DOE/PC/93051--T6

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: 4/01/95 - 6/30/95

Report Date: 11/20/95

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Quarterly Technical Progress Report: 4/01/95 - 6/30/95

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.

A revised work statement has been submitted to DOE, 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. Newly collected samples were added to the inventory records. 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 because of reduction in funding.

Ten samples were selected for monitoring. During the previous quarter, samples DECS-23 and DECS-24 were subjected to the usual quality monitoring analyses). Analyses of the other eight samples were performed during this quarter. The results, presented in the Appendix, show that the foil laminate bags continue to preserve DECS samples well in comparison to storage containers used previously. Gieseler fluidity is one of the most sensitive indicators; the initial loss in fluidity

exhibited by some samples may result from factors other than oxidation (degassing, physical relaxation, etc.). Low fluidity results from 1993 may be related to changes to the equipment made at that time.

Task 1D. Collection and Processing of Replacement Samples

Four samples, DECS-29 through -32, were collected from the Central Appalachians during the quarter, following discussion and approval by DOE.

DECS-29 is a whole-seam channel sample of the hvAb Upper Banner #3 seam collected from a surface mine in Dickenson County, Virginia, on June 19, 1995. At this site the Upper Banner is split into four separate seams, numbered 1 through 4 from bottom to top; they are exposed in separate pits in the mine. This channel sample was collected from the #3 split; separate block samples were also collected. It was later determined that the coal was somewhat oxidized prior to collection. Initial processing of this sample (which includes opening the argon-filled drums of coal, crushing, homogenization, partial splitting, and sealing all splits under argon in foil laminate bags) was accomplished on June 28.

DECS-30 is a whole-seam channel sample of the mvb Splash Dam seam from Buchanan County, Virginia. The sample was collected on June 20 from an underground mine; the coal had been exposed less than two days prior to collection. Separate block samples were also collected. Initial processing was performed on June 29.

DECS-31 is a whole-seam channel sample of the Pond Creek seam collected on June 21 from an underground mine in Pike County, Kentucky. The coal had been exposed less than two days prior to collection; separate block samples were also collected. Initial processing was performed on June 27.

DECS-32 is a whole-seam channel sample of the Stockton-Lewiston seam collected on June 23 from an underground mine in Kanawha County, West Virginia. The coal had been exposed approximately 20 hours prior to collection of the sample. Separate block samples were also collected. Initial processing was accomplished on June 26.

Collection of a fifth sample is planned for the next quarter.

Task 1E. Basic Characterization of Coal Samples

Characterization of samples DECS-29 through -32 by Gieseler plastometry, free-swelling index and alkali extraction was begun immediately after their initial processing. This coincided with performance of the standardized liquefaction test (except for the mvb DECS-30), and preparation of sample splits for other methods of basic characterization, which will follow immediately at the beginning of the next quarter.

Task 1F. Liquefaction Testing

In this Task, appropriate samples will be subjected to a standardized liquefaction test using tetralin and subsequent product workup.

The new samples were processed promptly and liquefaction testing began on the fresh samples. Because of its high rank (mvb), DECS-30 was not tested. Product workup on the liquefaction components will be performed early in the next quarter.

During the quarter, liquefaction runs and concurrent quality monitoring were performed on stored splits of samples DECS-23 and -24 in the year following their collection. Results of the liquefaction runs will be presented in a subsequent report.

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.

Further work on this Task has been suspended pending decisions on funding.

Task 1H. Organic Geochemistry

Nuclear Magnetic Resonance (NMR) Spectroscopy

Analysis of the newly collected samples will be performed in the next quarter.

Pyrolysis - Gas Chromatography - Mass Spectrometry

Analysis of the newly collected samples will be performed after liquefaction and NMR analyses have been completed.

Task 1I. Distribution of Coal Samples to Users

During the period 4/01/95 - 6/30/95 a total of 110 samples (94 DOE Sample Bank samples and 16 other Penn State samples) of various sizes in 125 containers 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

Some changes to the sample container inventory program were undertaken during the quarter.

Task 2B. Data Entry

Task 2C. Distribution of Data to Users

During the quarter a total of 60 data printouts were distributed. In addition, 10 special data requests were filled by database searches and printed output or creation of a data disk, resulting in distribution of limited information on 449 samples. Preliminary requests for Sample Bank and Database information 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:

American Colloid Company
Brigham Young University, Chemical Engineering Department
Buckeye Industrial Mining Company
Clark Atlanta University, Chemistry Department
Coal Technology Corporation
Exxon Production Research Company
Northeastern University, Mechanical Engineering Department
Pennsylvania State University, Geosciences Department (4)
Pennsylvania State University, Mining Engineering Department
Pennsylvania State University, Energy and Fuels Research Center (2)
Pennsylvania State University, Fuel Science Department (7)
SRI Corporation
University of British Columbia, Pathology Department (Canada)
University of Oklahoma, Geology and Geophysics Department
University of Southern California, Chemical Engineering Department
University of Utah, Chemistry Department
University of Utah, Chemical and Fuels Engineering Department
West Virginia University, Chemical Engineering Department
West Virginia University, Physics Department

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

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APPENDIX

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

DECS-2	Illinois #6 seam	hvCb				Ro max= 0.52
FOIL BAG	original analysis	21 of 256	100 of 256	109 of 256	114 of 256 115 of 256	136 of 256
particle size stored		-20 mesh	-20 mesh	-20 mesh	-20 mesh	-20 mesh
note						
date sealed		8/ 06/ 90	8/ 06/ 90	8/ 06/ 90	8/ 06/ 90	8/ 06/ 90
date analyzed	2/ 28/ 90	1/ 25/ 91	11/ 27/ 92	6/ 14/ 93	3/ 23/ 94	5/ 23/ 95
PROXIMATE						
% moisture	10.4	10.3	10.1	9.8	9.6	10.2
% ash (dry basis)	16.2	16.1	16.2	15.9	16.1	15.9
SULFUR FORMS (dry %)						
total sulfur	4.51	4.33	4.54	4.50	4.64	4.62
sulfate	0.01	0.06	0.01	0.03	0.01	0.04
pyritic	2.27	2.27	2.31	2.06	1.92	2.06
organic	2.24	2.00	2.22	2.41	2.72	2.51
CALORIFIC VALUE (dry)						
Btu/lb	11880	11740	11814	11787	11895	11833
GIESELER FLUIDITY						
maximum fluidity, ddpm	8	9	10	7	8	11
fluid range, °C	47	58	61	55	63	64
date analyzed	2/ 08/ 90	2/ 11/ 91	2/ 02/ 93	7/ 19/ 93	3/ 07/ 94	5/ 09/ 95
FREE SWELLING INDEX	3.5				3.5	3.5
GASEOUS OXYGEN						
% in bag atmosphere					0.12	0.00
date analyzed					3/ 08/ 94	5/ 09/ 95

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		
particle size stored		-20 mesh	-20 mesh	-20 mesh	-20 mesh		
note							
date sealed		10/ 22/ 90	10/ 22/ 90	10/ 22/ 90	10/ 22/ 90		
date analyzed	10/ 30/ 90	10/ 21/ 92	6/ 03/ 93	3/ 23/ 94	5/ 09/ 95		
PROXIMATE							
% moisture	28.4	28.3	28.6	28.1	28.6		
% ash (dry basis)	13.8	12.8	13.1	13.0	13.6		
SULFUR FORMS (dry %)							
total sulfur	0.73	0.77	0.81	0.95	0.87		
sulfate	0.02	0.01	0.01	0.00	0.01		
pyritic	0.28	0.20	0.22	0.22	0.27		
organic	0.43	0.56	0.58	0.72	0.58		
CALORIFIC VALUE (dry)							
Btu/lb	11168	11275	11287	11244	11210		
GASEOUS OXYGEN							
% in bag atmosphere				0.08	0.14		
date analyzed				3/ 08/ 94	5/ 09/ 95		

DECS- 12	Pittsburgh seam	hvAb			Ro max= 0.87	
FOIL BAG	original analysis	86 of 256	117 of 256	120 of 256	132 of 256	142 of 256
particle size stored		-20 mesh	-20 mesh	-20 mesh	-20 mesh	-20 mesh
note						
date sealed		7/ 30/ 90	7/ 30/ 90	7/ 30/ 90	7/ 30/ 90	7/ 30/ 90
date analyzed	8/ 23/ 90	7/ 31/ 91	10/ 23/ 92	6/ 04/ 93	3/ 23/ 94	5/ 23/ 95
PROXIMATE						
% moisture	2.40	2.47	2.99	1.98	2.35	2.46
% ash (dry basis)	10.25	11.05	10.35	9.52	9.86*	9.69
SULFUR FORMS (dry %)						
total sulfur	1.12	1.18	1.16	1.17	1.16	1.16
sulfate	0.01	0.01	0.01	0.00	0.00	0.01
pyritic	0.38	0.29	0.17	0.35	0.35	0.39
organic	0.73	0.88	0.98	0.82	0.81	0.76
CALORIFIC VALUE (dry)						
Btu/lb	13532	13337	13471	13563	13534	13603
GIESELER FLUIDITY					(a)	
maximum fluidity, ddpm	20002	6458	4309	2173	7866	6986
fluid range, °C	90	90	75	80	86	83
date analyzed	7/ 27/ 90	7/ 24/ 91	10/27/92	7/ 21/ 92	3/ 09/ 94	5/ 10/ 95
FREE SWELLING INDEX	7.5				8	8
GASEOUS OXYGEN						
% in bag atmosphere					0.01	0.04
date analyzed					3/ 08/ 94	5/ 10/ 95

(a) based on two of three runs which agreed within ASTM limits

* correction of Quarterly Technical Progress Report dated 10/14/94

DECS- 14		Upper Kittanning seam		hvAb		Ro max= 1.07	
FOIL BAG	original analysis	6 of 256	20 of 256	11 of 256	16 of 256		
particle size stored		-20 mesh	-20 mesh	-20 mesh	-20 mesh		
note							
date sealed		2/ 08/ 91	2/ 08/ 91	2/ 08/ 91	2/ 08/ 91		
date analyzed	2/ 15/ 91	11/ 27/ 92	6/ 15/ 93	3/ 24/ 94	5/ 23/ 95		
PROXIMATE							
% moisture	1.5	1.7	1.4	1.8	1.7		
% ash (dry basis)	10.5	10.3	10.6	10.5	10.7		
SULFUR FORMS (dry %)							
total sulfur	1.80	1.71	1.80	1.79	1.86		
sulfate	0.01	0.01	0.01	0.01	0.01		
pyritic	0.94	0.08	1.02	1.01	1.03		
organic	0.85	1.62	0.78	0.77	0.82		
CALORIFIC VALUE (dry)							
Btu/lb	13678	13723	13701	13803	13602		
GIESELER FLUIDITY							
maximum fluidity, ddpm	29930	27625	23619	29836	29945		
fluid range, °C	109	108	104	108	110		
date analyzed	2/ 19/ 91	2/ 04/ 93	7/ 23/ 93	3/ 10/ 94	5/ 16/ 95		
FREE SWELLING INDEX	8			7.5	8		
GASEOUS OXYGEN							
% in bag atmosphere				0.04	0		(a)
date analyzed				3/ 08/ 94	5/ 15/ 95		

(a) less than usual accuracy but definitely no significant oxygen

DECS-15		Lower Sunnyside seam		hvAb		Ro max= 0.80	
FOIL BAG	original analysis	3 of 256	6 of 256	5 of 256	8 of 256		
particle size stored		-20 mesh	-20 mesh	-20 mesh	-20 mesh		
note							
date sealed	7/ 05/ 91	10/ 15/ 92	10/ 15/ 92	10/ 15/ 92	10/ 15/ 92		
date analyzed		11/ 27/ 92	6/ 15/ 93	3/ 24/ 94	5/ 23/ 95		
PROXIMATE							
% moisture	2.9	2.9	2.6	3.1	3.0		
% ash (dry basis)	10.0	10.1	9.8	9.8	10.0		
SULFUR FORMS (dry %)							
total sulfur	1.65	1.75	1.62	1.64	1.66		
sulfate	0.05	0.03	0.02	0.02	0.02		
pyritic	0.61	0.94	0.69	0.76	0.70		
organic	0.99	0.78	0.91	0.87	0.94		
CALORIFIC VALUE (dry)							
Btu/lb	13429	13275	13396	13553	13415		
GIESELER FLUIDITY							
maximum fluidity, ddpm	959	258	170	215	246		
fluid range, °C	76	75	69	70	72		
date analyzed	6/ 13/ 91	2/ 05/ 93	7/ 26/ 93	3/ 11/ 94	5/ 11/ 95		
FREE SWELLING INDEX	4.5			4	4.5		
GASEOUS OXYGEN							
% in bag atmosphere				0.04	0		(a)
date analyzed				3/ 08/ 94	5/ 11/ 95		

(a) less than usual accuracy but definitely no significant oxygen

DECS-17		Blind Canyon seam		hvAb		Ro max=0.59	
REFRIGERATED, -20 MESH							
FOIL BAG		original analysis	204 of 256	205 of 256	17 of 256	10 of 256	
particle size stored			-20 mesh	-20 mesh	-20 mesh	-20 mesh	
note							
date sealed			12/ 04/ 91	12/ 04/ 91	12/ 04/ 91	12/ 04/ 91	
date analyzed		8/ 27/ 91	4/ 16/ 93	6/ 15/ 93	4/ 26/ 94	5/ 23/ 95	
PROXIMATE							
% moisture		3.7	3.9	3.8	3.9	4.1	
% ash (dry basis)		6.6	6.6	6.7	6.4	6.5	
SULFUR FORMS (dry %)							
total sulfur		0.44	0.43	0.42	0.47	0.44	
sulfate		0.01	0.01	0.01	0.01	0.01	
pyritic		0.02	0.03	0.05	0.04	0.04	
organic		0.41	0.39	0.36	0.43	0.39	
CALORIFIC VALUE (dry)							
Btu/lb		13826	13869	13904	13926	13745	
GIESELER FLUIDITY							
maximum fluidity, ddpm		5	4	2	2	2	
fluid range, °C		55	52	47	44	48	
date analyzed		8/ 16/ 91	10/ 28/ 92	7/ 27/ 93	4/ 20/ 94	5/ 16/ 95	
FREE SWELLING INDEX		2			2	2.5	
GASEOUS OXYGEN						(a)	
% in bag atmosphere					0.00	0	
date analyzed					4/ 20/ 94	5/ 16/ 95	

(a) less than usual accuracy but definitely no significant oxygen

DECS-17		Blind Canyon seam		hvAb		Ro max= 0.59	
UNREFRIGERATED, -20 MESH							
FOIL BAG		original analysis	7 of 16	15 of 16	8 of 16	10 of 16	
particle size stored			-20 mesh	-20 mesh	-20 mesh	-20 mesh	
note							
date sealed			8/ 15/ 91	8/ 15/ 91	8/ 15/ 91	8/ 15/ 91	
date analyzed			8/ 27/ 91	11/ 27/ 92	6/ 18/ 93	4/ 26/ 94	5/ 23/ 95
PROXIMATE							
% moisture		3.7	3.9	3.7	3.9	3.9	
% ash (dry basis)		6.6	6.4	6.5	6.4	6.6	
SULFUR FORMS (dry %)							
total sulfur		0.44	0.44	0.43	0.47	0.43	
sulfate		0.01	0.01	0.00	0.01	0.00	
pyritic		0.02	0.03	0.05	0.04	0.04	
organic		0.41	0.40	0.38	0.43	0.39	
CALORIFIC VALUE (dry)							
Btu/lb		13826	13858	13836	13862	13801	
GIESELER FLUIDITY							
maximum fluidity, ddpm		5	5	2	2	2	
fluid range, °C		55	50	36	46	42	
date analyzed		8/ 16/ 91	10/ 28/ 92	7/ 28/ 93	3/ 16/ 94	5/ 12/ 95	
FREE SWELLING INDEX		2			2	2	
GASEOUS OXYGEN						(a)	
% in bag atmosphere					0.05	0	
date analyzed					3/ 16/ 94	5/ 12/ 95	

(a) less than usual accuracy but definitely no significant oxygen

DECS-18		Kentucky #9 seam		hvBb		Ro max= 0.56
FOIL BAG	original analysis	3 of 256	2 of 256	5 of 256		
particle size stored		-20 mesh	-20 mesh	-20 mesh		
note						
date sealed		10/ 09/ 92	10/ 09/ 92	10/ 09/ 92		
date analyzed	11/ 22/ 91	6/ 18/ 93	4/ 26/ 94	5/ 25/ 95		
PROXIMATE						
% moisture	6.8	7.3	7.4	7.4		
% ash (dry basis)	12.3	11.3	11.8	11.7		
SULFUR FORMS (dry %)						
total sulfur	4.21	3.91	3.97	4.02		
sulfate	0.01	0.04	0.04	0.03		
pyritic	2.37	1.78	1.99	1.71		
organic	1.83	2.10	1.94	2.27		
CALORIFIC VALUE (dry)						
Btu/lb	12687	12763	12794	12723		
GIESELER FLUIDITY						
maximum fluidity, ddpm	527	409	159	677		
fluid range, °C	80	74	73	90		
date analyzed	2/ 11/ 93	7/ 29/ 93	4/ 11/ 94	5/ 12/ 95		
FREE SWELLING INDEX	2		3	4		
GASEOUS OXYGEN				(a)		
% in bag atmosphere			0.21	0		
date analyzed			3/ 29/ 94	5/ 12/ 95		

(a) less than usual accuracy but definitely no significant oxygen

DECS-23		Pittsburgh seam	hvAb	Ro max= 0.73
FOIL BAG		original analysis	17 of 256	
particle size stored			-20 mesh	
note				
date sealed			2/ 11/ 94	
date analyzed		2/ 21/ 94	2/ 08/ 95	
PROXIMATE				
% moisture		2.00	2.03	
% ash (dry basis)		9.44	9.64	
SULFUR FORMS (dry %)				
total sulfur		3.87	3.79	
sulfate		0.01	0.01	
pyritic		2.23	2.42	
organic		1.63	1.36	
CALORIFIC VALUE (dry)				
Btu/lb		13614	13553	
GIESELER FLUIDITY				
maximum fluidity, ddpm		30000	30000	
fluid range, °C		97	106	
date analyzed		2/ 07/ 94	1/ 23/ 95	
FREE SWELLING INDEX		7.5	7	
GASEOUS OXYGEN			(a)	
% in bag atmosphere			0	
date analyzed			5/ 16/ 95	

(a) less than usual accuracy but definitely no significant oxygen

DECS-24	Illinois #6 seam	hvBb	Ro max= 0.49
FOIL BAG	original analysis	105 of 328	
particle size stored		-20 mesh	
note			
date sealed		7/ 19/ 94	
date analyzed	9/ 07/ 94	2/ 08/ 95	
PROXIMATE			
% moisture	13.2	13.4	
% ash (dry basis)	13.4	13.5	
SULFUR FORMS (dry %)			
total sulfur	5.53	5.92	
sulfate	0.29	0.01	
pyritic	2.64	2.86	
organic	2.64	3.05	
CALORIFIC VALUE (dry)			
Btu/lb	12162	12074	
GIESELER FLUIDITY			
maximum fluidity, ddpm	49	55	
fluid range, °C	78	82	
date analyzed	6/ 14/ 94	1/ 24/ 95	
FREE SWELLING INDEX	3	3.5	
GASEOUS OXYGEN		(a)	
% in bag atmosphere		0	
date analyzed		5/ 16/ 95	

(a) less than usual accuracy but definitely no significant oxygen