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MAINTENANCE OF THE COAL SAMPLE BANK AND DATABASE**Quarterly Technical Progress Report****Contract Number DE-AC22-93PC93051****The Pennsylvania State University
Coal and Organic Petrology Laboratories****Reporting Period: 9/29/93 - 12/28/93**

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Quarterly Technical Progress Report: 9/29/93 - 12/28/93

Objectives

This five year project is intended to ensure the availability of well-characterized, high-quality coal samples for public and private coal research. It continues support of the DOE Coal Sample Bank and Database at The Pennsylvania State University. Thirty coal samples will be collected, processed, packaged, and analyzed, and a resulting database will be maintained. These samples and data, as well as 26 samples collected under previous contracts, will be distributed to DOE contractors and others performing coal research.

Samples will be chosen to maintain a sample bank of 56 coals representing the major U.S. coal fields and a variety of coal ranks and compositions. In addition to standard analyses, liquefaction tests and organic geochemical analyses will be performed. The samples will be stored to minimize deterioration, and will be monitored annually by proximate, sulfur forms, and gaseous oxygen analysis to evaluate their condition. These samples will be used for additional research on the influence of changes in surface chemistry on surface properties, the influence of sample deterioration on liquefaction properties, and the mechanisms of coal oxidation.

Summary of Technical Progress

Task 1A. Initiation of Sample Bank Activities

Samples and data were available from the first day of the contract; their distribution continued uninterrupted following the unfunded period after the previous contract. The sample inventory records and procedures for storage, selection, retrieval and distribution were in place and fully functional. This task is considered complete and will not be reported in the future.

Task 1B. Storage and Inventory of Samples

All samples supported by the contract are stored in containers purged with argon before sealing; container locations are tracked by a computer inventory program. PSOC-series samples are stored in drums or buckets at the Coal Sample Bank building, or in cans in the Coal and Organic Petrology Laboratories. All DECS-series samples are stored in foil laminate bags.

During the quarter, the new refrigerated coal storage room became available. This facility will hold existing DECS samples as well as all DECS samples collected under the current five-year contract. All containers of existing DECS samples were moved into the room and the inventory records were updated. The exceptions are 6 300 g (2/3 lb) bags of each DECS sample which will be stored under refrigeration in the Coal and Organic Petrology Laboratories for more ready access.

Storage and inventory of sample containers is expected to continue routinely through the next quarter.

Task 1C. Monitoring of Sample Quality

One 300 g (2/3 lb) bag of each DECS sample will be analyzed annually to monitor sample condition. No monitoring analyses were performed during this quarter. We anticipate that the first annual quality monitoring program will begin during the next quarter.

The soft package sampling arm to be used with the polarographic oxygen analyzer was purchased and successfully tested.

Task 1D. Collection and Processing of Replacement Samples

Arrangements were begun and permission from DOE was obtained for the collection of a sample of the Pittsburgh seam (hvAb) from western Pennsylvania, but the sample was not collected before the end of the quarter. We plan to collect and process this sample early in the next quarter.

In order to plan sample collection trips, a list of the other 29 samples to be collected must be established. The contract contains a tentative list, but changes (such as inclusion of samples coordinated with HRI projects) have been suggested in past discussions with DOE. During the quarter, specific requirements or changes to the list were requested from DOE. Information on possible HRI samples was received but the complete list of samples has not yet been finalized.

Task 1E. Basic Characterization of Coal Samples

No new samples were available for characterization during the quarter. Characterization of the first new sample (Pittsburgh seam, see Task 1D above) is anticipated for the next quarter.

Task 1F. Liquefaction Testing

A procedure for a standardized liquefaction test and product workup was established, but still requires approval of DOE. In this procedure, samples are first freeze dried. Tubing-bomb reactor tests are to be performed in duplicate with 5g coal and 5g phenanthrene per reactor, run in a sand bath at 350°C for 30 min. The product workup is to include percentages of total conversion, volatile hydrocarbons, asphaltenes, preasphaltenes, insoluble organic matter, and ash.

Thirteen existing samples will be tested beginning in the next quarter. The new Pittsburgh seam sample will also be tested as soon as it is processed.

This task includes a study to monitor liquefaction behavior in relation to sample storage. The study will begin with two samples collected in the first contract year.

Task 1G. Investigation of Changes in Surface Chemistry During Storage

The general objective of this task is to measure the changes in functional group concentration as a function of duration and type of storage method using reflectance-mode FT-i.r. and direct oxygen determination using electron optical techniques. Also,

microscopic measurements of fluorescence alteration and intensity will be employed to determine the extent of surface alteration during coal storage.

The Pittsburgh seam (see Task 1D) was selected as one of 8 coals to be studied under this task and, although the sample was not collected during this quarter, a general procedure was established for sample processing. It was decided that both vitrinite concentrates and bulk samples from eight fresh bituminous coals would be used in the experimental program. Vitrinite bands will be handpicked in a nitrogen-filled glovebox in an attempt to limit their exposure to atmospheric oxygen, to drastic changes in temperature and relative humidity, and exposure to direct sunlight. From these samples, base levels of near-surface oxygen chemistry and concentration will be obtained to compare with measurements of fluorescence intensity and alteration. Subsamples of each bulk coal will be stored under argon in foil bags and will be exposed to the atmosphere. Using the same analytical procedures, these samples will be tested for changes in surface chemistry following two periods of storage (4-6 months and 12 months).

Task 1H. Organic Geochemistry

Procedures for organic geochemical characterization were discussed and established internally during the quarter. Characterization of three to four existing samples will begin in the next quarter, and new samples will be characterized as they are collected. The existing samples to be characterized are:

DECS-2	hvCb	Illinois #6 seam	Illinois
DECS-8	subC	Smith-Roland ("Wyodak") seam	Wyoming
DECS-12	hvAb	Pittsburgh seam	Pennsylvania
DECS-17	hvAb	Blind Canyon seam	Utah

Task 1I. Distribution of Coal Samples to Users

During the period 9/29/93-12/28/93 a total of 28 samples (25 DOE Sample Bank samples and 3 other Penn State samples) of various sizes in 42 containers were distributed. Twenty of these samples were provided to DOE contractors. No bags of DECS-17 (for the DOE dispersed catalyst study) were distributed during the quarter. See Task 2C for a list of sample and data recipients. This task will continue in the same manner through the next quarter.

Task 2A. Programming

No significant programming was performed during the quarter. Programming required for the creation of the new data printout formats is expected to be a major effort in the next quarter.

The new printout format will be printed in portrait rather than landscape orientation, using larger fonts than the previous format. Following extensive discussions, many improvements will be instituted. Analyses will be expressed in SI metric units followed by their U.S. customary equivalents. A completely new cover sheet will be included. The same categories of data will be included in the same order as in the previous five-page format; these are: Sample Location, Sample History, Sample Information, Proximate Analysis, Sulfur Forms, Moisture, Ultimate Analysis, Elemental Analysis, Calorific Value,

Atomic Ratios, Rank Calculations, Maceral Composition, Reflectance Data, Caking and Mechanical Properties, Inorganic Element Analysis, Mineralogical Analysis (where available), and Ash Fusion Temperatures. A new one-page format will also be developed.

Task 2B. Data Entry

No data entry was required during the quarter because no new samples were collected or analyzed. In the next quarter, collection and analysis of the Pittsburgh seam sample is expected to generate data which will be entered.

Task 2C. Distribution of Data to Users

During the quarter a total of 42 data printouts were distributed. In addition, 11 special data requests were fulfilled by database searches and output or creation of a data disk, resulting in distribution of limited information on 2229 samples. Preliminary requests for Sample Bank and Database information and price quotations have also been filled. This task will continue in the same manner through the next quarter.

Organizations supplied with coal samples and/or data include:

Advanced Fuel Research (2)
Buckeye Industrial Mining Company
Coal Technology Corp. (5)
Exxon Production Research
Instituto Nacional del Carbon (Spain)
Pennsylvania State University, Fuel Science Department (3)
Pennsylvania State University, Energy & Fuels Research Center (2)
PSI Technology Co.
Southern Illinois University, Geology Department
Technical University of Denmark, Lab. of Heating and Air Conditioning (Denmark)
University of Utah, Center for Microanalysis (2)
University of Utah, Chemistry Department
West Virginia University, Chemical Engineering Department

The numbers in parentheses represent multiple requests which were separately processed.

References/ Publications

No manuscripts supported by the contract were published during the quarter.

Penn State personnel collaborated on a paper to be presented at the American Chemical Society meeting in March: Perspectives on the Status of Coal Research from Shipments of Samples, by K. S. Vorres, Carl W. Kruse, Koen A. Nater, David C. Glick and Alan Davis.

Conference Record

No publications were acquired at contract-supported conferences during the quarter.

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