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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: 4/01/96 - 6/30/96

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Anticipated Completion Date: 9/28/98

Government Award: \$156,000. for current contract year

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Quarterly Technical Progress Report: 4/01/96 - 6/30/96

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. Eleven coal samples will be collected, processed, packaged, and analyzed, and a resulting database will be maintained. These samples and data, as well as 45 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 10 samples will be monitored annually by proximate, sulfur forms, and gaseous oxygen analysis to evaluate their condition.

This report reflects the level of effort described in the revised work statement submitted to DOE on February 2, 1996, setting out a reduction in effort following a cut in the level of funding of the project.

Summary of Technical Progress

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 under refrigeration in foil laminate bags.

During the quarter, samples were distributed and inventory records were updated. Storage and inventory of sample containers is expected to continue routinely through the next quarter.

Task 1C. Monitoring of Sample Quality

It was planned that one 300 g (2/3 lb) bag of each DECS sample would be analyzed annually to monitor sample condition. This Task has been reduced in scope, to 10 samples per year, because of reduction in funding. This year's results were presented in the last report. A repeat analysis for the calorific value of sample DECS-8 became available after that report was submitted. The Appendix of this report contains the complete results for DECS-8 including the changed result for calorific value. Effort in the related study of liquefaction behavior (under Task 1F) over time has also been reduced.

Task 1D. Collection and Processing of Replacement Samples

Collection and processing of 11 samples, as described in the Revised Statement of Work (Feb. 2, 1996) has been completed.

Six DECS samples collected under a prior contract were processed to minus 60 mesh (-0.25 mm) during the quarter. Such processing will continue in succeeding quarters.

Task 1E. Basic Characterization of Coal Samples

All basic characterization of samples planned for collection under the contract has been completed.

Task 1F. Liquefaction Testing

In this Task, appropriate samples will be subjected to a standardized liquefaction test using tetralin and subsequent product workup. Work in this Task was completed during the quarter, although the total number of samples to be tested has been reduced following a cut in the level of funding. Table 1 shows results for the final 10 samples analyzed.

Table 1. Conversion of DECS samples in a standardized liquefaction test using tetralin, with detailed gas composition (average of two runs, except gas components based on one run; see Task 1F).

sample	conversion, percent of dry ash-free coal											
	total	asph.	pre-asph.	oil	gas							
					total	c1	c2	c3	c4	c5	c1-c5	other
DECS-4	48.5	6.1	11.7	7.6	23.1	0.5	0.4	0.2			1.1	22.0
DECS-5	45.0	6.9	27.2	6.8	4.1	0.5	0.4	0.1			1.0	3.1
DECS-6	30.1	7.9	7.7	7.0	7.5	0.5	0.2	0.1			0.8	6.7
DECS-7	44.6	8.3	4.9	10.3	21.1	1.1	0.6	0.2			1.9	19.2
DECS-9	35.0	6.1	9.1	4.2	15.6	0.6	0.4	0.1			1.1	14.5
DECS-10	35.5	7.2	8.6	4.7	15.0	0.7	0.3	0.2			1.2	13.8
DECS-14	44.9	7.4	15.7	7.7	14.1	1.4	0.9	0.2			2.5	11.6
DECS-15	43.5	7.8	18.7	8.6	8.4	1.1	0.5	0.1			1.7	6.7
DECS-16	51.9	19.2	9.8	13.2	9.7	1.5	0.8	0.3			2.6	7.1
DECS-22	42.0	8.2	14.7	8.9	10.2	1.2	0.8	0.2			2.2	8.0

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

This Task has been discontinued.

Task 1H. Organic Geochemistry

Nuclear Magnetic Resonance (NMR) Spectroscopy

Dried powdered samples of coal were placed in the bullet-type rotor of a Chemagnetics, Inc. M-100 NMR spectrometer and analyzed according to the cross-polarization, magic-angle spinning (CPMAS) and dipolar dephasing with magic angle spinning (DDMAS) procedures outlined by Hatcher (1988). Approximately 10,000 transients having a contact time of 1 ms and a cycle time of 1 s were acquired in 0.5K of data, zero-filled to 4K. The chemical shifts were referenced to hexamethylbenzene. For DDMAS, approximately 8000 transients with a cycle time of 1 s were used at each of 10 dephasing times.

CPMAS analyses planned for this contract have been completed.

DDMAS measurements on the final set of samples are nearly complete.

Bloch decay experiments planned for this contract have been completed.

Task 1I. Distribution of Coal Samples to Users

During the period 4/01/96 - 6/30/96 a total of 65 samples (31 DOE Sample Bank samples and 34 other Penn State samples) of various sizes in 103 containers were distributed. In addition, 16 30g bags of special sample DECS-17 were distributed. 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

Only minor programming was undertaken during the quarter. See also the discussion of the world wide web site under 'Distribution of Data to Users.'

Task 2B. Data Entry

Entry of basic characterization data for the 11 samples collected under the contract has been completed.

Task 2C. Distribution of Data to Users

During the quarter a total of 84 data printouts were distributed. In addition, 16 special data requests were filled by database searches and printed output or creation of a data disk, resulting in distribution of information on 3826 samples. Preliminary requests

for Sample Bank and Database information have been filled. This task will continue in the same manner through the next quarter.

Organizations supplied with coal samples and/or data include:

Auburn University, Chemical Engineering Department
 Cominco, Inc. (Canada)
 GeoMet, Inc.
 Little Bear Laboratories, Montana
 National Institute of Standards and Technology
 National Mining Association (2)
 New York University Medical Center, Nelson Institute of Environmental Medicine (2)
 Northeastern University, Mechanical Engineering Department
 Original Fuels Co.
 Pennsylvania State University, Coal and Organic Petrology Laboratories
 Pennsylvania State University, Energy and Fuels Research Center
 Pennsylvania State University, Fuel Science Department (4)
 Ralph Gray Services
 Rust College, Science and Mathematics Division
 Salem Tool, Inc.
 SRI International
 Mr. James Truman
 Università degli studi di Napoli Federico II, Chemical Engineering Department (Italy)
 University of British Columbia, Earth and Ocean Sciences Department (Canada)
 University of Delaware, Chemical Engineering Department
 University of Texas of the Permian Basin, Chemistry Faculty
 University of Utah
 Unocal Science and Technology Division
 Utah Geological Survey

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

In order to further publicize the availability of the Coal Sample Bank and Database, a world wide web site (<http://www.ems.psu.edu/COPL/>) was made publicly available during the quarter. A searchable subset of the Penn State Coal Database (including DECS samples) is operational on the world wide web and is undergoing final improvements before being announced.

References

- Hatcher, P.G., 1988. Dipolar-dephasing ^{13}C NMR studies of decomposed wood and coalified xylem tissue: evidence for chemical structural changes associated with defunctionalization of lignin structural units during coalification. *Energy & Fuels*, v. 2, pp 40-58.

APPENDIX. Monitoring of DOE Sample Bank Coals to Evaluate Sample Condition after Storage

DECS-8	Smith-Roland seam		subC		Ro max= 0.37	
FOIL BAG	original analysis	27 of 256	127 of 256	138 of 256	144 of 256	159 of 256
particle size stored		-20 mesh	-20 mesh	-20 mesh	-20 mesh	-20 mesh
note						
date sealed		10/ 22/ 90	10/ 22/ 90	10/ 22/ 90	10/ 22/ 90	10/ 22/ 96
date analyzed	10/ 30/ 90	10/ 21/ 92	6/ 03/ 93	3/ 23/ 94	5/ 09/ 95	2/ 13/ 96
PROXIMATE						
% moisture	28.4	28.3	28.6	28.1	28.6	28.7
% ash (dry basis)	13.8	12.8	13.1	13.0	13.6	13.0
SULFUR FORMS (dry %)						
total sulfur	0.73	0.77	0.81	0.95	0.87	0.80
sulfate	0.02	0.01	0.01	0.00	0.01	0.01
pyritic	0.28	0.20	0.22	0.22	0.27	0.25
organic	0.43	0.56	0.58	0.72	0.58	0.54
CALORIFIC VALUE (dry)						(a)
Btu/lb	11168	11275	11287	11244	11210	11263
GASEOUS OXYGEN						
% in bag atmosphere				0.08	0.14	0.28
date analyzed				3/ 08/ 94	5/ 09/ 95	1/ 18/ 96

(a) result from repeat analysis 4/29/96, change from Quarterly Technical Progress Report dated 4/25/96.