STATUS OF THE PREMIUM COAL SAMPLE PROGRAM AT THE ARGONNE NATIONAL LABORATORY

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PURPOSE OF THE PREMIUM COAL SAMPLE PROGRAM

The purpose of the Premium Coal Sample Program is to provide the 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 remain in a pristine condition over long periods of time.

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.

WHAT A PREMIUM SAMPLE IS

A premium coal sample has been specially selected, processed and stored to keep it as close to its original condition as possible. Specifically: contact with oxygen has been minimized at all stages from mining, transport and processing in a nitrogen filled facility to sealing in amber colored glass vials. Relative humidity and temperature are controlled in the processing facility to maintain the equilibrium moisture of the original coal. Uniformity of samples is achieved by processing about 750 kg of coal in a single batch, mixing thoroughly in a special blender, and finishing with a spinning riffler to assure well-mixed samples. Activation analyses have confirmed the thoroughness of the mixing. Stability of the samples is maximized by sealing in amber-colored glass with a fuel-rich hydrogen-oxygen flame. Secure, long-term supplies result from an initial production of 10,000 five gram ampoules and 5,000 ten gram ampoules with 50 five gallon sealed glass carboys in reserve for future ampoule production from each metric ton sample of coal. Some special needs can be met from lumps stored in argon in two reserve 55 gallon drums, and two 15 gallon drums as part of the original sample. A separate nitrogen filled glove box will be used for processing these requests.

SELECTION, SAMPLE COLLECTION, AND TRANSPORT

Initially the coals have been selected to cover a wide range of chemical composition. The samples will include low-, medium- and high-volatile bituminous coals as well as lignite and sub-bituminous. These samples are channel-type samples, representing a uniform cross section of the seam from top to bottom. Sample collection, under the supervision of coal geologists from the U. S. Geological Survey, involves removal of coal up to 6" lumps from a freshly exposed face to special double plastic bags, transfer to stainless steel drums in a refrigerated semi-trailer at the surface, purging of samples in the drums with argon, and immediate transport to the processing facility at Argonne National Laboratory (ANL). A careful description of the geology of

the sample area and location will be prepared and available as a referenceable document.

SAMPLE PROCESSING

The processing facility is a large glove box made of aluminum panels about 12' tall, 4-5' wide and about 40' long. Clear plastic windows containing 70 pairs of rubber gloves permit observation and manipulation of the sample or equipment. During processing the box is filled with nitrogen and the oxygen concentration is kept at or below 100 ppm. The humidity is kept as high as possible but low enough to avoid condensation on the windows during operation. At the processing facility, the stainless steel drums are weighed and loaded into an airlock, which is then purged with nitrogen. The drums are emptied by means of a hydraulic drum dumper into a crusher which reduces the particle size to -1/2". The sample is then pulverized in an impact mill to obtain -20 mesh material. The pulverized material is collected in a nitrogen filled mixer-blender selected for gentle but thorough mixing. After thorough mixing the pulverized coal is conveyed to a spinning riffler and sealed in 10 gram ampoules and 5 gallon glass carboys or special 5 gallon transfer containers. The contents of the transfer containers are then recycled to the pulverizer and crushed to pass a 100 mesh screen. After thorough blending this material is conveyed to the packaging unit for sealing in 5 gram amber colored ampoules and 5 gallon borosilicate glass carboys. Figure 1 is a block diagram of the coal sample preparation.

CHARACTERIZATION

The coals are analyzed for three purposes: (1) homogeneity testing, (2) characterization, and (3) stability monitoring. Results are available for each coal in the form of a printed sample announcement. Requests to be placed on a mailing list should be sent to the author. The requestor should include mailing address, telephone number, and research interests.

Homogeneity testing includes sampling the product flow into carboys and ampoules throughout the processing to obtain 39 representative samples. These are placed in polyethylene containers for irradiation at the the University of Illinois TRIGA reactor. The samples are counted at ANL to monitor the Na, K and As activities. Other techniques are being evaluated to complement these measurements. After the results are analyzed and found to be satisfactory an announcement of the availability of samples is sent to individuals on the mailing list.

Characterization includes the efforts of over 70 different laboratories to establish physical and chemical properties of the samples. This number is desirable to permit a statistical analysis of the results. The analyses will include proximate, ultimate, calorific values, sulfur forms, equilibrium moisture, maceral analysis, Gieseler plasticity for the bituminous coals, and mineral matter major elements among others. Round robin analyses have been organized.

A variety of stability monitoring tests are used including evolved gas analysis and slurry pH. The gas analysis includes determinations of oxygen and light hydrocarbons to follow any possible diffusion of gas into the ampoules and diffusion of volatiles from the sample. The slurry pH monitors

the change in pH of the filtrate from a slurry of sample with distilled, deaerated water to monitor oxidation and release of products of pyrite decomposition. Sulfate ion concentration is also determined. In addition the bituminous samples will be monitored by repetitive Gieseler plasticity analyses.

AVAILABILITY AND DISTRIBUTION

Samples are made available to research personnel at a nominal replacement cost. A special glove box filled with nitrogen is available to transfer contents of ampoules to special sample holders on request. Also, a very limited quantity of lump coal, stored under similar inert conditions will be available on special request for special physical property measurements. The processing facility can be made available for occasional processing of special samples.

Orders are placed on the forms sent with the announcement of availability of the samples with the author. Prepayment is requested. Samples are available in packages of 6 ampoules of one size. Special cartons are used to assure safe delivery. Samples are shipped by United Parcel Service (UPS).

FIRST SAMPLE

The first sample, a medium volatile bituminous coal, was collected from the Lucerne #6 mine, owned by the Rochester and Pittsburgh Mining Company of Indiana, Pennsylvania. The sample was collected from the Upper Freeport seam near Homer City, Pennsylvania. A wedge shaped block was exposed by a continuous miner and used for the channel type sample.

INFORMATION ON SAMPLES

Each recipient of samples is asked to provide either a literature reference to papers in widely circulated journals, or a copy of less widely circulated public reports and papers, to be shared with other users of the samples. Listings of these references will be available on request to the author (phone 312-972-7374) either in printed versions or via computer terminal. The Premium Coal Sample Program expects to work with other coal sample programs in providing samples and sharing information.

Following the reports from the use of of a number of samples, workshops are planned to facilitate sharing research results and to foster basic understanding of the chemistry and physical properties of the coal. The first is expected to be scheduled in 1987 at ANL.

USERS ADVISORY COMMITTEE

A Users Advisory Committee provides useful suggehtions to the Program Manager. This group includes Drs.: Blaine Cecil, U. S. Geological Survey; Marvin Poutsma, Oak Ridge National Laboratory; Ronald Pugmire, University of Utah; William Spackman, Pennsylvania State University; Irving Wender, University of Pittsburgh; Randall Winans, Argonne National Laboratory; John Young, Argonne National Laboratory.

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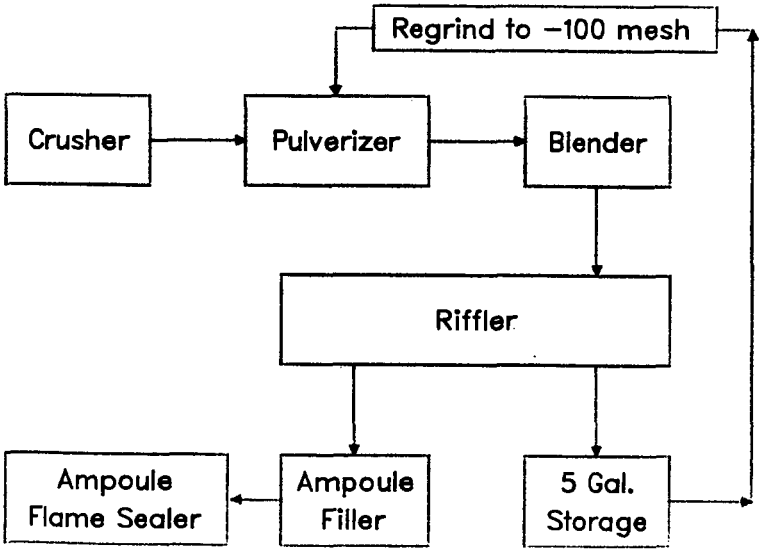
FIGURE 1

COAL SAMPLE PREPARATION