THE EIGHT COALS

IN THE

ARGONNE PREMIUM COAL SAMPLE PROGRAM*

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ABSTRACT

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 resinite rich, an inertinite rich and a Pittsburgh #8 coking coal. The coals were selected on the basis of C, H, S and O content as well as maceral content and geological age. Ampoules are typically available in 5 grams of -100 mesh or 10 grams of -20 mesh material. Some analytical information is available. The methods of selection, collection, transportation, processing, packaging, distribution and characterization are summarized.

INTRODUCTION

The Premium Coal Sample Program is intended to provide the basic coal research community with the best quality samples of a limited number (8) of coals for basic research. The availability of the ampoules is the result of the cooperation of many individuals within a number of organizations whose efforts made the high quality of the samples possible.

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 have been mined, transported, processed into the desired particle and sample sizes and packaged in humid nitrogen environments as free of oxygen as possible. The need for a Premium Coal Sample Program was expressed at the Coal Sample Bank Workshop held March 27 and 28, 1981 in Atlanta, Georgia.

SELECTION OF THE COALS

Support has been provided by the Office of Basic Energy Sciences to make eight carefully selected coals available. The selection of these coals was based on those parameters which would represent significant differences among the available coals and maximize our understanding of the fundamental properties of coal. A cluster analysis was carried out to establish desirable samples in terms of the significant compositional parameters, C, H, O and S. Paul Neill used data from the Penn State data base on over 200 coals which had been described as representing channel samples. These were also representative of a variety of coal ranks and compositions. The cluster analysis resulted, using increasing carbon and decreasing oxygen on one axis and hydrogen and sulfur content on the other, in identification of compositional characteristics for eight coal samples. These were used to indicate a set of initial sample choices.

The sample characteristics from the cluster analysis are indicated in Table I.

Table I. The Composition Characteristics of The Eight Coals Selected in the Cluster Analysis.[a,b] (Moisture, ash-free basis)

Group	% Carbon	% Hydrogen	% Sulfur	% Oxygen
1	73.6 (1.5)	4.9 (.2)	0.5 (.2)	20.0 (1.5)
2	74.5 (1.9)	5.6 (.5)	1.1 (.4)	17.5 (2.0)
3	79.0 (1.7)	5.5 (.4)	4.4 (.3)	10.0 (1.5)
4	79.2 (2.0)	5.6 (.4)	0.9 (.6)	12.8 (2.0)
5	82.7 (2.0)	5.7 (.4)	1.3 (.6)	8.1 (1.8)
6	85.4 (1.4)	5.4 (.3)	0.8 (.3)	6.8 (1.4)
7	89.6 (1.0)	4.9 (.3)	0.8 (.3)	3.2 (1.0)
8	91.3 (0.5)	4.3 (.2)	0.7 (.2)	2.3 (0.6)

a) Oxygen is by difference
b) Values in () represent + or - values.

The list was then examined to consider the variety of maceral contents which exist in U. S. coals. There are three important maceral types, vitrinite, liptinite and inertinite. Vitrinite is the major maceral type in U. S. coals, accounting for about 85% of the total. Liptinites are hydrogen-rich macerals and inertinites are hydrogen deficient. Variations in hydrogen content can be achieved by seeking coal samples rich in certain of the liptinites or inertinites. The liptinites include resinite, from the waxy parts of plants, and sporinite derived from plant spores.

With help from the U. S. Geological Survey, individual coals were identified for collection. Details of the samples are given at the end of this paper. Samples #1,2,3,5 and 8 were selected to give a range of compositional parameters, primarily carbon,

hydrogen and oxygen which vary with the degree of coalification of the sample. In addition #3 was selected to provide the sample with a relatively high sulfur content. Coal #4 was also selected for its known coking properties, coal #6 was selected for high resinite and #7 for sporinite and inertinite contents.

COLLECTION OF THE COALS

The collection of coal samples began by identifying the potential sources of coal samples and seeking permission to acquire the samples. The U. S. Geological Survey provided at least one staff geologist at each site for supervision of the actual collection of the sample and to document the seam for later description in USGS Circulars to provide a permanent, referenceable description of the sample.

For a typical underground sample, the mine operator met with the USGS, sample collection crew, truck driver and Program Manager in advance of the sample collection to discuss details of the collection. On the morning of the collection, the coal seam face was freshly exposed to provide a block of coal the thickness of the coal seam, about one foot wide and long enough to provide for the one ton sample. The seam was exposed with a continuous miner, and a roof bolter immediately followed to secure the seam roof. The loose coal was scraped away from the sample block, and several layers of plastic sheets were placed on the mine floor. After measuring and calculating the amount needed for the sample, the block was marked by the USGS representative. The sample was then removed by the collection crew with hand picks from the roof to the floor to provide a channel type sample. Partings over an inch thick were discarded. Particular care was taken to avoid contamination of the sample with material nearby. When the seam thickness exceeded four feet, stainless steel drums were taken into the mine for sample collection. Alternatively, double plastic bags were used to transport the coal to the surface. In all cases, the coals were loaded into stainless steel drums from 1/2 to 5 hours after collection and then purged with argon.

The subbituminous sample was collected as a 6" core sample from the Wyodak seam that was about 120' thick at the sample site. The drilling contractor also obtained two additional 3" cores, one for the USGS, and the other for long term storage of the sample. All cores were rinsed with distilled, deaerated water immediately after they were released from the core barrel and loaded into the stainless steel drums for shipment.

The lignite sample was obtained as a series of 3" core-type samples drilled through the seam at about 20' intervals over a freshly exposed top surface. Cold wether limited reactions at the surface. This approach provided channel type samples representative of a sample area of about one acre. The samples were also quickly loaded into the drums and purged.

TRANSPORTATION OF THE SAMPLE

The samples were always moved in the same truck which was operated by the same driver. The truck was loaded with stainless steel drums, argon cylinders and tools at ANL and then driven to the mine site. The drums were usually loaded on the truck with a forklift. The samples were purged with 99.999% argon gas using a calculated volume to reduce the oxygen concentration to below 100 ppm. The sealed drums were pressurized to about 6 psig and then transported in a refrigerated semi-trailer at temperatures of 40-45 F to prevent freezing and limit chemical reactions on the way to the processing facility at ANL. The samples arrived at the laboratory within one-half to two days of the purging, and were immediately unloaded.

PROCESSING OF THE SAMPLES

After the stainless steel drums were unloaded from the truck, they were weighed, recorded and transferred to the first airlock in the nitrogen filled processing facility. This facility is a large enclosure in two parts, each of which was constructed of sheets of aluminum and plastic windows. Seventy pairs of long rubber gloves mounted in the windows permit manipulation of the samples and equipment during the processing. The dimensions of the facility are about 5-6' wide, 13' high and the equivalent of 40' long. The two parts are separated by a sealed mixer-blender. The oxygen concentration in the facility was maintained below 100 ppm during processing of the samples. Oxygen control is part of the gas handling system design which includes a cyclone separator, high efficiency particulate filter, industrial blower and cooling coils as well as steam supply for humidity control. A part of the gas stream is passed over a palladium on alumina catalyst with a slow stream of hydrogen to react with and convert the trace amounts of oxygen to steam.

After the first airlock containing the coal drums was purged with nitrogen to less than 100 ppm oxygen, as established by analysis of the gas by the fuel cell analyzer, the drum lids were removed. The drums were moved to the hydraulic drum dumper and fastened in place. The contents of the drum were dumped into a crusher which reduced the coal particles to a size such that one dimension was no more than 1/2". The crusher discharge flowed down a chute with a variable gate to control the flow rate to a Syntron vibratory lift which raised the crushed coal to the top of the enclosure. A pulverizer feeder controlled the rate of flow to the pulverizer, which has a nominal capacity of 300 pounds per hour. Initially a 20 mesh screen was used with the hammer mill. The pulverized coal flowed down into the mixer blender which has a 2000 liter capacity, and can hold the entire one ton sample. The sample was accumulated and then thoroughly mixed in the blender. Earlier studies at the vendor's facility demonstrated that mixing was achieved in less than four minutes.

PACKAGING OF THE SAMPLES

The thoroughly mixed sample was then conveyed by means of a tubular conveyor to a filling station for five gallon "leverlock" pails or borosilicate carboys. One half of the batch was transferred in the five gallon pails back through airlocks for further pulverizing to pass a 100 mesh screen. The balance of the -20 mesh material was transferred to ampoule filling and sealing equipment. Ten grams of -20 mesh or five grams of -100 mesh material (except for the Wyoming sample) were dispensed into amber borosilicate ampoules. The ampoules were sealed with a hydrogen-oxygen flame. The gas supply was controlled with two gas mass flow controllers and set to provide for stoichiometric combustion. The oxygen levels do not change in the box due to torch operation. The sealed ampoules were removed through airlocks and then placed in storage.

STORAGE

The samples and borosilicate carboys are kept in racks in a dark 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. It is expected that about 75,000 ampoules can be provided from each sample. This quantity of ampoules can meet requests for 30 ampoules per day for the next decade and is expected to meet the needs for a substantially longer period.

DISTRIBUTION OF THE SAMPLES

Product announcements are sent out to a mailing list of individuals who have asked to be included or are active in basic coal research. Orders are placed through the Assistant Controller at ANL, and are sent to the Program Manager for filling. Each ampoule is inspected before being placed in special cardboard cartons for shipping. These cartons have foam bottom and top layers, and partitions to provide for space between the ampoules and the wall of the carton for added protection. Samples are typically sent by United Parcel Service (UPS).

Each researcher is asked to provide a short statement of the nature of the work, such as molecular structure studies by pyrolysis- mass spectrometry, so that an understanding of the different types of research activity may be obtained for the overall program. In addition the researcher is asked to provide references to public domain documents such that a bibliography of research with the samples can be made available to all researchers to further the progress of basic coal science. This bibliographic information will become available in either a printed version or through a dial-up modem.

Samples are available in 5 grams of -100 mesh or 10 grams of -20 mesh material for the first seven coals except for the Wyodak sample which is only available in the -20 mesh size. The eighth sample should become available in April 1987.

CHARACTERIZATION OF THE SAMPLES

The coal samples are being characterized for three purposes: establishing the homogeneity of the samples, characterizing the samples physically and chemically, and monitoring the stability of the samples in storage. The homogeneity of the samples was evaluated using 39 samples taken during the course of the packaging and submitting them for irradiation at the University of Illinois TRIGA reactor. The three or four most suitable radioisotopes that were produced were then counted over a period of two weeks to establish the activity per unit of mass and then compared to examine variation and trends. The characterization consisted of an analysis for a range of chemical and physical properties by up to 70 different laboratories. The list of chemical and physical analyses is given below:

Chemical analyses:
- Ultimate, C,H, S, N, O (by difference)
- Proximate: moisture, ash, volatile matter, fixed carbon
- Major and minor elements in the ash
- Equilibrium moisture
- Direct oxygen by fast neutron activation

Physical measurements and analyses:
- Petrographic
- Maceral analysis
- Ash fusion temperatures
- Gieseler plasticity

The stability in storage has been evaluated in several ways. For bituminous coals the Gieseler plasticity has been measured periodically. For all coals the gas atmosphere over the samples has been analyzed periodically.

DESCRIPTION OF THE SAMPLES

1. The first is an Upper Freeport seam medium volatile bituminous sample collected near Homer City, Pennsylvania (Indiana County) in January 1985. The seam was 4' thick at the collection point. Sample characterization provides the following values (prelim. as received basis):

carbon:	75.7%	ash:	13.1%
hydrogen:	4.82%	moisture:	0.85%
total sulfur:	2.42%		

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

carbon:	65.4%	moisture:	28.7%
hydrogen:	5.18%	ash:	9.8%
sulfur:	0.66%		

Due to the high moisture content of this sample it is being offered only in the ampoules containing 10 grams of -20 mesh.

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 (St. Clair county) in December, 1985. The 55 gallon drums were taken into the mine since the seam was about 7' thick at the collection site. The preliminary analytical values, on a dry basis (except moisture) are:

carbon:	65.2%	moisture:	8.8%
hydrogen:	4.82%	ash:	16.2%
sulfur:	4.6%		

4. The fourth coal, a Pittsburgh #8 seam, was collected about 60 miles south and west of Pittsburgh in Greene County, Pennsylvania in March 1986. This seam was about 6' thick at the collection site. The sample drums were again taken into the mine to facilitate the loading of the drums at the surface. The preliminary analytical data on an as-received basis (in %) follow:

carbon:	75.6	moisture:	1.8
hydrogen:	5.34	ash:	9.3
total sulfur:	2.1		

5. The fifth sample is a low volatile bituminous from the Pocahontas #3 seam, collected in Buchanan County, Virginia in June, 1986. The seam was about 6' thick at the collection site, and the drums were taken into the mine. The preliminary analytical data on an as-received (in %) basis are:

carbon:	85.4	moisture:	0.7
hydrogen:	4.5	ash:	4.9
total sulfur:	0.6		

6. The sixth sample is a Utah Blind Canyon seam sample collected in Emery County about 150 miles south and east of Salt Lake City, Utah. The seam was about 7' thick at the collection site. Some preliminary analytical data on an as-received basis are:

carbon: 74.1% hydrogen 5.7% resinite 11%

7. The seventh sample is from the Lewiston-Stockton seam of the Kanawha formation, collected about 20 miles east of Charleston, West Virginia (Kanawha County). The seam was about 6' thick at the collection site. Preliminary data indicate that this sample contains about 73% vitrinite, 16% inertinite and 11% exinites, almost all of which is sporinite, 65.1% carbon and 4.38% hydrogen.

8. The eighth sample is from the Beulah-Zap seam collected in Mercer county, North Dakota about 8 miles northwest of Beulah. The seam was about 18 feet thick at the collection site. Collection was done by accumulating about 50 of 3" sample cores spaced about 20' apart in each direction. These were immediately loaded into the drums.

ADVISORY BOARD

A group of seven prominent coal researchers is involved with the Program in an advisory capacity to provide the Program Manager with advice and counsel on a range of issues. These people met on an annual basis and also provided advice informally at other meetings and through phone conversations. Those serving at present and in the past include: C. Blaine Cecil, USGS; John Larsen, Lehigh University; Marvin Poutsma,ORNL, Ronald Pugmire, University of Utah; William Spackman, Pennsylvania State University; Leon Stock, University of Chicago; Irving Wender, University of Pittsburgh; Randall Winans, Argonne National Laboratory, John Young, Argonne National Laboratory

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