Coal Samples Available from the Premium Coal Sample Program

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INTRODUCTION

The purpose of the Premium Coal Sample Program (PCSP) is to provide the basic coal science research community with long term supplies of a small number of premium coal samples that can be used as standards for comparison and correlation. The premium coal samples produced from each coal and distributed through this program are chemically and physically as identical as possible, have well characterized chemical and physical properties, and will be stable over long periods of time. Coals are mined, transported, processed into the desired particle and sample sizes, and packaged into environments as free of oxygen as possible. Humidity is also to be controlled to keep the coals as pristine and in as stable a condition as possible.

There had been a feeling that such a program was needed for quite some time (1,2,3). Different authors and workers have expressed a concern over the difficulty in obtaining well selected, collected, prepared and characterized samples over a long period of time. The variation in properties through a coal seam over relatively short distances and the sensitivity of coal samples to oxidation in typical sample containers lead to difficulties in attempts to reproduce the work of other coal researchers. The PCSP is intended to relieve these concerns.

A number of steps are involved in the preparation and distribution of coal samples. These include selection, collection, transportation, preparation or processing (including mixing and packaging), storage, characterization, shipping and data dissemination. Each of these is necessary, and it one of the goals of the PCSP to provide the best quality possible in each of the steps to ultimately provide the best quality samples that can be distributed. Accordingly considerable care and planning has gone into each of the steps mentioned.

SAMPLE SELECTION

The support from the Chemical Sciences Division of the Office of Basic Energy Science will provide for a suite of eight samples. These samples are to provide as broad a range of representation as possible of U. S. coals. The choice of samples has involved a

consideration of the chemical composition, specifically the carbon, hydrogen, sulfur and oxygen contents to obtain representatives of the important ranges available. After consideration of the chemical composition, some specific coals were selected. These include a North Dakota lignite, Wyoming subbituminous, Illinois #6 high volatile bituminous, Upper Freeport medium volatile bituminous, Pocahontas low volatile bituminous, and a Pittsburgh #8 bituminous coal. Other coals are still under consideration for the remaining two samples.

SAMPLE COLLECTION

Sample collection must take place in an active commercial mine to assure the "freshest" coal possible. The collection is done under the supervision of at least one person from the U. S. Geological Survey. Either or both of Drs. Blaine Cecil and Ron Stanton supervise the collection of the whole seam or channel sample. For an underground sample, a continuous miner is used to expose a fresh seam face. When possible the miner is used to isolate a wedge shaped or rectangular block of coal. After roof bolting the floor around the sample is cleared and plastic sheets are put down to collect the actual sample. A three man crew from the Pittsburgh Testing Laboratory at Homer City, Pennsylvania led by David Allen uses hand picks to remove the sample. Coal is placed in double plastic bags in thinner seams (less than 5' thick), tied and taken to the surface for transfer to 55 gallon stainless steel drums. For thicker seams, the drums are taken directly into the mine, and the samples are shoveled carefully into the drums. Typical collection periods are 3-4 hours from the beginning of the collection to arrival of the sample at the surface.

A core sample was taken for the thick subbituminous sample. A contractor was obtained to provide three cores. A 3" core was taken for the USGS log, another 3" core was obtained to provide chunks of coal for long term storage, and a 6" core was used for the coal to be processed. Cores were rinsed and loaded into drums in the same manner as coal from thinner seams.

TRANSPORTATION

A refrigerated semi-truck is used to transport the sample to ANL for processing. The truck loads the empty drums, cylinders of argon used for purging and other necessary equipment at ANL. This load is taken to the mine site. At the mine the plastic bags of coal are dumped into the drums or the full drums are placed on the truck. The drums are purged with 10 or more volumes of argon gas to reduce the oxygen concentration in the drums to below 100 ppm. The load is then taken directly back to ANL for processing. Typically the sample arrives at ANL within 24 hours after being loaded on the truck. The truck temperature is kept at about 42 F en route.

PROCESSING

A unique facility has been built at ANL to process the coal samples. It is a large glove box about 12' tall, 5' wide and 40 feet long. There are 70 pairs of long rubber gloves in the walls of the box to permit manipulation of the sample and equipment during the processing. The oxygen concentration is maintained below 100 ppm during the processing through the use of a catalytic system to combine hydrogen with impurity oxygen.

At ANL the truck is unloaded and drums are taken to the processing facility with a forklift, weighed and placed in groups of three into an initial airlock. After purging the airlock, the drums are opened, and rolled on casters to a hydraulic drum dumper. The dumper picks the drums up and pours the contents into a crusher. The coal is broken into pieces no larger than 1/2" thick. The crushed coal passes through a chute to a vibrating lift which raises the coal to the top of the box for feeding to a pulverizer. Initially the pulverizer grinds the coal so that the particles pass through a 20 mesh screen. The ground coal is accumulated in a Littleford blender with a 2000 liter capacity. This will hold one ton of coal. After the entire batch has been ground it is then thoroughly mixed in the blender. Special studies were carried out to establish the mixing characteristics with coal samples and set the required duration of mixing. The mixed coal is moved from the blender with a tubular conveyor to either a discharge chute used to fill 5 gallon pails for transfer to the initial airlock for regrinding to pass a 100 mesh screen or to fill 5 gallon borosilicate glass carboys for long term storage. Intermittently portions of the sample are conveyed to an ampoule filler-sealer. Here amber borosilicate vials are filled with either 10 grams of -20 mesh material or 5 grams of -100 mesh material. A hydrogen-oxygen torch is used at stoichiometric flame conditions (controlled with a gas mass flow controller) for the sealing.
In processing,it is planned to prepare 5,000 of the 10 gram ampoules of -20 mesh material and 10,000 of the 5 gram ampoules of -100 mesh material. About 80% of the coal is stored in carboys which can be used later to fill additional ampoules when the supply is depleted.

STORAGE

The ampoules and carboys are kept in a separate storage room at about 72 F. This room is usually dark.

CHARACTERIZATION

The samples are characterized for three purposes. The first purpose is homogeneity determination. Initially, samples taken during the filling of the pails, carboys and ampoules are placed in holders for irradiation in the University of Illinois TRIGA reactor. The disintegration rates of induced Na, K and As are measured for bituminous coals, while the Na, As, La and Sc were used for the subbituminous coal due to the limited amount of K. The rates are compared for the different samples to establish the

homogeneity of the samples. The second purpose for characterization is the establishment of the analytical qualities of the coal. Samples are then analyzed in an interlaboratory program that is ongoing. Information on the program is available from the Program Manager. Additioal participation is possible. Standard ASTM methods or known variations of these are used. The analyses include the ultimate (C,H,N,S), proximate (volatile matter, fixed carbon, ash and moisture), calorific or Btu values, sulfur forms, major and minor elements in the ash, and equilibrium moisture. Maceral analysis and vitrinite reflectance are also carried out. For bituminous coals the free swelling index and Gieseler plasticity are also determined. The third type of analysis is used to establish the stability of the samples. The Gieseler plasticity is used as a sensitive indicator of oxidation for the bituminous samples. This test is done for the initial coals and is repeated periodically to establish the constancy of the values. For non-bituminous coals other techniques are being used and developed. The slurry pH is used to establish the oxidation which results in conversion of pyrite to sulfate.

AVAILABLE COAL SAMPLES

1. The first sample is an Upper Freeport sample collected near Homer City Pennsylvania in January 1985. This is a medium volatile bituminous coal from a 4' thick seam at the point of collection. The sample characterization is continuing. The preliminary values are: (as received basis)

carbon:	74-75%
hydrogen:	4-5%
total sulfur:	2-3%
ash:	12-13
moisture	1-2%

These samples are available in 5 grams of -100 mesh or 10 grams of -20 mesh material.

2. The second sample, a subbituminous coal from the Wyodak seam, was collected about six miles northeast of Gillette, Wyoming in October, 1985. The seam is about 120' thick at the point of collection. In this case the sample for processing consisted of a 6" core through the entire seam. The preliminary analysis of the sample on an as-received basis is:

carbon:	63-64%
hydrogen:	4-5%
sulfur:	0.4-0.6%
moisture:	28-30%
ash:	6-7%

Due to the high moisture content of this sample, it is being offered only in -20 mesh size, in 5 and 10 gram ampoules.

3. The third sample is a high volatile bituminous coal, from the Illinois #6 or Herrin seam, and was collected about 60 miles southeast of St. Louis in December, 1985. The 55 gallon drums

used for transporting the samples were taken into the mine since the seam thickness was about 7'. The preliminary analytical values on this coal (on an as-received basis) are:

carbon:	61-62%
hydrogen:	4-5%
sulfur:	4-5%
ash:	15-16
moisture:	9-10

This material is being offered in 5 grams of -100 mesh and 10 grams of -20 mesh coal.

4. The fourth coal is a Pittsburgh #8 seam sample from about 60 miles south and west of Pittsburgh, Pennsylvania. This was collected in March, 1986. The seam was about 6' thick at the collection point. The sample drums were taken into the mine to facilitate the loading and purging of the drums on the surface. This sample is being offered in ampoules of 5 grams of -100 mesh and 10 grams of -20 mesh material. The preliminary analytical data for the as-received samples were not available at the time of the writing.

SAMPLE AVAILABILITY

Samples are available to researchers upon completion of an order form. These forms are available from the Program Manager, Dr. Karl S. Vorres, at the address given in the heading, or by calling (312) 972-7374, or FTS 972-7374. The order forms accompany the periodic Product Announcements. These announcements briefly describe the sample in terms of its source and type of coal. When available, initial analytical data is included. Individuals may request that they be placed on the mailing list to receive these announcements by writing the Program Manager, indicating their address, telephone number and research interests. Upon receipt of the order and payment in the Assistant Controller's office, the orders are transmitted to the Program Manager for packing and shipping. A nominal replacement charge, currently $1.60 per gram, is made for the samples. An additional charge is made for foreign shipment to defray the added shipping charges. Samples are normally sent by United Parcel Service. Samples are shipped in specially designed corrugated cardboard cartons with foam padding above and below the ampoules, and an air space around the outside of them.

Samples are available in reasonable quantities. The Program Manager reserves the right to limit quantities in order to provide a continuing supply to all workers in the field over a decade or more.

USERS ADVISORY COMMITTEE

A Users Advisory Committee serves to provide a range of comments and suggestions on the Program to maximize its value to the users community. The individuals come from a range of backgrounds to

permit a variety of input on the many aspects of the sequence of events from the sample selection through distribution. The initial Committee included: Drs. Blaine Cecil of the U. S. Geological Survey, Marvin Poutsma of Oak Ridge National Laboratory, Ronald Pugmire of the University of Utah, William Spackman of Pennsylvania State University, Irving Wender of the University of Pittsburgh, Randall Winans and John Young of Argonne National Laboratory. The committee does rotate after a period of time. Newcomers to the group are: John Larsen of Lehigh University and Leon Stock of the University of Chicago. The Program Manager is deeply appreciative of the counsel and suggestions given by this group.

CURRENT RESEARCH

A wide range of programs is making use of the samples. Some examples include: new methods for direct determination of oxygen and organic sulfur in the coal matter, new methods of characterization using inverse chromatography, solvent swelling studies, reactions of organic sulfur species in the coal. A number of proposals to different funding agencies have specified that the investigators were planning to use these samples in their work.

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