintain accessible databases of published reports describing work carried out with Argonne Premium Coal Samples. The eight carefully selected samples are available in glass ampoules in quantities to provide for 10 or more years' requirements. The term "premium" in the name refers to the extreme care taken in each step, from selection through distribution, including the minimal exposure to oxygen, through mixing, and extensive inventory of ampoules for long-term supplies. The samples have been kept in as pristine a condition as possible through careful control of the conditions in all stages from sample collection throughout processing and packaging.

A number of "coal banks" supply coal samples for research and analysis. The purpose of the Argonne Premium Coal Sample Program, however, goes beyond simply supplying coal samples. By ensuring sample uniformity and maintaining premium quality, researchers are assured that results generated from analysis of Argonne Premium Coal Samples are comparable with results generated by other workers using premium coal samples. By maintaining long-term supplies of high-quality coal samples, the APCS program is intended to allow researchers to develop, over time, a comprehensive database of information for a carefully selected suite of U.S. coal samples. Maintenance of these databases is a key function of the Premium Coal Sample Program, and differentiates the APCS Program from other coal banks.

The full set of eight coals for the Premium Coal Sample Program includes a lignite, subbituminous, high volatile, medium volatile, and low volatile bituminous, as well as a liptinite-rich, an inertinite-rich, and a coking coal. The coals are:

<b>APCS 1</b>	<b>Upper Freeport</b>	<b>PA</b>	<b>Med. Vol. Bit.</b>
<b>APCS 2</b>	<b>Wyodak-Anderson</b>	<b>WY</b>	<b>Subbituminous</b>
<b>APCS 3</b>	<b>Illinois #6</b>	<b>IL</b>	<b>High Vol. Bit.</b>
<b>APCS 4</b>	<b>Pittsburgh #8</b>	<b>PA</b>	<b>High Vol. Bit.</b>
<b>APCS 5</b>	<b>Pocahontas #3</b>	<b>VA</b>	<b>Low Vol. Bit.</b>
<b>APCS 6</b>	<b>Blind Canyon</b>	<b>UT</b>	<b>High Vol. Bit.</b>
<b>APCS 7</b>	<b>Lewiston-Stockton</b>	<b>WV</b>	<b>High Vol. Bit.</b>
<b>APCS 8</b>	<b>Beulah-Zap</b>	<b>ND</b>	<b>Lignite</b>

The coals were selected on the basis of C, H, S, and O contents, as well as maceral content and geological age. Ampoules containing 5 grams of -100 mesh or 10 grams of -20 mesh material from each sample are available. Analytical information is available on the web site. The methods of selection, collection, transportation, processing, packaging, distribution, and characterization are summarized. The eight samples were each collected in about 1 to 1-1/2 ton quantities, placed in steel drums, purged with argon, and taken to Argonne National Laboratory (ANL) for processing. After transfer to a nitrogen-filled enclosure, they were crushed, pulverized, mixed, and packaged in sealed amber borosilicate ampoules. Five-gallon carboys hold about 80% of the batch in reserve for filling more ampoules after the original samples (about 5,000 of -20 mesh or 10,000 of -100 mesh) are depleted.

To date, more than 33,500 coal samples have been shipped to 1500 government, academic, and industrial researchers all over the world. The bibliographic database of reports describing work carried out with Argonne Premium Coal Samples currently contains more than 720 citations, and this number is continually increasing.

## **Results and Discussion**

### **1. APCS Sample Bank Supply**

One of the important characteristics of a sample supplier is the ability to make the samples available over a long period of time. The preparation of sample material was planned to provide for the equivalent of 50,000 of 5 gram ampoules and 25,000 of the 10 gram ampoules. This is accomplished by the initial preparation of 10,000 of the 5 gram ampoules and 5,000 of the 10 gram ampoules (see Figure 1). The eight samples are available in glass ampoules in quantities to provide for 15 or more years' requirements.



**Figure 1. The Argonne Premium Coal Samples are available in 5g (-100 mesh) and 10g (-20 mesh) in sealed glass ampoules.**

The balance of the ampoules is prepared by placing an appropriate number of the five-gallon carboys into the nitrogen filled enclosure of the processing facility. Each carboy holds about 10 kilograms. The carboys are opened in the nitrogen filled environment, remixed and sealed into more ampoules to replenish the inventory. The samples and borosilicate carboys are kept in racks in a dark storage room that is kept close to 72 °F year-round. The borosilicate carboys can be used to replenish supplies of the ampoules whenever needed to sustain the inventory for shipments (see Figure 2).

At the present rate of shipping, we estimate that 7-10 years of all premium coals are available. Historically, the samples in greatest demand, Illinois #6 and Wyodak, will meet the minimum time frame outlined above without having to restart the processing facility. In this reporting year, the samples in greatest demand have shifted to Upper Freeport seam, medium-volatile bituminous coal and Pocahontas, low-volatile bituminous coal.

Argonne has doubled the nominal fee charged for each coal sample delivered. We also expect to recover all shipping and packaging costs.



**Figure 2. Long-term storage of Argonne Premium Coal Samples in five-gallon carboys.**

## **2. Coal Sample Processing Facility**

There are sufficient supplies of all coal samples for the next several years based on the current rate of sample distribution. The coal processing facility continues to be maintained under this contract, but is available for other uses. A description of the facility follows. A facility is available which can be used to pulverize quantities of a solid, up to one ton, to different degrees of fineness in an inert atmosphere, thoroughly mix them, and package them in uniform, small Pyrex ampoules containing 5-10 grams or larger containers, such as five-gallon carboys.

This facility was designed and built in the period 1983-85 by members of the Chemical Technology Division. It currently fully occupies one large room (B-140) in Building 211 at Argonne National Laboratory. The room is accessible through a padlocked wire gate. The dimensions of the room are about 30' x 40' with headroom of about 13'.

The processing facility consists of a coal sample processing glove box complex and a gas handling facility (see Figure 3). The glove box complex is a U-shaped structure, consisting of two separate glove boxes linked by a mixer-blender.



**Figure 3. Argonne Premium Coal Sample processing glove box complex.**

Each glove box consists of aluminum sheets with Plexiglas windows to allow for viewing and ports for 70 pairs of rubber gloves long enough to allow use of the full length of an arm. Each glove box has a large air lock at one end for insertion or removal of various material and equipment. A second air lock is on the second glove box to facilitate the insertion or removal of ampoules. The glove boxes are connected by means of ducts to the gas handling system, which was designed to change the atmosphere in the boxes from ambient air to nitrogen, with an oxygen content at or less than 100 ppm, and maintain that atmosphere during coal sample processing. The change is made at ambient atmospheric pressure. The gas handling facility includes a large blower fan to circulate gas through the boxes and handling system. A manifold allows one to purge the system with large dewars of liquid nitrogen. A separate manifold allows introduction of hydrogen, which is used either as a redundant for oxygen in special catalytic beds or as a fuel for an oxygen-hydrogen torch for sealing the Pyrex ampoules used to contain the coal samples. This manifold is also used to provide oxygen for the torch. Purging is used to reduce the oxygen content to about 15 and then the catalytic unit is used to reduce the oxygen content to 100 ppm or less.

In normal processing, a fork lift equipped with clamps was used to pick up a 55-gallon drum, which was then fitted with casters on the bottom. Three drums were

placed in the first airlock. The airlock door was closed and nitrogen was used to change the airlock atmosphere to less than 100 ppm oxygen by means of inlets for the nitrogen and vents for the mixed gases. A gas sample tube attached to the top of the air lock was used to convey gas to an oxygen analyzer to establish the oxygen content. When the desired purity was reached, the nitrogen purge was stopped, and the door separating the air lock was opened to allow access to the processing facility, which had previously been purged. Drums were rolled, one at a time, to a hydraulic drum dumper made by the Tubar company. This hoist would raise the drum and empty its contents into a Jacobsen rotating bar crusher. The screen in the crusher was selected to crush the coal chunks to less than 1/2" diameter. The crushed coal was allowed to flow to the base of a vibrating lift (made by FMC). The lift raised the coal to an elevated Fitzmill pulverizer equipped with either an initial -20 mesh or later -100 mesh screen to control the particle size of the product pulverized coal. All of these operations were carried out in the L-shaped glove box with heights up to 13' and width up to 5'.

The pulverized coal was blown by the force of the rotating hammers into the Littleford 2000 liter mixer-blender (see Figure 4). This blender will hold an entire one ton sample of pulverized coal. Blending is done for a time long enough to ensure thorough mixing. The temperature inside the blender is monitored to avoid thermal stress to the sample.



**Figure 4. Littleford 2000-liter mixer-blender in the APCS Facility.**

The coal is transferred to a long horizontal tube or conveyor system made by Dynamet, containing a chain and paddles to transport the coal to an intermediate filling station for five-gallon lever-lock pails for repulverizing to -100 mesh, or Pyrex carboys for long-term storage, or an ampoule filling station made by Kuchar Industrial Service and Supply of Joliet, Illinois. At the ampoule filling station, the coal is fed to individual ampoules for a calibrated period of time to deliver the desired weight of sample using a Mateer-Burt system. After each ampoule is filled, the station rotates a turntable to move an empty ampoule into place while the oxygen-hydrogen torch seals the top of the ampoule. The stoichiometric composition of the flame is controlled by a Linde mass flow controller. The ampoule sealing process is computer-controlled using software, which can be modified to match the calibration times. Ampoules are accumulated in stainless steel trays for transfer on racks of rollers to the airlocks. Empty ampoules are loaded into the glove box through an evacuable airlock as the operation progresses.

### **3. On-line Information and Publications**

The Argonne Premium Coal Samples represent the broad array of United States coals in a single set that has been very carefully selected, prepared, and stored. To make information concerning the Argonne Premium coals more accessible to researchers, a World Wide Web home page provides access to APCS information available via the Internet. The APCS Home Page can be reached via the Argonne National Laboratory World Wide Web Home Page at: <http://www.anl.gov/PCS/pcshome.html>. The home page provides complete access to essentially all of the information currently contained in the APCS Users Handbook. Fully searchable bibliographic information for all of the references currently in the citations database is also available on-line via the APCS Home Page. The scope of the scientific research contributions continues to be broad. The United States, Japanese, Canadian, Australian, and European contributions dominate the research contributions.

Publications based on the use of the coal samples remained strong and the samples retain a prominent role in work reported at different national and international meetings. As stated on the web site, authors are requested to self-report publications that utilize the coal samples. An additional 25 publications were reported in this way. Not all authors report the use of coal sample in research publications. A search of the literature citing Argonne Premium Coal Samples revealed an additional 17 unique publications and reports. Not surprisingly, the use of APCS in gas sorption studies, particularly carbon dioxide sorption research, has increased markedly in this past year.

#### 4. Distribution of Coal Samples to Users

In the time frame covered by this report, over 100 coals ampoules and several kilograms of coal chunks were shipped to government, academic, and industrial organizations (see Table 1). The orders were roughly equally split between United States and international scientific research communities. A number of research laboratories have reported that they currently use only use the Argonne Premium Coal Samples for basic coal research.

**Table 1. Number of Argonne Premium Coal Sample Program Shipments  
February 13, 2006 - February 15, 2007**

<b>APCS</b>	<b>5g -100 mesh</b>	<b>10g -20 mesh</b>	<b>Chunks</b>
UF	23	3	
WY	9	6	
IL	6	3	
PITT	1	3	260 g
POC	28	5	253 g
UT	1	4	732 g
WV	1	3	
ND	2	3	

Organizations supplied with coals samples and/or data include:

ETH Honggerberg, Zurich, Switzerland  
Tokoku University, Sendai, Japan  
NIOZ, Department of Marine Biogeochemistry, Den Hoorn- Texel, The Netherlands  
University of Nottingham, United Kingdom  
Argonne National Laboratory, Argonne, IL USA  
Private individual, Colorado, USA  
NETL, Pittsburg, PA USA (multiple samples used for various reports and publications)  
Cornell University, Ithaca, NY USA  
Bonner Analytical, Hattiesburg, MS USA  
Carnegie Institution, Geophysical Laboratory, Washington, DC USA  
ExxonMobil Research & Engineering, Annandale, NJ USA  
Seishin Trading Co, Kobe, Japan  
Pennsylvania State University, State College, PA USA

Additional clients have received information from the on-line Users Handbook via World Wide Web, accessed by researchers from around the globe.

## **Conclusions**

The Argonne Premium Coal Samples, and data supported under this contract, continue to be distributed to DOE contractors and others performing coal research. There is every indication that the demand for these premium coal samples will remain high in the coming years.



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