FINAL TECHNICAL REPORT

For the period from September 1, 1986 through September 30, 1987

Project title: Illinois Basin Coal Sample Program
ICCI Project Number: 86-87/3.3A-5
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ABSTRACT

The Illinois Basin Coal Sample Program (IBCSP) was initiated in 1983 by the Illinois State Geological Survey for the Illinois Coal Development Board. It became an interstate program in 1986 when the State of Indiana placed an Indiana coal in the program and accepted a share of the program's annual maintenance cost. Homogenized samples of five coals (3/8"x 0) are being maintained under a nitrogen atmosphere in 55-gallon barrels at the Illinois State Geological Survey Applied Research Laboratory at Oak and Gregory in Champaign, Illinois. One additional sample, a 3-ton channel sample which matches the only Illinois coal in Premium Coal Sample Program (PCSP) funded by the U. S. Department of Energy, was collected for IBCSP by Argonne National Laboratory (ANL) and prepackaged in three sizes of containers (5 gallon, 1 gallon, and 1 pint). This sample is of higher quality than the remaining five because is was maintained in an argon atmosphere from the mine to the processing facility and all processing was carried out under nitrogen at controlled humidity in ANL's inert atmosphere facility.

The sizes of samples and the focus on Illinois Basin coals are distinguishing characteristics of the IBCSP. Nominal 20-pound bags (22-25 lbs) of 3/8" x 0 coal are prepared for distribution by riffling a barrel of coal into 16 equal portions. Smaller samples (1/256$^{\underline{th}}$ of a barrel; nominally 1 lb) are prepared by crushing one of the nominal 20-pound samples to approximately 8 mesh x 0 and then riffling it into 16 equal parts. The sample preparation is carried out in air but all bagged samples are returned quickly to a nitrogen atmosphere in 55-gallon barrels until they are delivered to a requester. Analyses over the last 4 years show that the sampling procedures do produce representative samples and that changes in the samples due to exposure to air are relatively minor as judged by changes in the Free Swelling Index.

The IBCSP Advisory Committee provides guidance to the IBCSP by making recommendations on the sample selection, budgets and related matters. Findings based on a questionnaire which was circulated in June and July of 1987 for The Advisory Committee will be one of the factors used to establish a priority for future addition of samples to the Program. The questionnaire was circulated to managers and planners who deal with coal in addition to research personnel in government, educational and private research laboratories.

September in New Orleans before the Division of Fuel Chemistry at the 194th National Meeting of the American Chemical Society (Kruse, Harvey and Rapp, 1987a). The second will appear in the Proceedings of the Second International Conference on High Sulfur Coals (Kruse, Harvey, and Rapp, 1987b) to be held in Carbondale, Illinois, in late September.

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OBJECTIVES

The Illinois Basin Coal Sample Program (IBCSP), was established to facilitate comparisons of results among laboratories conducting basic and applied research on Illinois Basin coal, initially those funded by the State of Illinois through the Center for Research on Sulfur in coal but with time to other laboratories and programs outside the state. The inclusion of coals from neighboring states mining Illinois Basin coals and the participation by organizations in those states in the IBCSP is a long range objective which was partially achieved by this year's joining of the program by the State of Indiana. An additional objective this year was the strengthening of the support for the program by an active Advisory Committee and by publicizing the Program.

Tasks for this year included:

- Maintenance of samples - The samples in barrels were to be maintained in a nitrogen atmosphere on shelving at the Applied Research Laboratory and the prepackaged sample (IBCSP-5) was to be protected from a harsh environment.

- Analyses and tests - Representative samples of each of the samples in the program were to be analyzed twice during the year.

- Maintain records - Records shall include inventory records and correspondence with requesters.

- Process orders for coal monthly - Orders received were to be processed once a month.

- Provide reports and minutes - These include quarterly reports to the CRSC and minutes of the IBCSP Advisory Committee meetings.

- Publicize the program - Prepare a Research Focus sheet for distribution through the CRSC and submit papers for acceptance at technical meetings where coal sample programs are featured.

- Make arrangements for addition of a sixth sample - At the beginning of the year it was anticipated that Indiana would join the program.

- Further characterize coals in the program and provide advice to researchers - Although not written into the program and not specifically funded, it was our intention to use any uncommitted time to further characterize samples and to assist those needing simple beneficiation techniques to alter samples to meet specific research needs.

INTRODUCTION AND BACKGROUND

Coal sample programs, frequently called sample banks, facilitate a comparison of results among laboratories by providing splits of the same coal to the user group. A number of sample programs are in operation in the United States but few provide multi-pound quantities and they are not designed to meet regional needs. A summary on the status of various coal banks in the United States can be found in the Final Technical Report for this project covering the period January 1, 1983 through August 31, 1986 (Kruse et al., 1986). Of special importance to Illinois is the U. S. Department of Energy funded the Premium Coal Sample Program (PCSP) at ANL in late 1982. It was designed to meet nation-wide, small-scale, basic-research needs of the research community. A rigid-wall facility was constructed for processing barrel quantities of coal in a nitrogen environment with remote-control equipment. Exceptional care was planned in collection, transport, processing, and storage of the channel samples to maintain the coal's quality. The relative humidity of the nitrogen atmosphere was controlled to avoid changes in the moisture level. Samples are now available in 5 and 10 gram quantities, hermetically sealed in glass ampules (Vorres, 1986). Close cooperation between the PCSP and the IBCSP has been maintained from the beginning. It was recognized that the availability of the same Illinois Basin coal in both programs would likely result in a wider range of users than for any other coal in the PCSP and perhaps more than for a single coal in any program.

The Illinois Coal Development Board (ICDB) (previously the Illinois Coal Research Board) funds a variety of coal research projects in the State of Illinois and requires its contractors to use at least one coal from the IBCSP to provide a link with other research. The goal of the IBCSP is to maintain multi-ton quantities of a few coals under conditions which minimize their deterioration with time. The program has some of the features of an aborted Coal Repository funded by the DOE's Pittsburgh Energy Technology Center at TRW's plant at San Juan Capistrano, California (TRW Energy Development Group, 1983), but it is directed at Illinois Basin coals. With the exception of IBCSP-5, a sample prepared by ANL, the samples in the IBCSP are not pristine and have undergone some oxidation before leaving the mine site, during transport to Champaign by truck, and during the time required to crush, riffle and package the samples in air. For many types of tests, applied bench-scale testing in particular, this degree of oxidation is acceptable. Coals entering a commercial process have suffered some degree of oxidation in mining and cleaning.

Members of the Illinois research community working on the problems of sulfur and chlorine in Illinois coals want to study a selection of coals having differences in (a) rank, (b) ratios of organic sulfur to pyritic sulfur, (c) total sulfur, (d) washability and (e) seam number. If only low, medium, and high values had been chosen for each of the first four parameters for the two major seams (No. 5 and No. 6) the number of possible combinations would have been 162. If the number of samples in the IBCSP program remains less than ten, it is evident that many users will need to supplement the number of coals they get from the IBCSP with additional ones chosen to cover the range of differences for the properties of their interest. Mineral beneficiation techniques can and have been used to expand the number of uses of the samples. The following samples are among those which are being considered for addition to the Program:

- A sample with high chlorine content, 0.4 -0.5% (the current six samples have less than 0.2% Cl)

- A sample low in organic sulfur, one which can be cleaned sufficiently by physical cleaning alone to give a coal producing less than 1.2 lbs of sulfur dioxide per million BTU (a compliance coal for plants 73 MW or less built between 1971 and 1978)

- A sample having an appreciable difference between the $^{34}S/^{32}S$ isotope ratio of the sulfur in the organic form and the sulfur in the pyritic form so that the sulfur in products of chemical reactions of each form can be traced to its organic or inorganic origin and measured quantitatively.

- Samples of state-of-the-art purity coal macerals. Macerals would be available in small quantities (milligrams or grams at most). Vitrinite, sporinite and inertinites (mainly semi-fusinite) comprise the greatest volume of Illinois coal macerals.

- A deep-cleaned coal which is a state-of-the-art, physically-cleaned coal which would contain primarily organic sulfur.

- A higher rank bituminous coal.

- A coal with a significantly different maceral composition than currently available.

EXPERIMENTAL PROCEDURES

Selection and Procurement of Coals

The head of the ISGS Minerals Engineering Section, with the assistance from Dr. R. D. Harvey of the ISGS Coal Section and suggestions from investigators funded by the ICDB, chose the first three coals in the summer of 1983. They were mine-washed coals and their ash analyses are representative of current commercial production. Because coal rank increases from north to south in Illinois, west-central and southern mines were included to cover the range in ranks for Illinois coals. A Colchester (No. 2) coal from a west-central Illinois surface mine, a Herrin (No. 6) coal from a west central Illinois underground mine and a mixture of 80 percent Springfield (No. 5) coal and 20 percent Herrin (No. 6) coal from a cleaning plant processing southern Illinois slope and strip mine coals, respectively, were the three coals initially selected. In December 1983, the same group chose a fourth sample, a tipple sample of Herrin (No. 6) coal from a Southern Illinois underground mine. It has all the initial components of the raw coal including clay and water soluble components (chlorine in particular).

Arrangements were made with the mines to obtain three tons each of the first three coals at a time when freshly-minded coal was moving through the cleaning plant without delay. The coal was transported to the Applied Research Laboratory in a dump truck equipped with a grain bed. Care was taken to minimize loss of moisture during transport by covering the coal with a plastic drop cloth. It was held down by a wooden frame of 2" x 6" lumber nailed into place at the mine site after loading. Additionally, the top of the grain bed was covered with a canvas tarpaulin. At the Applied Research Laboratory each three-ton sample was transferred to sixteen 55-gallon barrels having removable tops. The fourth sample, a 1.5-ton tipple sample, was placed in eight barrels at the mine.

The selection of the fifth coal, a channel sample, was made by the PCSP committee advising Dr. Vorres. It was collected December 3, 1985, at a west-central Illinois underground mine. This four-ton sample of Herrin (Illinois No. 6) coal was collected from a single block of coal seven feet thick. After exposing the block, the samples were immediately loaded into stainless steel drums in the mine and the drums were purged with argon gas as they reached the surface. Upon reaching the Argonne National Laboratory, the drums were weighed, and processing of a one-ton sample for the PCSP began. The processing of the IBCSP samples in three one-ton batches in an atmosphere of nitrogen took place in January 1986. The coal (-20 mesh) was sealed in one pint cans, one gallon cans (about 6 lbs) and 5 gallon pails (about 30 lbs). The sample as delivered by ANL consisted of; 65 5-gallon pails; 360 1-gallon cans and 1230 1-pound cans.

The selection of sixth sample (IBCSP-6) was made by Dr. Donald Carr of the Indiana Geological Survey in consultation with Richard Harvey of the Illinois State Geological Survey. The procedure for collection of the Indiana sample was essentially the same as that described for the first four except that the processing site was Cepheus Laboratories in Marion, Illinois. Additionally, ten of the sixteen barrels of coal were additionally riffled into 16 subsamples. These 160 fractions were packaged in plastic bags and returned to barrels (14 barrels were required for the bagged coal which requires more volume than bulk coal). This prepackaging not only reduces the amount of labor required to deliver this coal to requesters, but it also assured that splitting took place in a uniform manner at a time when atmospheric conditions were relatively constant. The sealed barrels were transported to Champaign by truck. Once in Champaign, the barrels were purged with nitrogen and the seals checked to ensure that the barrels held nitrogen pressure. The six barrels which were not subdivided can be made available to users needing larger quantities of this sample.

When the supply of smaller samples (smaller than 1-barrel) from any of the five coals stored in barrels has been exhausted, the stock is replenished by opening and riffling a previously unopened barrel. Sixteen samples are produced from a barrel of coal in the first stage of riffling. Each sample at this stage weighs a little more than 20 pounds. These bagged samples are also maintained in barrels under a nitrogen atmosphere. These nominal 20-pound bags are the smallest samples of the 3/8" by 0 coal. Smaller samples of IBCSP-1 through IBCSP-4 and IBCSP-6 are prepared for requesters by first crushing a 20-pound bag to approximately 8 mesh and then riffling it into 16 equal samples. These approach 1.5 pounds (1/256th of a barrel) but have been distributed as 1-pound samples.

Storage Facility

The location of the storage on the ground floor inside the ISGS Applied Research Laboratory is convenient for frequent monitoring of the integrity of the containers and for immediate access by personnel preparing samples for shipment. Five racks with four levels each have a capacity of 72 barrels (stored on their sides). Two of these racks were covered with plywood sheeting for storage of IBCSP-5, the channel sample of Illinois No. 6 prepackaged in 1-pint cans, 1-gallon cans and 5-gallon pails at ANL. A walkie stacker is available for lifting and lowering the samples to and from their storage positions. A nitrogen manifold keeps the barrels under 1-3 psig nitrogen pressure.

Homogenization of IBCSP-1 through IBCSP-4 and IBCSP-6

The crushing, screening and homogenization operations for the three-ton samples are summarized in the 1986 Final Report (Kruse, 1986). One stage of riffling was omitted when the 1.5-ton tipple sample (IBCSP-4) was processed. The three tons of coal for each sample was transferred to sixteen 55-gallon barrels. The first four barrels of a given sample were opened one at a time, put through a jaw crusher, and then screened through a 3/8" (9mm) screen before riffling twice into four barrels. The plus 3/8" material was reduced in size by a roll crusher until it passed through the 3/8" screen. These operations were repeated with the remaining three groups of four barrels each. At this point the material in each set of four was uniform. A second riffling in groups of four, taking one barrel from each of the four sets, provided the final homogenization needed to produce sixteen barrels of uniform composition. A plastic liner was inserted in each barrel before the final riffling.

Modification of Barrels

The barrel lids (tops) were modified to facilitate purging with nitrogen and subsequent checking of pressures when the barrels are attached to a manifold supplying nitrogen. Each lid was fitted with a valve stem on one side and a 1/4" (6 mm) tubing connector placed near the rim on opposite side of the top. A flexible tube extending to the bottom of the barrel was attached to copper tubing extending through the tubing connector (Kruse, 1986). The gaskets used for reliable closure of the 55-gallon barrels were obtained from Barrel Accessories and Supply Company (BASCO), 4647 West 47th street, Chicago. These white, tubular, hard-rubber gaskets are the variety that Kraft Incorporated has found to be best for preservation of cheese in during shipment. The standard type of gaskets furnished by barrel vendors leaked.

Nitrogen Gas Purge

No attempt was made to purge air from the interstitial space in the bagged coal but the void space outside the bag(s) was purged. Preliminary tests with four barrels of fresh coal, obtained early in 1983 before collection of the first sample (Herrin [No. 6] coal) showed the coal consumed the oxygen of the air in the interstitial space in bagged samples within a few days if not hours. Because nitrogen is the gas remaining after coal has reacted with the air, it was considered to be the environment of choice for storage.

RESULTS AND DISCUSSION

Analyses of the Coal Samples

Barrel to barrel variations in the initial composition of coal from the same sample are believed to be small based on the analyses of samples taken during a pilot test of the homogenization procedure (Kruse, 1986). This homogenization test was performed on four barrels of coal from the same washing plant where IBCSP-1 was later obtained. Subsequent analyses of fractions of coal riffled from four barrels of the tipple coal, IBCSP sample 4, showed a low standard deviation between samples from different barrels (Kruse, 1986). Four of the eight barrels of the tipple coal were riffled into 20-pound bags during the initial preparation. One bag from each of the four barrels was crushed, sampled and analyzed. The variations are not much more than the variation expected on a single sample analyzed by different analysts or in different laboratories. Ultimate, proximate, and free swelling index analyses for several time periods for IBCSP samples 1 through 5 are shown in tables 1 through 5. Trace elements analyses for IBCSP-1 and IBCSP-5 are shown in table 7. Analyses of 10 portions of IBSCP-6 taken from five barrels at the same time appear in table 6.

The ISGS coal analysis laboratory has installed LECO analyzers. The March analyses were carried out both by the previous ASTM procedures and with the new LECO and are shown in tables 1-5. The August analyses were by the LECO analyzers only. The decision by ISGS coal laboratory analysts to change to LECO was made only after checking the results thoroughly in round robins. The LECO results were shown to be closer to the mean values of results from a large number of laboratories than the ASTM procedures used heretofore by the ISGS coal lab. Nevertheless, it will be seen that there is a discontinuity at the point of changing our methods when considering a large number of analyses of the same sample over time. Moisture is a little higher by the LECO method and this raises the BTU values. Volatile matter is generally lower by the LECO method and fixed carbon higher. Carbon values are generally a little higher by LECO and hydrogen may be lower.

Changes in Properties During Storage

Periodic testing of samples in storage is highly desirable to follow the changes in properties occurring with time. Because a barrel must undergo a series of riffling and size reduction steps to provide a representative sample, the 15 grams of material needed for a check cannot be withdrawn from a barrel of 3/8" x 0 coal without disrupting of the storage conditions. Prepackaging of initial samples in small quantities would have facilitated a systematic checking of properties; representative units of material could then have been sacrificed for the scheduled checking without changing the characteristics of major fractions of the material. The initial samples were not prepackaged for three reasons. One was the hope that with the availability of a facility to do the work in a nitrogen atmosphere (at ANL) further riffling and splitting of

Table 1. Analyses of IBCSP-1 (Moisture Free values)

	Average of all previous values[1]	March, 1987 (C26164) ASTM[2]	March, 1987 (C26164) LECO[3]	August, 1987 (C27346) LECO[3]
Moisture	14.1	14.1	14.9	14.9
Volatile Matter	44.1	43.7	40.4	40.6
Fixed Carbon	45.6	46.1	49.1	49.0
H-T Ash	10.3	10.2	10.5	10.4
Carbon	67.66[4]	68.61	69.26	69.26
Hydrogen	4.86	4.99	4.93	5.31
Nitrogen	1.18	1.49	1.51	1.34
Oxygen	11.74	10.37	9.41	9.34
Sulfatic Sulfur[5]	0.06	0.00	0 00	0.08
Pyritic Sulfur	1.20	1.21	1.22	1.41
Organic Sulfur	3.00	3.13	3.17	2.85
Total Sulfur	4.26	4.34	4.39	4.35
Total Chlorine	0.13	0.13	0.13	0.11
BTU/lb	12606	12584	12700	12656
FSI	4.5	4.5	4.5	4.5

[1] Avg of all analyses through December 1986

[2] Moisture, Ash, Volatile Matter and Fixed Carbon obtained using ASTM methods of analysis. Other values determined as in column one, but adjusted for ASTM moisture value

[3] Moisture, Ash, Volatile Matter and Fixed Carbon determined using LECO MAC400 proximate analyzer. C, H, N determined using LECO 600. Other values converted to dry basis using LECO 400 moisture.

[4] Ultimate Analysis listed is an average of three determinations by Carlos Erba and one by LECO 600.

[5] ASTM method used (D-2792) is subject to minor loss of $BaSO_4$ precipitate. A reported value of 0.00% can be as high as 0.04%.

Table 2. Analyses of IBCSP-2 (Moisture Free Values)

	Average of all previous values[1]	March, 1987 (C26164)		August, 1987 (C27346)
		ASTM[2]	LECO[3]	LECO[3]
Moisture	13.6	13.1	14.1	14.1
Volatile Matter	43.3	42.6	39.8	39.8
Fixed Carbon	49.9	50.9	53.3	53.2
H-T BAsh	6.8	6.5	6.9	7.0
Carbon	73.31[4]	72.52	73.36	74.85
Hydrogen	5.21	4.94	4.87	5.50
Nitrogen	1.47	1.76	1.78	1.48
Oxygen	10.08	10.88	9.65	7.78
Sulfatic Sulfur[5]	0.10	0.00	0.00	.14
Pyritic Sulfur	2.34	2.27	2.29	2.42
Organic Sulfur	0.92	1.13	1.14	.83
Total Sulfur	3.23	3.40	3.44	3.39
Total Chlorine	0.03	.02	.02	0.00
BTU/lb	13526	13535	13696	13640
FSI	4.3	4.5	4.5	4.5

1 Avg of all analyses through December 1986

2 Moisture, Ash, Volatile Matter and Fixed Carbon obtained using ASTM methods of analysis. Other values determined as in column one, but adjusted for ASTM moisture value

3 Moisture, Ash, Volatile Matter and Fixed Carbon determined using LECO MAC400 proximate analyzer. C, H, N determined using LECO 600, other values converted to dry basis using LECO 400 moisture

4 Ultimate Analysis listed is an average of three determination by Carlos Erba and one by LECO 600

5 ASTM method used (D-2792) is subject to minor loss of $BaSO_4$ precipitate. A reported value of 0.00% can be as high as 0.04%.

Table 3. Analyses of IBCSP-3 (Moisture Free Values)

	Average of all previous values[1]	March, 1987 (C26164) ASTM[2]	March, 1987 (C26164) LECO[3]	August, 1987 (C27346) LECO[3]
Moisture	5.4	5.2	5.9	5.8
Volatile Matter	39.2	38.3	36.1	36.1
Fixed Carbon	52.5	53.1	55.4	55.4
H-T Ash	8.3	8.6	8.5	8.5
Carbon	73.82[4]	73.84	74.46	74.73
Hydrogen	4.94	4.61	4.56	5.18
Nitrogen	1.68	1.97	1.99	1.65
Oxygen	8.99	8.60	8.09	7.59
Sulfatic Sulfur[5]	0.09	0.00	0.00	.05
Pyritic Sulfur	1.03	1.03	1.03	1.14
Organic Sulfur	1.3	1.35	1.37	1.16
Total Sulfur	2.27	2.38	2.40	2.35
Total Chlorine	0.18	0.17	.18	.16
BTU/lb	13437	13432	13532	13478
FSI	5.2	5.0	5.0	5.5

1 Avg of all analyses through December 1986

2 Moisture, Ash, Volatile Matter and Fixed Carbon obtained using ASTM methods of analysis. Other values determined as in column one, but adjusted for ASTM moisture value

3 Moisture, Ash, Volatile Matter and Fixed Carbon determined using LECO MAC400 proximate analyzer. C, H, N using LECO 600. Other values converted to dry basis using LECO 400 moisture.

4 Ultimate Analysis listed is an average of four determination by Carlos Erba and one by LECO 600

5 ASTM method used (D-2792) is subject to minor loss of $BaSO_4$ precipitate. A reported value of 0.00% can be as high as 0.04%.

Table 4. Analyses of IBCSP-4 (Moisture Free Values)

	Average of all previous values[1]	March, 1987 (C26164) ASTM[2]	March, 1987 (C26164) LECO[3]	August, 1987 (C27346) LECO[3]
Moisture	10.2	10.2	10.6	10.4
Volatile Matter	30.8	30.6	28.3	28.1
Fixed Carbon	31.2	31.9	33.3	32.9
H-T Ash	38.0	37.5	38.4	39.0
Carbon	45.97[4]	46.87	47.07	46.58
Hydrogen	3.46	2.96	2.93	3.59
Nitrogen	0.80	1.11	1.12	.84
Oxygen	7.48	7.40	6.30	5.76
Sulfatic Sulfur[5]	0.10	0.00	0.00	0.08
Pyritic Sulfur	2.33	2.49	2.47	2.56
Organic Sulfur	1.32	1.67	1.71	1.60
Total Sulfur	4.19	4.16	4.18	4.23
Total Chlorine	0.05	0.05	0.06	0.03
BTU/lb	8466	8565	8601	8471
FSI	2.1	2.0	2.0	1.5

[1] Avg of all analyses through December 1986

[2] Moisture, Ash, Volatile Matter and Fixed Carbon obtained using ASTM methods of analysis. Other values determined as in column one, but adjusted for ASTM moisture value

[3] Moisture, Ash, Volatile Matter and Fixed Carbon determined using LECO MAC400 proximate analyzer. C, H, N determined using LECO 600. Other values converted to dry basis usng LECO 400 moisture.

[4] Ultimate Analysis listed is an average of three determinations by Carlos Erba and one by LECO 600.

[5] ASTM method used (D-2792) is subject to minor loss of $BaSO_4$ precipitate. A reported value of 0.00% can be as high as 0.04%.

Table 5. Analyses of IBCSP-5 (Moisture Free Values)

	July, 1986	May, 1987		August, 1987
	Mean of 10 Analyses[1]	C26856		C27350
		ASTM[2]	LECO[3]	LECO[3]
Moisture	9.57	9.6	9.7	9.4
Volatile Matter	40.38	39.9	36.9	36.6
Fixed Carbon	41.61	42.2	44.6	45.1
HT Ash	18.00	17.9	18.5	18.3
Carbon	63.26	63.52	63.59	64.14
Hydrogen	4.40	4.23	4.23	4.72
Nitrogen	1.23	1.24	1.25	1.23
Oxygen	8.47	8.58	7.84	6.98
Sulfatic Sulfur[4]	0.00	0.00	0.00	0.00
Pyritic Sulfur	2.55	2.46	2.46	2.55
Organic Sulfur	2.08	2.07	2.13	2.08
Total Sulfur	4.63	4.53	4.59	4.63
Chlorine	0.09	0.11	0.11	.09
BTU/lb	11522	11466	11478	11613
FSI	3.83	3.5	3.5	4.0

1 Mean of 10 analyses (ASTM Proximate Analysis)

2 Moisture, Ash, Volatile Matter and Fixed Carbon obtained using ASTM methods of analysis. Other values determined as in column one, but adjusted for ASTM moisture value

3 Moisture, Ash, Volatile Matter and Fixed Carbon obtained using LECO MAC400 proximate analyzer, C, H, N determined using LECO 600, Other values converted to dry basis usng LECO moisture.

4 ASTM method used (D-2792) is subject to minor loss of $BaSO_4$ precipitation. A reported value of 0.00% can be as high as 0.04%.

Table 6. Analyses of IBCSP-6

	March, 1986 Mean[1]	Standard deviation	August, 1987 (C27351)
Moisture[2]	10.4	0.09	10.7
Volatile Matter	39.6	0.18	39.8
Fixed Carbon	51.4	0.17	51.0
H-T ash	9.0	0.07	9.2
Carbon[3]	71.63	0.260	72.63
Hydrogen	4.73	0.065	5.34
Nitrogen	1.77	0.051	1.52
Oxygen	9.08	0.320	7.51
Sulfatic Sulfur[4]	0.00	0.0	0.02
Pyritic Sulfur	1.83	0.053	1.94
Organic Sulfur	1.94	0.054	1.87
Py/org sulfur ratio	0.92		
Total Sulfur	3.77	0.070	3.83
Chlorine	.02	0.012	0.0
BTU/lb	13240	37	13232
FSI	4.7	0.26	4.0

[1] Avg of 10 analyses

[2] Proximate analysis by LECO Mac 400

[3] Ultimate analysis by LECO 600

[4] ASTM Method (D-2792) is subject to minor loss of $BaSO_4$ precipitation. A reported value of 0.00% can be as high as 0.04%.

Table 7. Minor and trace elements[1]

Oxide/element	IBCSP sample number 1	2	3	4	5	Average IL coal*
Minors (%)						
SiO_2	4.6	1.8	4.1	22.1	8.2	5.5
Al_2O_3	1.6	0.9	1.8	6.5	2.9	2.5
Fe_2O_3	1.7	2.8	1.5	3.8	3.4	2.7
MgO	0.09	0.038	0.073	0.529	0.185	0.046
CaO	0.5	0.2	0.1	1.4	1.2	1.0
Na_2O	0.139	0.0182	0.0297	0.337	0.168	0.0987
K_2O	0.21	0.11	0.2	0.99	0.33	0.22
P_2O_5	0.02	0.01	0.03	0.09	0.02	0.02
TiO_2	0.08	0.03	0.09	0.31	0.15	0.11
Traces (ppm)						
Ag	<1	<1	<1	<1	<0.2	0.06
As	2	32	16	5	2.6	11
Ba	32	14	28	135	73	140
Be	1.4	3.3	1.2	2.7	1.0	1.5
B	193	109	71	317	179	118
Br	6	3	12	3	6.5	12
Cd	1.1	0.8	0.1	<0.4	-	1.5
Ce	6	2	10	21	19	14.7
Co	3	6	5	9	3.8	5
Cr	31	7	16	44	19	18
Cs	1.1	0.8	1.2	4	1.9	1.0
Cu	9.7	21.9	8.0	14.4	9.5	12.5
Dy	0.6	1.5	0.9	1.7	-	1.1
Eu	0.2	0.2	0.2	0.5	0.2	0.3
F	63	26	56	460	-	68
Ga	3	3	3	10	3.4	3.9
Ge	<5	30	<5	<5	5	5
Hf	0.4	0.2	0.5	1.7	1.0	0.6
La	4	2	7	16	5.9	7
Li	11.3	18.1	29.9	38.9	8.2	16.3
Lu	0.1	0.1	0.1	0.3	0.08	0.1
Mn	31	16	13	112	71	55
Mo	15	4	13	6	9	9
Ni	11	22	14	23	15	18
Pb	8	149	57	28	6	28
Rb	9	5	11	63	20	16
Sb	0.2	3.4	1.1	0.3	0.3	1.0
Sc	2.1	2.1	2.6	6.4	2.4	2.7
Se	1.5	1.3	2.2	2.2	2.4	2.4
Sm	0.9	0.9	1.4	2.9	1.2	1.4
Sn	<1	<1	<1	1.7	<5	-
Sr	25	12	33	58	29	34
Ta	0.1	0.1	0.1	0.4	0.25	0.2
Tb	0.1	0.2	0.2	0.2	0.13	0.2
Th	1.2	0.7	1.3	3.9	3.2	2.2
Tl	<2	<2	<2	<2	1.0	1.0
U	<2	<1.5	<4	<3	1.2	1.5
V	25	22	26	50	23	31
W	<0.5	<0.5	<0.7	0.9	1.5	0.6
Yb	0.4	0.6	0.5	1.0	0.5	0.6
Zn	172	99.8	45.1	175	77	248
Zr	16	13	23	51	28	35

[1] From Harvey, et al., 1986.

samples in the air would not be necessary. Secondly, a larger up-front expenditure of funds would have been required to riffle all 48 barrels of coal. And finally, we wished to avoid riffling material that might be shipped by the barrel to some users.

All of the samples distributed to date have undergone a degree of exposure to air and loss of moisture during the processing into the 20-pound and 1-pound quantities. Additionally, air was not purged from the bags of coal when producing the coal for distribution. We believe the amount of oxygen in the interstitial space is small compared to the amount of oxidation that occurred during mining, washing, transport, crushing, screening, and riffling. Purging the interstitial space would change the moisture content, consume large volumes of purge gas and require additional labor for what was considered a minor benefit. The objective was the production of uniform samples (not pristine samples) at low cost with the facilities available. We believe additional oxidation after storage is minimal. We are satisfied to accept a low degree of oxidation in preparing these samples recognizing that this degree of oxidation may not be tolerable for some research. It has been reported by Duran, Mahasay and Stock (1986) that IBCSP-1, IBCSP-2, and IBCSP-3 contain elemental sulfur; 0.07%, 0.12% and 0.3%, respectively. This is contrasted to sulfur levels less than 0.0005% for three pristine samples collected for the PCSP program by Argonne National Laboratory. The results are interpreted to suggest that sulfur is not a natural constituent of coal, but is produced after exposure to air.

Analyses of the sulfatic and pyritic sulfur in IBCSP-2 have been unpredictable. Very early in the program, Dr. Muchmore at Southern Illinois University could not find sulfatic sulfur in IBCSP-2 from barrel #15 when he analyzed samples dried under vacuum. Sulfatic sulfur of the same coal retained in Champaign and subjected to a standard air drying procedure was reported to be 0.4 percent. A reanalysis of the coal in the same sample bottle after more time showed even higher values of sulfatic sulfur with complimentary lower values for pyritic sulfur. Low sulfatic sulfur values could also be gotten in Champaign if the coal was not given a customary overnight drying period in air, a practice used for years to let the coal samples reach a constant weight through moisture loss before weighing the sample on an analytical balance.

It has been the practice to store a few smaller bags of all IBCSP coals in a common barrel to minimize the number of barrels which must be opened and purged when samples are delivered to requesters monthly. Several bagged, 1-pound samples of IBCSP-2 from barrel #16 were among others stored in the common barrel. Despite careful purging of this barrel with nitrogen each time the barrel was opened to obtain samples for distribution, the sulfatic sulfur in IBCSP-2 samples in this barrel rose to 0.52 percent. A barrel holding the remaining riffled samples of IBCSP-2 samples, barrel #16 (unopened for more than a year) was opened and a sample was prepared for analysis. Its sulfate sulfur remained low (0.06%), even lower than the earlier sample and its FSI remained high.

Delivery of Samples to Requesters

A listing of the samples distributed to date is shown in table 8. Thirty-six individuals obtained one or more samples this year. Requests from 52 different users had been honored in all previous years of the programs history. The number of inquiries is increasing as the availability of samples becomes more widely known. All requests for samples have been honored, both those from research groups funded through the CRSC and from others as well. Most samples have been delivered within a month of the receipt of the request. Very little of IBCSP-5, the one for which a charge is made, has been delivered to requesters to date.

Sample Characterization

Chemical and petrographic analyses of IBCSP-5 screen fractions - In addition to providing samples from the IBCSP to researchers wishing to do research on Illinois Basin coals, the ISGS has provided sample preparation information to several researchers using the samples. Coal is a heterogenous mixture of organic and inorganic material. When preparing samples for precise research, proper procedures are necessary to avoid segregation of maceral types or minerals on the basis of friability and specific gravity. The mineral constituents vary in amount, hardness and patterns of distribution in the coal matrix. Coal researchers often wish to utilize size fractions for test work, or to determine particle size effects. When preparing size fractions, staged crushing is the best procedure to ensure that each fraction is as representative of the whole coal as possible and that the size fractions being compared are similar in composition.

The magnitude of the problem can be assessed by a screen analysis such as that shown in table 9 for IBCSP-5. This sample, packaged at Argonne National Laboratory, had been crushed to 100% minus 20 mesh. A 200 gram sample was dry-sieved for 20 minutes using a mechanical sieve shaker to obtain the fractions shown. Chemical analyses were performed on each fraction and petrographic analyses were performed on combined fractions (table 10).

The data reveal an enhancement of ash in the minus 200 fraction while fractions ranging from 35 x 48 to 150 x 200 mesh have mineral matter content lower than that for the feed coal. Total sulfur values also vary in each fraction. The petrographic analysis also shows differences in maceral composition of size fractions. Vitrinite is noticeably enhanced in the 100 x 200 fraction, liptinite in the +48 fraction and inertinite and minerals in the minus 200 fractions.

To minimize the possibility of maceral and mineral segregation during sample preparation, a stage crushing and screening technique should be used. This involves crushing (or grinding) to a size which leaves a majority of the sample coarser than the size fraction(s) desired. The sample is then sieved to obtain the material in the desired size range. The oversize material is then crushed to a slightly smaller size and the sample is resieved to obtain the newly produced material in the desired size range. This procedure continues until all the material is crushed to the desired size range. When more than one size fraction is desired,

Table 8. Samples delivered September 1, 1986 through August 31, 1987

Requestor	Institution	Location	Sample and Amount
A. Attar	Perfect View In	Rahleigh, NC	#2 – 5 lbs
D. Buchanan	Eastern Illinois University	Charleston, IL	#1 – 1 lb #2 – 1 lb #4 – 1 lb #5 – 1 lb #6 – 1 lb
D. Carr	Indiana Geological Survey	Bloomington, IN	#6 – 21 lbs
C. Chaven	ISGS	Champaign, IL	#3 – 3 lb
A. G. Comolli	Hydrocarbon Research Institute	Lawrencevill, NJ	#1 – 5 lbs #2 – 5 lbs
J. Conrad	ICT	Chicago, IL	#1 – 20 lbs
R. Davenport	Science Appl. In.	San Diego, CA	#3 – 1 barrel
P. Davis	SIU	Carbondale, IL	#3 – 1 barrel
D. Dollimore	University of Toledo	Toledo, OH	#1 – 1 lb #2 – 5 lbs #3 – 1 lb #4 – 1 lb #6 – 1 lb
P. Dussia	Atlantic Research Corp.	Alexandria, VA	#1 – 20 lbs #2 – 20 lbs #3 – 20 lbs
M. Elliot	Dept of Mines and Minerals	Benton, IL	#1 – 1 lb #2 – 1 lb #3 – 1 lb #6 – 1 lb
S. R. Gallahalli	U. of Oklahoma	Norman, OK	#3 – 50 lbs #4 – 50 lbs
R. Gupta	IIT	Chicago, IL	#2 – 280 lbs #4 – 80 lbs
K. Hackley	ISGS	Champaign, IL	#2 – 2 lbs
D. Harvey	ISGS	Champaign, IL	#5 – 1 pt #6 – 1 lb
L. Kohlenberger	ISGS	Champaign, IL	#5 – 1 pt
R. Knight	IGT	Chicago, IL	#1 – 20 lbs
C. Landis	Texas Tech	Lubbock, TX	#1 – 1 lb #2 – 1 lb #3 – 1 lb
J. Larsen	Lehigh University	Bethlehem, PA	#2 – 1 lb #3 – 1 lb #6 – 1 lb
D. Lee	U of Akron	Akron, OH	#1 – 11 lbs
O. Manuel	Univeristy of Missouri	Rolla, MO	#1 – 1 lb #2 – 1 lb #3 – 1 lb #4 – 1 lb #6 – 1 lb
V. Mathur	U of New Hampshire	Durham, NH	#5 – 4 pts
G. Olson	National Bureau of Standards	Gaithersburg, MD	#1 – 5 lbs #6 – 5 lbs
R. Prokopuk	Canadian Center for Mineral and Energy Technology	Ottawa,Ontario Canada	#1 – 20 lbs
L. Qioung	U. of I.	Urbana, IL	#1 – 20 lbs
S. Rajan	SIU	Carbondale, IL	#1 – 195 lbs #3 – 195 lbs
D. Rapp	ISGS	Champaign, IL	#3 – 1 lb
R. Read	ISGS	Champaign, IL	#4 – 43 lbs
A. Rohlik	Standard Oil-Ohio	Cleveland,OH	#4 – 1 lb
M. Rostam-Abadi	ISGS	Champaign, IL	#3 – 6 barrels #3 – 22 lbs
R. Shiley	ISGS	Champaign, IL	#1 – 5 lbs #2 – 5 lbs #4 – 5 lbs
S. L. Soo	UIUC	Champaign, IL	#1 – 20 lbs
M. Stephenson	ISGS	Champaign, IL	#2 – 20 lbs #4 – 41 lbs #5 – 1 pt #6 – 1 lb
M. Taylor	Bradley University	Peoria, IL	#1 – 1 lb #3 – 1 lb #4 – 1 lb #6 – 1 lb
T. Tewksbury	Battelle	Columbus, OH	#1 – 20 lbs #2 – 20 lbs #3 – 20 lbs #4 – 20 lbs
T. Wheelock	Iowa State U.	Ames, IA	#5 – 10 pints #5 – 5 gal

Table 9. Sieve analysis for IBCSP-5[1].
(Illinois No. 6 channel sample matching PCSP-3)

Sieve fraction Mesh	Sieve fraction wt %	Cumulative[2] passing wt %	Analysis Ash wt %	Analysis Sulfur wt %	Distribution[3] Ash wt %	Distribution[3] Sulfur wt %
+35	8.0	92.0	18.4	4.9	8.4	8.7
35x48	12.0	80.0	16.6	4.0	11.3	12.4
+48	**20.0**	**80.0**	**17.3**	**4.8**	**19.7**	**21.1**
48x65	14.2	65.8	15.3	4.6	12.3	14.2
65x100	14.1	51.7	15.1	4.5	12.1	14.0
48x100	**28.3**	**51.7**	**15.2**	**4.5**	**24.4**	**28.2**
100x150	12.3	39.4	14.8	4.5	10.3	12.0
150x200	9.8	29.6	15.4	4.7	8.6	10.1
100x200	**22.1**	**29.6**	**15.0**	**4.6**	**18.9**	**22.1**
-200	29.6	-	22.1	4.4	37.0	28.6

[1] Calculated combination of two size fractions appear in bold-faced type

[2] The cumulative wt % passing through successive screens from fine to coarse

[3] Distribution is the weight of the component in a given fraction divided by the total weight of that component in all fractions.

Table 10. Petrographic analysis of sieve fractions (Table 9)[1].

	Whole coal vol%	+48 Mesh vol%	48x100 Mesh vol%	100x200 Mesh vol%	-200 Mesh vol%
Vitrinite	76.5	77.0	78.7	80.5	72.0
Liptinite	3.0	5.5	3.3	2.7	1.4
Inertinite	8.8	5.1	7.4	6.5	11.3
Minerals	11.7	12.4	10.6	10.3	15.3

[1] The greatest change for each class is underlined

the sample is riffled into equal splits and each split is treated in the described manner.

To prepare these size fractions an adjustable roll crusher is needed for crushing samples of coarser sizes (28 mesh). For finer size material, a rod mill is used. A rod mill is preferable to a ball mill because it produces a narrower size distribution due to the nature of the grinding action. This results in less undersize material. For sieving, a set of sieves and a mechanical sieve shaker are needed.

When preparing multiple size fractions to study particle size effects, it is often useful to use sieves from the Tyler Standard screen series or the equivalent U.S. Standard sieves. For the Tyler Standard series, the ratio of edge lengths for the square holes in successive screens is the square root of 2 (1.4142). For closer sizing, intermediate screens are available with the ratio of edge lengths for successive screens in the Double Tyler series being the fourth root of 2 (1.1892). The utility of the square root of 2 series relates to the small whole number ratio it gives to properties that are directly related to a power of the average particle radius. For example, the average surface areas of particles ($A = \pi d^2$) in successive screen fractions will stand in the ratio of small whole numbers. Thus, the area of the opening for a 65 mesh screen is twice that of a 100 mesh screen and four times that of a 150 mesh screen.

Partially beneficiated IBCSP-4, deslimed and tabled - Some researchers wishing to work on an Illinois No. 6 coal have been hesitant to use IBCSP-4 because of its high percentage of ash-forming minerals. The sample is a run-of-mine coal, and much of the ash is due to the clay material that was taken from above and below the seam in the mining process. The amount of ash-forming minerals is readily reduced in one of two ways; desliming (wet screening on a fine screen to remove the clay) or tabling on a gravity table. Desliming reduces the amount of ash-forming minerals without reducing the amount of pyrite, whereas tabling rejects both ash forming minerals and pyrite. These processes were used to produce samples representing more than one level of physical cleaning for a project investigating the effects of cleaning levels on process variables. The desliming step consists of sieving at 200 mesh after agitating the sample in warm water for 5 minutes. Tabling of a 100% minus 6-mesh feed produces a product with relatively small amounts of free pyrite (26% on a diameter basis) and mean grain diameter of 4.9 microns for the free pyrite and 7.7 microns for the enclosed pyrite. Material balances for each method of treatment are shown in tables 11 and 12. These simple steps can increase the versatility of IBCSP-4 substantially by enabling researchers to alter the mineral matter composition to fit specific research requirements.

Special Services

Sample preparation information and special services were provided to several CRSC funded investigators in the past year.

1. A sieve analysis was performed for Dr. Rostam-Abadi, ISGS, on ICSP-3 to determine this coal's suitability for charring in a United Coal Company pyrolysis unit. Additionally, information on the proper riffling procedures and sample storage techniques were provided to Dr. Rostam-Abadi.

2. A series of wet sieve analyses were performed for Professor Buckius on IBCSP-1 and IBCSP-3 for his project Flammability Characteristics of Desulfurized Illinois Coal. Also, arrangements were made to provide Professor Buckius a gravity-cleaned sample and a sample cleaned by Aggregate Flotation for use in combustion testing.

Table 11. Material balance for desliming of IBCSP-4 at 200 mesh.

Fraction	Weight %	Ash %	Pyritic Sulfur %	Total Sulfur %	BTU
ANALYSIS:					
+200	66.43	18.34	2.87	5.67	11554
-200	33.57	78.74	1.39	1.73	2110
DISTRIBUTION:[1]					
+200	66.43	31.55	80.35	86.64	91.55
-200	33.57	68.45	19.65	13.36	8.45
	100.00	100.00	100.00	100.00	100.00

[1] Weight percent of component in the given fraction

Table 12. Material balance for tabling IBCSP-4[1].

Fraction	Weight %	Ash %	Pyritic Sulfur %	Total Sulfur %	BTU
ANALYSIS:					
Coal concentrate	63.77	16.1	1.19	3.44	11836
Middling (ash/coal)	5.10	61.80	2.69	3.43	4709
Pyrite conc.	17.00	80.00	7.48	8.00	1814
Slime	14.13	59.70	0.90	1.78	5211
DISTRIBUTION:[2]					
Coal concentrate	63.77	28.98	33.12	55.28	85.45
Middling (ash/coal)	5.10	8.89	5.97	4.38	2.72
Pyrite conc.	17.00	38.34	55.37	34.04	3.49
Slime	14.13	23.79	5.54	6.30	8.34
	100.00	100.00	100.00	100.00	100.00

[1] Size 100% minus 6 mesh

[2] Weight percent of component in the given fraction

3. Pyrite concentrates were provided to Dr. Miller (ISGS) and Dr. Fitzpatrick (Northwestern University) for use in their project investigating microbial enhancement of pyrite suppression. This sample was derived from IBCSP-4.

4. A pyrite concentrate was provide to Dr. Chaven (ISGS) for use in his forms of sulfur project. This sample was also derived from IBCSP-4.

5. Mr. Mike Stephenson (ISGS) was informed that IBCSP-4 (high ash run-of-mine sample) could be beneficiated by desliming and tabling to make it more suitable for charring studies. This information was used on his project, Desulfurization of Illinois by Thermal, Chemical and Magnetic Methods.

Advisory Committee for the IBCSP

One representative from each of the following organizations is a voting member: the Center for Research on Sulfur in Coal, the Indiana Geological Survey, Argonne National Laboratory, Southern Illinois University, and the Illinois State Geological Survey. The sixth voting member is a private consultant (a geologist) who is also a member of the Illinois Coal Development Board. This committee met twice this year to make recommendations on policy matters including what coals should be stocked, requirements for receiving samples and the amount to be charged requesters. A questionnaire was developed to obtain guidance from users of the program and prospective users. More than 1000 copies were mailed in June. Approximately 80 have been returned. Analysis of the data is incomplete. Results will be one of the factors used to select the future coals to be put into the IBCSP.

CONCLUSIONS AND RECOMMENDATIONS

The properties of the coal samples have remained remarkably constant and the Program is meeting its objectives. The Illinois Basin Coal Sample Program is meeting a need for samples of coal and is promoting a sharing and comparison of results among research groups working on Illinois coals. The number of research groups needing special assistance in preparing samples for their research has grown. These special services together with assistance about how to properly sample and protect reactive, heterogenous materials may be an advisable function of an effective coal sample program serving laboratories which do not have equipment or trained personnel for grinding, crushing, and sieving coal and for preparing concentrates of certain fractions of coal.

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Exhibit A

ILLINOIS BASIN COAL SAMPLE PROGRAM

Program Director: Carl W. Kruse (217)-333-5161)

REQUEST FOR COAL SAMPLE

Samples available (Note - chemical data are given on the reverse side)

Sam. no.	Product	Seam	Location	Rank	Ash (% dry	Sulfur basis)	Ratio Py/OrS
1	Prep plant	Illinois No. 6	W. Central IL	HVCB	10.3	4.3	0.4
2	Prep plant	Illinois No. 2	Western IL	HVCB	6.7	3.2	2.5
3	Prep plant	80% Illinois No. 5 20% Illinois No. 6	Southern IL	HVBB	8.4	2.3	0.9
4	Mine-run	Illinois No. 6	Southwestern IL	HVCB	38.1	4.2	1.3
5[1]	Channel	Illinois No. 6	Southwestern IL	HVCB	18.0	4.6	1.2
6	Prep plant	Indiana V	Southwestern IN	HVCB[3]	9.0	3.8	0.9

[1] A pristine sample processed for the IBCSP by Argonne National Laboratory in a nitrogen atmosphere. The charges (for sample #5 only) are $10 per 1-pt can, $50 per 1-gal can and $200 per 5-gal can.

[2] Indiana V seam is the Springfield (No. 5) seam in Illinois

[3] Borderline between HVBB and HVCB

All samples are delivered under nitrogen seal. You will be responsible for maintenance of the sample after you break the seal. Subsamples should be prepared by riffling and the sample container should be resealed.

You will be provided recent analytical results for the sample(s) requested. In return, please provide information on the behavior of the sample(s) in your tests or reprints of your publications which give your results.

	IBCSP SAMPLE NO.					
1. Circle the sample(s) you request:	1	2	3	4	5	6
2. Circle the amount needed in lbs. (except for sample no. 5)	1	1	1	1	1-pt	1
	5	5	5	5	1-gal	5
	20	20	20	20	5-gal	20
Other amounts, please specify:	—	—	—	—	—	—

3. Give the title of your project:

4. Briefly describe the objective of your project:

Your name: Date:

Address:

Return to: Illinois State Geological Survey
615 E. Peabody Drive
Champaign, IL 61820
Attn: Mr. David Rapp (217/244-4998)

(March 1987)