DOE/PC/93051--T8

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: 1/01/96 - 3/31/96

Report Date: 4/25/96

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

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Quarterly Technical Progress Report: 1/01/96 - 3/31/96

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. Effort in the related study of liquefaction behavior (under Task 1F) over time has also been reduced.

The third annual monitoring program on the ten samples selected was completed during the quarter. 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 right-hand column of each table in Appendix A, show that the foil laminate bags continue to preserve DECS samples very well in comparison to storage containers used previously. Gieseler fluid behavior is one of the most sensitive indicators; the initial loss in fluidity exhibited by some samples may result from factors other than oxidation (degassing, etc.). Low fluidity results from 1993 may be related to changes to the equipment made at that time. Fluid range is a more stable indicator than maximum fluidity.

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.

Processing of earlier DECS samples to minus 60 mesh (-0.25 mm) 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 continues in this Task, although the total number of samples to be tested has been reduced following a cut in the level of funding.

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.

Table 1 summarizes preliminary results from CPMAS for the 5 coals collected during the second contract year. Appendix B includes the trace for each of these runs. This completes the planned CPMAS analyses.

Table 1. Structural parameters of DECS coals determined by CPMAS measurements

DECS-	seam	rank	carbon aromaticity	(aromatic + carbonyl + carboxyl) / total carbons	phenolics/ total carbons	(carboxyl+ carbonyl)/ total carbons	oxygenated aliphatic carbons / total carbons
29	U. Banner #3	hvAb	0.72	0.74	0.11	0.02	0.007
30	Splash Dam	m vb	0.76	0.78	0.11	0.02	0.002
31	Pond Creek	hvAb	0.70	0.72	0.13	0.02	0.009
32	Stockton-Lew.	hvAb	0.73	0.75	0.15	0.02	0.002
33	Ohio #4A	hvBb	0.75	0.80	0.15	0.05	0.019

Definitions:

carbon aromaticity*	$fa' = I_{100-165 \text{ ppm}} / I_{\text{total}}$
(aromatic + carbonyl + carboxyl) / total carbons	$fa = I_{100-220 \text{ ppm}} / I_{\text{total}}$
phenolics* / total carbons	$fp = I_{140-165 \text{ ppm}} / I_{\text{total}}$
(carboxyl + carbonyl) / total carbons	$fc = I_{165-220 \text{ ppm}} / I_{\text{total}}$
oxygenated aliphatic carbons / total carbons	$fo = I_{60-90 \text{ ppm}} / I_{\text{total}}$

where:

I = area within the indicated chemical shift ranges in a CPMAS spectrum

$I_{\text{total}} = 1 - 220 \text{ ppm}$

* corrected for contributions from spinning sidebands of aromatic carbons

DDMAS measurements have been completed for 5 DECS coals except for final data reduction, and the two remaining samples will be completed shortly.

Bloch decay experiments were performed as described by Hatcher et al. (1992), principally to verify the spectra obtained by conventional CPMAS (presented in this and other quarterly progress reports). Approximately 2500 transients with a cycle time of 80 s were collected. The spectra for 7 DECS coals are shown in Appendix C.

Task 11. Distribution of Coal Samples to Users

During the period 1/01/96 - 3/31/96 a total of 19 samples (16 DOE Sample Bank samples and 3 other Penn State samples) of various sizes in 28 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.

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 136 data printouts were distributed. In addition, 8 special data requests were filled by database searches and printed output or creation of a data disk, resulting in distribution of information on 325 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:

Indiana Geological Survey
Little Bear Laboratories, Montana
Pennsylvania State University, Fuel Science Department (7)
Pennsylvania State University, Materials Research Laboratory
Pennsylvania Bureau of Mining and Reclamation
Rice University, Chemistry Department
Rust College, Division of Science and Mathematics
TDA Research
University of Newcastle, Chemical Engineering Department (Australia)
University of Oklahoma, School of Geology and Geophysics
University of Utah
US Department of Energy, Pittsburgh Energy Technology Center
West Virginia University, Chemical Engineering Department

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, materials were developed during the quarter for inclusion on the world wide web (<http://www.ems.psu.edu/COPL/>).

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.
- Hatcher, P.G., Faulon, J.-L., Wenzel, K.A., and Cody, G.D., 1992. A structural Model for Lignin-Derived Vitrinite from High-Volatile Bituminous Coal (Coalified Wood). *Energy & Fuels*, v. 6, pp 813-820.

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APPENDIX A. 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	138 of 256
particle size stored		-20 mesh	-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	8/ 06/ 90
date analyzed	2/ 28/ 90	1/ 25/ 91	11/ 27/ 92	6/ 14/ 93	3/ 23/ 94	5/ 23/ 95	2/ 13/ 96
PROXIMATE							
% moisture	10.4	10.3	10.1	9.8	9.6	10.2	10.0
% ash (dry basis)	16.2	16.1	16.2	15.9	16.1	15.9	16.0
SULFUR FORMS (dry %)							
total sulfur	4.51	4.33	4.54	4.50	4.64	4.62	4.59
sulfate	0.01	0.06	0.01	0.03	0.01	0.04	0.01
pyritic	2.27	2.27	2.31	2.06	1.92	2.06	2.27
organic	2.24	2.00	2.22	2.41	2.72	2.51	2.30
CALORIFIC VALUE (dry)							
Btu/lb	11880	11740	11814	11787	11895	11833	11813
GIESELER FLUIDITY							
maximum fluidity, ddpm	8	9	10	7	8	11	9
fluid range, °C	47	58	61	55	63	64	58
date analyzed	2/ 08/ 90	2/ 11/ 91	2/ 02/ 93	7/ 19/ 93	3/ 07/ 94	5/ 09/ 95	1/ 23/ 96
FREE SWELLING INDEX	3.5				3.5	3.5	2.5
GASEOUS OXYGEN							
% in bag atmosphere					0.12	0.00	0.17
date analyzed					3/ 08/ 94	5/ 09/ 95	1/ 18/ 96

APPENDIX A. 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)						
Btu/lb	11168	11275	11287	11244	11210	11654
GASEOUS OXYGEN						
% in bag atmosphere				0.08	0.14	0.28
date analyzed				3/ 08/ 94	5/ 09/ 95	1/ 18/ 96

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

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	155 of 256
particle size stored		-20 mesh	-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	7/ 30/ 90
date analyzed	8/ 23/ 90	7/ 31/ 91	10/ 23/ 92	6/ 04/ 93	3/ 23/ 94	5/ 23/ 95	2/ 13/ 96
PROXIMATE							
% moisture	2.40	2.47	2.99	1.98	2.35	2.46	2.23
% ash (dry basis)	10.25	11.05	10.35	9.52	9.86*	9.69	9.34
SULFUR FORMS (dry %)							
total sulfur	1.12	1.18	1.16	1.17	1.16	1.16	1.16
sulfate	0.01	0.01	0.01	0.00	0.00	0.01	0.01
pyritic	0.38	0.29	0.17	0.35	0.35	0.39	0.40
organic	0.73	0.88	0.98	0.82	0.81	0.76	0.74
CALORIFIC VALUE (dry)							
Btu/lb	13532	13337	13471	13563	13534	13603	13646
GIESELER FLUIDITY					(a)		
maximum fluidity, ddpm	20002	6458	4309	2173	7866	6986	17800
fluid range, °C	90	90	75	80	86	83	87
date analyzed	7/ 27/ 90	7/ 24/ 91	10/27/92	7/ 21/ 92	3/ 09/ 94	5/ 10/ 95	1/ 24/ 96
FREE SWELLING INDEX	7.5				8	8	8
GASEOUS OXYGEN							
% in bag atmosphere					0.01	0.04	0.00
date analyzed					3/ 08/ 94	5/ 10/ 95	1/ 18/ 96

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

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

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

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	23 of 256
particle size stored		-20 mesh	-20 mesh	-20 mesh	-20 mesh	-20 mesh
note						
date sealed		2/ 08/ 91	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	2/ 13/ 96
PROXIMATE						
% moisture	1.5	1.7	1.4	1.8	1.7	1.6
% ash (dry basis)	10.5	10.3	10.6	10.5	10.7	10.3
SULFUR FORMS (dry %)						
total sulfur	1.80	1.71	1.80	1.79	1.86	1.90
sulfate	0.01	0.01	0.01	0.01	0.01	0.01
pyritic	0.94	0.08	1.02	1.01	1.03	1.15
organic	0.85	1.62	0.78	0.77	0.82	0.74
CALORIFIC VALUE (dry)						
Btu/lb	13678	13723	13701	13803	13602	13739
GIESELER FLUIDITY						
maximum fluidity, ddpm	29930	27625	23619	29836	29945	29953
fluid range, °C	109	108	104	108	110	108
date analyzed	2/ 19/ 91	2/ 04/ 93	7/ 23/ 93	3/ 10/ 94	5/ 16/ 95	1/ 24/ 96
FREE SWELLING INDEX	8			7.5	8	7.5
GASEOUS OXYGEN					(a)	
% in bag atmosphere				0.04	0	0.08
date analyzed				3/ 08/ 94	5/ 15/ 95	1/ 18/ 96

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

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

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	10 of 256
particle size stored		-20 mesh	-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	10/ 15/ 92
date analyzed		11/ 27/ 92	6/ 15/ 93	3/ 24/ 94	5/ 23/ 95	2/ 13/ 96
PROXIMATE						
% moisture	2.9	2.9	2.6	3.1	3.0	3.0
% ash (dry basis)	10.0	10.1	9.8	9.8	10.0	10.1
SULFUR FORMS (dry %)						
total sulfur	1.65	1.75	1.62	1.64	1.66	1.58
sulfate	0.05	0.03	0.02	0.02	0.02	0.05
pyritic	0.61	0.94	0.69	0.76	0.70	0.77
organic	0.99	0.78	0.91	0.87	0.94	0.76
CALORIFIC VALUE (dry)						
Btu/lb	13429	13275	13396	13553	13415	13295
GIESELER FLUIDITY						
maximum fluidity, ddpm	959	258	170	215	246	60
fluid range, °C	76	75	69	70	72	63
date analyzed	6/ 13/ 91	2/ 05/ 93	7/ 26/ 93	3/ 11/ 94	5/ 11/ 95	1/ 25/ 96
FREE SWELLING INDEX	4.5			4	4.5	3.5
GASEOUS OXYGEN					(a)	
% in bag atmosphere				0.04	0	0.18
date analyzed				3/ 08/ 94	5/ 11/ 95	1/ 18/ 96

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

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

DECS-17 REFRIGERATED, -20 MESH		Blind Canyon seam	hvAb		Ro max=0.59	
FOIL BAG	original analysis	204 of 256	205 of 256	17 of 256	10 of 256	11 of 256
particle size stored		-20 mesh	-20 mesh	-20 mesh	-20 mesh	-20 mesh
note						
date sealed		12/ 04/ 91	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	2/ 13/ 96
PROXIMATE						
% moisture	3.7	3.9	3.8	3.9	4.1	4.0
% ash (dry basis)	6.6	6.6	6.7	6.4	6.5	6.7
SULFUR FORMS (dry %)						
total sulfur	0.44	0.43	0.42	0.47	0.44	0.43
sulfate	0.01	0.01	0.01	0.01	0.01	0.01
pyritic	0.02	0.03	0.05	0.04	0.04	0.04
organic	0.41	0.39	0.36	0.43	0.39	0.38
CALORIFIC VALUE (dry)						
Btu/lb	13826	13869	13904	13926	13745	13942
GIESELER FLUIDITY						
maximum fluidity, ddpm	5	4	2	2	2	2
fluid range, °C	55	52	47	44	48	46
date analyzed	8/ 16/ 91	10/ 28/ 92	7/ 27/ 93	4/ 20/ 94	5/ 16/ 95	1/ 26/ 96
FREE SWELLING INDEX	2			2	2.5	1.5
GASEOUS OXYGEN					(a)	
% in bag atmosphere				0.00	0	0.10
date analyzed				4/ 20/ 94	5/ 16/ 95	1/ 18/ 96

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

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

DECS-17 Blind Canyon seam UNREFRIGERATED, -20 MESH		hvAb		Ro max= 0.59		
FOIL BAG	original analysis	7 of 16	15 of 16	8 of 16	10 of 16	9 of 16
particle size stored		-20 mesh	-20 mesh	-20 mesh	-20 mesh	-20 mesh
note						
date sealed		8/ 15/ 91	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	2/ 13/ 96
PROXIMATE						
% moisture	3.7	3.9	3.7	3.9	3.9	3.5
% ash (dry basis)	6.6	6.4	6.5	6.4	6.6	6.6
SULFUR FORMS (dry %)						
total sulfur	0.44	0.44	0.43	0.47	0.43	0.42
sulfate	0.01	0.01	0.00	0.01	0.00	0.01
pyritic	0.02	0.03	0.05	0.04	0.04	0.04
organic	0.41	0.40	0.38	0.43	0.39	0.38
CALORIFIC VALUE (dry)						
Btu/lb	13826	13858	13836	13862	13801	13925
GIESELER FLUIDITY						
maximum fluidity, ddpm	5	5	2	2	2	2
fluid range, °C	55	50	36	46	42	44
date analyzed	8/ 16/ 91	10/ 28/ 92	7/ 28/ 93	3/ 16/ 94	5/ 12/ 95	1/ 26/ 96
FREE SWELLING INDEX	2			2	2	2
GASEOUS OXYGEN					(a)	
% in bag atmosphere				0.05	0	0.16
date analyzed				3/ 16/ 94	5/ 12/ 95	1/ 18/ 96

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

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

DECS-18	Kentucky #9 seam		hvBb	Ro max= 0.56	
FOIL BAG	original analysis	3 of 256	2 of 256	5 of 256	8 of 256
particle size stored		-20 mesh	-20 mesh	-20 mesh	-20 mesh
note					
date sealed		10/ 09/ 92	10/ 09/ 92	10/ 09/ 92	10/ 09/ 92
date analyzed	11/ 22/ 91	6/ 18/ 93	4/ 26/ 94	5/ 25/ 95	2/ 13/ 96
PROXIMATE					
% moisture	6.8	7.3	7.4	7.4	7.1
% ash (dry basis)	12.3	11.3	11.8	11.7	11.7
SULFUR FORMS (dry %)					
total sulfur	4.21	3.91	3.97	4.02	4.05
sulfate	0.01	0.04	0.04	0.03	0.01
pyritic	2.37	1.78	1.99	1.71	2.03
organic	1.83	2.10	1.94	2.27	2.01
CALORIFIC VALUE (dry)					
Btu/lb	12687	12763	12794	12723	12818
GIESELER FLUIDITY					
maximum fluidity, ddpm	527	409	159	677	513
fluid range, °C	80	74	73	90	82
date analyzed	2/ 11/ 93	7/ 29/ 93	4/ 11/ 94	5/ 12/ 95	1/ 25/ 96
FREE SWELLING INDEX	2		3	4	3.5
GASEOUS OXYGEN				(a)	
% in bag atmosphere			0.21	0	0.09
date analyzed			3/ 29/ 94	5/ 12/ 95	1/ 18/ 96

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

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

DECS-23	Pittsburgh seam	hvAb	Ro max= 0.73	
FOIL BAG	original analysis	17 of 256	18 of 256	
particle size stored		-20 mesh	-20 mesh	
note				
date sealed		2/ 11/ 94	2/ 11/ 94	
date analyzed	2/ 21/ 94	2/ 08/ 95	11/ 30/ 95	
PROXIMATE				
% moisture	2.00	2.03	2.09	
% ash (dry basis)	9.44	9.64	9.67	
SULFUR FORMS (dry %)				
total sulfur	3.87	3.79	3.66	
sulfate	0.01	0.01	0.00	
pyritic	2.23	2.42	2.45	
organic	1.63	1.36	1.20	
CALORIFIC VALUE (dry)				
Btu/lb	13614	13553	13565	
GIESELER FLUIDITY				
maximum fluidity, ddpm	30000	30000	30000	
fluid range, °C	97	106	100	
date analyzed	2/ 07/ 94	1/ 23/ 95	11/ 28/ 95	
FREE SWELLING INDEX	7.5	7	7.5	
GASEOUS OXYGEN		(a)		
% in bag atmosphere		0	not	
date analyzed		5/ 16/ 95	determined	

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

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

DECS-24	Illinois #6 seam	hvBb	Ro max= 0.49	
FOIL BAG	original analysis	105 of 328	106 of 328	
particle size stored		-20 mesh	-20 mesh	
note				
date sealed		7/ 19/ 94	7/ 19/ 94	
date analyzed	9/ 07/ 94	2/ 08/ 95	11/ 29/ 95	
PROXIMATE				
% moisture	13.2	13.4	13.4	
% ash (dry basis)	13.4	13.5	13.6	
SULFUR FORMS (dry %)				
total sulfur	5.53	5.92	5.76	
sulfate	0.25	0.01	0.01	
pyritic	2.64	2.86	2.77	
organic	2.64	3.05	2.98	
CALORIFIC VALUE (dry)				
Btu/lb	12162	12074	12242	
GIESELER FLUIDITY				
maximum fluidity, ddpm	49	55	53	
fluid range, °C	78	82	82	
date analyzed	6/ 14/ 94	1/ 24/ 95	11/ 29/ 95	
FREE SWELLING INDEX	3	3.5	3.5	
GASEOUS OXYGEN		(a)		
% in bag atmosphere		0	not	
date analyzed		5/ 16/ 95	dertermined	

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

APPENDIX B. Conventional CPMAS NMR spectra

Figure B-1. Conventional ^{13}C CPMAS NMR spectrum for DECS-29, hvAb Upper Banner #3 seam, VA.

Figure B-2. Conventional ^{13}C CPMAS NMR spectrum for DECS-30, mvb Splash Dam seam, VA.

APPENDIX B. Conventional CPMAS NMR spectra

Figure B-3. Conventional ¹³C CPMAS NMR spectrum for DECS-31, hvAb Pond Creek seam, KY.

Figure B-4. Conventional ¹³C CPMAS NMR spectrum for DECS-32, hvAb Stockton-Lewiston seam, WV.

APPENDIX B. Conventional CPMAS NMR spectra

Figure B-5. Conventional ^{13}C CPMAS NMR spectrum for DECS-33, hvBb Ohio #4A seam, OH.

APPENDIX C. Bloch Decay NMR spectra

Figure C-1. Bloch decay ^{13}C NMR spectrum for DECS-12, hvAb Pittsburgh seam, PA.

Figure C-2. Bloch decay ^{13}C NMR spectrum for DECS-17, hvAb Blind Canyon seam, UT.

APPENDIX C. Bloch Decay NMR spectra

Figure C-3. Bloch decay NMR spectrum for DECS-29, hvAb Upper Banner #3 seam, VA.

Figure C-4. Bloch decay NMR spectrum for DECS-30, mvb Splash Dam seam, VA.

APPENDIX C. Bloch Decay NMR spectra

Figure C-5. Bloch decay NMR spectrum for DECS-31, hvAb Pond Creek seam, KY.

Figure C-6. Bloch decay NMR spectrum for DECS-32, mvb Stockton-Lewiston seam, WV.

APPENDIX C. Bloch Decay NMR spectra

Figure C-5. Bloch decay NMR spectrum for DECS-33, hvBb Ohio #4A seam, OH.