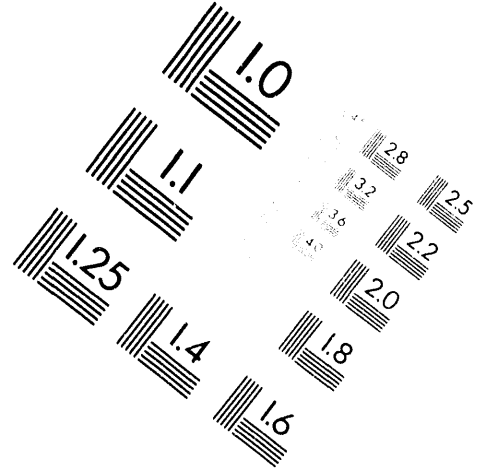
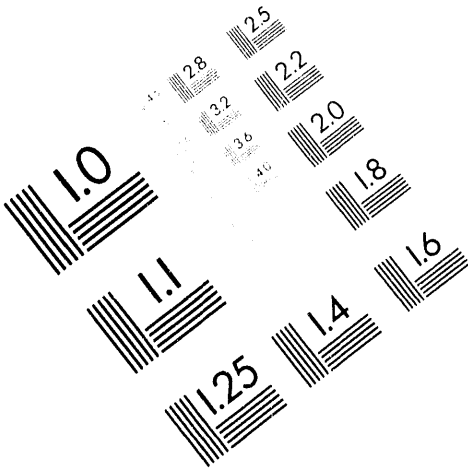




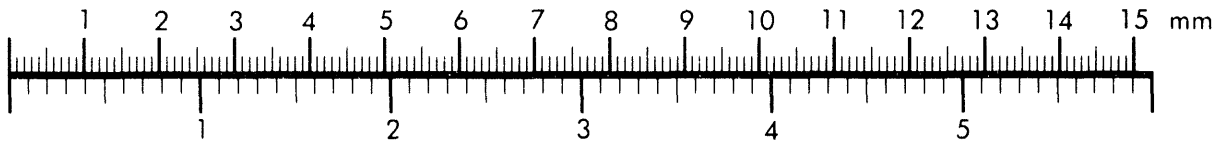
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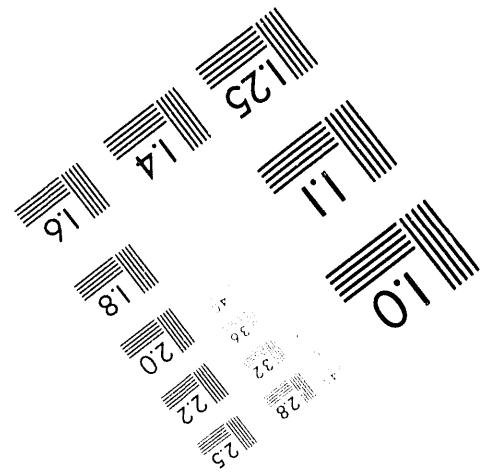
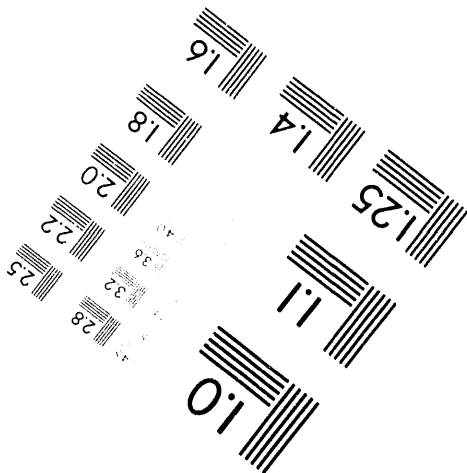
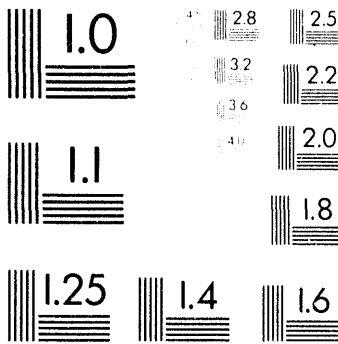
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DOE/PC/92521--T/50

TECHNICAL REPORT

December 1, 1993 through February 28, 1994

Project Title: **WASHABILITY OF TRACE ELEMENTS IN PRODUCT  
COALS FROM ILLINOIS MINES**

DOE Grant Number : DE-FC22-92PC92521(YEAR2)  
ICCI Project Number : 93-1/6.2A-1P  
Principal Investigator: Ilham Demir, ISGS  
Co-investigators : Rodney R. Ruch, Richard D. Harvey,  
John D. Steele, and Saleem Khan, ISGS  
Project Manager : K. K. Ho, ICCI

ABSTRACT

The existing trace element washability data on Illinois coals are based on float-sink methods, and these data are not applicable to modern froth flotation or column flotation processes. Particularly, there is a lack of washability data on samples from modern preparation plants, as well as other product (as-shipped) coals. The goal of this project is to provide the needed trace element washability data on as-shipped coals that were collected during 1992-1993 from Illinois mines. The results generated by this project will promote Illinois coals for utilization in prospective markets such as advanced gasification processes and the production of synthetic organic chemicals, and to meet new environmental requirements for their use in utility steam generation.

During the second quarter, froth flotation/release analysis (FF/RA) tests on 34 project samples were completed at -100, -200, and -400 mesh particle sizes. Products from the FF/RA tests were analyzed for ash, moisture, and some for total S and heating value (BTU), and the resulting data are being used to construct a series of washability curves. For example, these curves can show variation in BTU or combustible recovery as a function of the amount of ash or S rejected. Composite samples, each having 80% of the total BTU (or combustibles), were prepared for the -100 and -200 mesh FF/RA tests and submitted for trace element analysis. The composite samples for the -400 mesh FF/RA tests will be submitted soon, and the analytical results are expected to be available in 3-4 months. The trace element data on the composite samples will indicate the potential for the removal of each element from the coals at the chosen flotation conditions and particle sizes.

Two splits (-100 mesh and -400 mesh) of one of the product coals were subjected to FF/RA tests, and all the products from these tests were submitted for trace element analysis. The results will permit the computation of the mass balance for individual trace elements that can be achieved under the FF/RA test conditions used.

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## EXECUTIVE SUMMARY

The goal of this project is to generate advanced trace element washability data on product (as-shipped) coals from Illinois mines. The results will indicate how much further advanced cleaning techniques can reduce the trace element levels in the marketable coals that have already been cleaned with conventional cleaning techniques. With the advent of new and improved coal cleaning technologies and expanding opportunities for reaching foreign markets, the availability of advanced washability data will help promote Illinois coal products. The project results will provide advanced washability data for evaluation of trace element emissions at power generating plants. In addition, the results will facilitate national and international recognition of further processability of product coals from Illinois to be used as feed materials for advanced gasification processes and for production of synthetic organic chemicals.

Given the sensitive nature of the data to be generated by this project, only averages of the results for multi-county regions of the state will be reported. The results on individual mines may be reported by laboratory numbers without revealing the identity and location of the mines.

During the second quarter of the project, grinding 34 project samples to three different sizes (-100, -200, -400 mesh) and subjecting the ground samples to froth flotation/release analysis (FF/RA) tests were completed. The FF/RA tests were carried out at -100 mesh and -200 mesh sizes for all 34 samples and at -400 mesh size for 6 selected samples. Resources saved by omitting some of the -400 mesh runs were directed towards 1) expanding trace element mass balance characterization, 2) increasing the number of concentrates produced in each FF/RA test to obtain better washability curves, and 3) increasing the number of heating value (BTU) determinations to establish better empirical relationships among BTU, combustibles, and ash contents of the samples.

Chemical analyses of all the flotation products (5 concentrates and a tail from each test) for ash, moisture, and some for BTU and total S were completed. The ash, BTU, and total S data are used to construct a series of washability curves. For example, washability curves for %BTU or combustible recovery as a function of the amount of ash or S rejected, or as a function of the weight of the flotation products, can be generated.

Using a washability curve indicating relationship between % cumulative BTU or combustibles and % cumulative weight of flotation products, composite samples (each having 80% of the total BTU) were prepared. To date, sixty eight composite samples were prepared from the products of the -100 and -200 mesh FF/RA tests and submitted for trace element analysis.

The remaining composite samples for the -400 mesh FF/RA tests will be prepared and submitted early in the next reporting period. The trace element analyses on the composite samples will be available in 3 to 4 months. These data will then be compared to each other and to trace element data obtained previously on the as-shipped coals that were used as feeds for the FF/RA tests. The results from trace element analyses of the 80%-BTU recovery composite samples will indicate the beneficiation potential for each element at the generalized flotation conditions and mesh sizes used.

One feed sample with -100 mesh size and another with -400 mesh size, produced by grinding one of the 34 project samples, were cleaned using the FF/RA procedures in an attempt to achieve mass balances for the trace elements. All solid float and sink fractions, representative samples of the water used for grinding and froth flotation, and a representative feed coal for each FF/RA run were submitted to be analyzed for trace elements, ash, total sulfur, forms of sulfur, and chlorine. The data from this set of analyses will be displayed as to indicate the beneficiation obtained in the progressive clean concentrates and tail fractions. The results will also be compared to the 80% composite data available from the previous tasks.

#### **DISCLAIMER**

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

## OBJECTIVES

The main objective of this project is to generate advanced trace element washability data on available coals or other types of marketed material from operating mines in Illinois. The availability of trace element washability data will permit a wide variety of advanced engineering and environmental calculations, such as improved capabilities to match specific coals for 1) prospective markets such utilization in advanced gasification processes and in the production of synthetic organic chemicals, and 2) meeting environmental requirements for noxious emissions at power generating plants.

To meet the stated objective the following specific tasks were identified.

1. Quality control/quality assurance (QC/QA). A comprehensive QC/QA plan, already in place for ISGS work, are modified and applied to all phases of this project.
2. Sample preparation. We already collected all the samples needed for this project. The samples are currently stored under nitrogen gas in our laboratory. Representative splits of the samples are ground to three different sizes (-100 mesh, -200 mesh, -400 mesh) for the proposed washability tests.
3. Washability tests. This research project will evaluate the trace element washability of coal at fine to ultrafine particle sizes with special regard for their industrial application.
4. Analyses of the products from washability tests for moisture, ash, heating value, and trace elements.

## INTRODUCTION AND BACKGROUND

With the advent of new and improved technologies and expanding possibilities for exporting to foreign markets, and with recent environmental legislation, the availability of trace element washability data would help promote Illinois coal products. The data generated by this project will enhance the knowledge that could be provided to coal-producing and -utilization companies and help open up new market possibilities for the utilization of Illinois coals. The data will also be useful for an environmental evaluation that is needed in response to new federal air quality legislation.

The levels of washability efficiency obtained by traditional density or float-sink separation of fine coal in the laboratory may not be achievable by industrially practicable cleaning procedures. It is important, therefore, to develop

a washability database that has industrial application. The washability testing process we use has potential economic and commercial feasibility. The proposed process utilizes multi-stage flotation in a standard sub-aeration flotation cell to generate washability curve, which have been demonstrated to correlate to industrially feasible cleaning procedures, namely froth flotation and column flotation.

### Related Research

**Trace elements in coal.** A study by the U.S. National Committee for Geochemistry [National Research Council (NRC), 1980] discussed the potential health hazards of various trace elements that might be encountered during the development and utilization of coal. Three categories of elements of environmental concern were identified with respect to coal:

- Of greatest concern - As, B, Cd, Pb, Hg, Mo, and Se
- Of moderate concern - V, Cr, Ni, Cu, Zn, and F
- Of minor concern - Li, Na, Sr, Ba, Mn, Co, Ge, Cl, Br, and radioactive elements Ra, Po, Rn, Th, and U

These judgments were based upon known toxicity, levels of occurrence of each element in coal, and the anticipated mobility upon combustion or disposal of ash. These recommendations are only guidelines and eventual emission standards should be based on valid biological data.

The Clean Air Act Amendments of 1990 [Public Law 101-549, 1990] lists 18 trace elements as "Hazardous Air Pollutants" (HAP): As, Be, Cd, Cl, Cr, Co, F, Hg, Mn, Ni, Pb, Po, Ra, Rn, Sb, Se, Th, and U. A parallel regulation is also underway in Illinois [Illinois Pollution Control Board, 1990]. All of these HAP elements are present in Illinois and other coals [Gluskoter et al., 1977; Swanson et al., 1976; Zubovic et al., 1979, 1980; Cahill et al., 1982; Harvey, et al., 1985] and their concentrations vary considerably. Utilities are presently exempt from having to comply; however, this may eventually change after the U.S. EPA completes its risk studies and establishes emission standards as prescribed by law. This process may take years, but eventually emission limitations on all or some of these elements will likely be imposed. The ultimate burden to coal-burning utilities may eventually be to determine the amounts of each HAP element actually being emitted to the atmosphere for a particular coal under the combustion conditions in which they operate.

Mass balance studies on the Allen cyclone boiler power plant at Memphis, Tennessee, using coals from Illinois and western Kentucky, indicated that most coal-derived Hg, some Se, and probably most Cl and Br are released as gases to the

atmosphere [Klein et al., 1975]. . The elements As, Cd, Cu, Ga, Mo, Pb, Sb, Se, and Zn were concentrated in the fly ash and are partially released to the atmosphere by this mechanism. The electrostatic precipitator removed about 96.5-99.5% of the fly ash on the two runs that were tested. Kaakinen et al. [1975] measured the concentrations of 17 elements in a mass balance study of a power plant fueled with a western coal. The elements Pb, Cu, Zn, As, Mo, Hg, and Se were found to be partly volatile. Natusch et al. [1974] independently observed that certain elements, especially As, Sb, Cd, Pb, Se, and Tl, are more concentrated on the smaller, respirable-sized fly ash particles.

Swaine [1989] reviewed some environmental aspects of trace elements in coal. With respect to combustion, modern electrostatic precipitation systems are able to trap up to 99% of the fly ash. Thus, trace elements that "escape" during combustion are either attached to ultra fine fly ash particles or in a gaseous state. Swaine concluded that, in general, no trace element posed a significant environmental problem. This assumes the use of state-of-the-art electrostatic precipitators and only burning coals with no exceptionally high concentrations of those noxious elements that would be emitted in a gas phase or attached to untrapped fly ash particles that pass through electrostatic precipitators.

**Removal of trace elements from coal.** Over the years a number of studies have resulted in the evaluation of trace element removal from coal as a result of the particular cleaning method utilized. Most certainly deep physical cleaning of coal should significantly reduce the level of most trace elements [Gluskoter et al., 1977; Cavallaro et al., 1978; Harvey et al., 1983] as shown by float/sink testing. However, there has been a lack of data on the trace element washability of product coals from modern preparation plants. In general, physical cleaning is limited and only becomes efficient (70-90% removal) if a high degree of comminution is utilized, freeing the mineral matter from the macerals. Those trace elements organically bound or associated with the macerals are not removed; their concentrations are actually enhanced in the cleaned coal. Studies tracking trace element removal using chemical means indicate good removal; however, costs would be quite high.

Various reviews have summarized progress in the removal of trace elements via coal cleaning practice. These include Jacobsen et al. [1992], Norton et al. [1992], Kaiser Engineers [1989], Norton et al. [1989], Streeter [1986], Norton et al. [1985], Wheelock and Markuszewski [1981, 1984], National Research Council [1980], National Academy of Science [1979], and Mezey [1977].

Float-sink gravity separation studies have shown that significant reduction can be achieved for some trace elements through conventional coal cleaning; however, the coarse particles (~ 3/8") generally limit the amount of liberation. Extensive work done at the ISGS by Gluskoter et al. [1977], Fiene et al. [1979], and Harvey et al. [1983], and on Illinois coals indicate many elements (As, Ba, Ca, Cd, Fe, Mn, Mo, Pb, Tl, and Zn) have strong inorganic association and at 80% recovery can be significantly (~50% or greater) removed. Other elements (B, Be, Ge, Ni, Sb, U, and V) are strongly associated with organic matter and are generally not removed by physical cleaning. The behavior of some 32 other elements during float-sink separation indicated mixed association. Kuhn et al. [1980] similarly characterized an eastern seam (Pittsburgh 8) and a western seam (Rosebud), indicating differences in trace element/mineral association and possible cleaning potential. For example, in the Rosebud coal, Sb was classified to be associated with the organic fraction, not with the inorganic fraction (as with Illinois Basin coal). Cavallaro et al. [1978] reported on the float-sink behavior of some eight elements (Cd, Cs, Cu, F, Hg, Mn, Ni, and Pb) in 10 coals from various producing areas of the U.S. Results indicated that although the concentration of individual trace elements in the feed coal varied from region to region and within a region, the removal of the heavier fraction (> 1.6 S.G.) would result in significant reduction in the 14-mesh clean coal product for all the coals tested. The data indicated that 64 to 88% composite trace element concentrations were in the 1.6 S.G. sink fraction depending upon the geographical region.

Akers and Dospay [1992] reported washability data on an Upper Freeport coal for As and Ni at several size fractions indicating better removal at the smaller sizes. They also presented data on reductions for some 10 trace elements for four coal seams at several energy recoveries using conventional gravity separations.

Ford et al. [1976] demonstrated that rough coal cleaning at a top size of 30 mesh via a concentrating table was effective in partially removing Hg, Pb, As, Mn, and Se from eight diverse coal types. The percentage ranges of removal were reported for Hg (3-68%), Pb (8-63%), As (11-67%), Mn (9-76%), and Se (2-61%). Ford and Price [1980] reported on results of conventional as well as non-conventional coal cleaning of some 20 run of mine coals. The elements As, Cd, Cr, Co, Cu, F, Pb, Mn, V, Zn and most major ash elements were significantly removed. Ford and Price [1982] reported on trace element removal via heavy media cyclone cleaning. Averages of the % original amount of trace elements that remained in clean coal fractions for four Appalachian and two midwestern coals were as follows: As (53), B (98), Be (75), Cd (54), Co (63), Cr

(60), Cu (60), F (49), Hg (89), Mn (37), Ni (62), Pb (32), Sb (91), Se (67), V (76), Zn (51). Conzemius et al. [1988] determined the fate of 75 elements after cleaning via heavy medium cyclone. Although the concentrations of most elements followed the ash content, the cleaned coal was enriched in Be, Mo, U, I, Co, and Sb.

Kulinenko and Barma [1990] used a combination of screening, density separation, jigging, flotation, centrifugation and vacuum filtration and monitored some 29 trace elements in the various streams. Arsenic and Hg were most abundant in jigging refuse. Fluorine and Ga were most abundant, and Hg, Be, and Ge were least abundant, in density separation refuse. Cesium was least abundant in flotation refuse.

Capes et al. [1974] reported that levels of As, Cs, Pb, Mn, Mo, Ni, and V were reduced by 50-80% during separation by oil agglomeration. Knott et al. [1985] reported on trace element reduction comparisons obtained by oil agglomeration, froth flotation, and float-sink processes on Australian coals. The OTISCA process, an oil agglomeration approach utilizing a fluorocarbon liquid (freon), [Jacobsen et al., 1992] indicated >90% removal of As, Cd, and Pb from coal.

A combination process of froth flotation and oil agglomeration called "aggregate flotation" was able to remove greater than 50% of Cd, Co, Cr, Li, Mn, Na, Sb, Sr, Th, and V from Illinois coals [Buckentin et al., 1985].

A combination of heavy media cyclone or table separation and advanced froth flotation approach utilizing microbubble processing on fine size Illinois coal [Bechtel National, Inc., 1988] achieved over 80-90% removal for As, Be, Co, F, Ni, Pb, Sb, Th, U, Cr, and Sc.

The LICADO process [Jacobsen, et al., 1992] based upon surface-separation using liquid CO<sub>2</sub> indicated only moderate separation for As, Ni, and Pb.

Limited data for two caustic leaching processes (the Battelle hydrothermal and TRW/Molten-caustic leaching) [Jacobsen et al. 1992] show high removal of Pb but mixed reduction for Cd, Ni, Se, and As. Data on the Battelle Alkaline Desulfurization process for three Ohio coals indicate high (> 70%) removal for Li, Ba, Be, B, K, P, Mo, V, As, and Ba [Mesey, 1977; Stambaugh et al. 1979].

The Jet Propulsion Laboratory chlorinolysis process is run at 50°-100° C, passing chlorine gas through a fine-ground slurry of coal, organic solvent, and water [Jacobsen et al., 1992; DuFresne and Kalvinskas, 1980]. Available data indicated high removal for Pb, medium removal for As, and low removal for

Cd, Hg, and Se.

The Meyers process [Mesey, 1977; Hamersma et al., 1974] uses and regenerates  $\text{Fe}_2(\text{SO}_4)_3$  and is said to effectively remove pyrite. In a study of some western, eastern interior, and Appalachian coals, As, Zn, Cd, Pb, Ni, Mn, Cr and other elements normally known to be associated with pyrite and other minerals were significantly reduced. The data capability for B, Be, Cu, F, Hg, Li, and V was somewhat mixed.

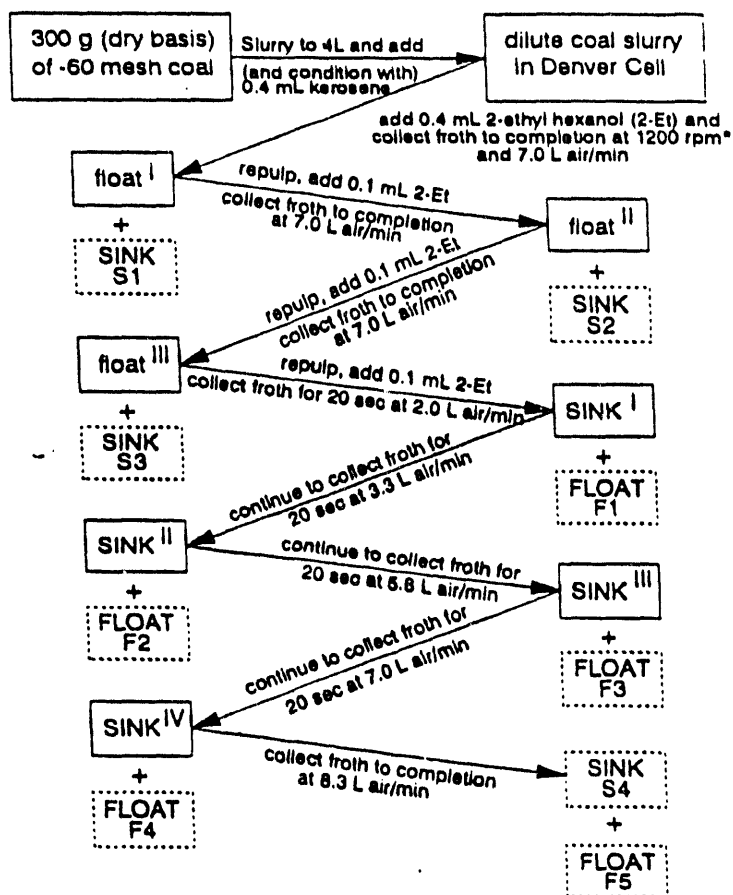
Norton et al. [1986] reported on the comparative treatment of the Herrin (Illinois No. 6) coal using molten NaOH/KOH or 1.0 M  $\text{Na}_2\text{CO}_3$  addition under elevated temperature/pressure conditions followed by acid wash. Levels of Mn, Pb, Rb, Sr, and Zn were lowered by 75% or more, while those of Ba, Cd, Cr, Ni, Se were lowered by 30-60%. They also reported on results obtained by the TRW Gravimelt process where the levels of As, Be, Cd, Hg, Pb, Se, and Sr were reduced by 75% or more. Kuhn et al. [1980] reported on "mineral free" values of some 52 trace and minor elements in eastern and western coals that were subjected to float-sink separation and extensive extractions with dilute  $\text{HNO}_3$  and HF, followed by washing with water. This approach indicates the amount of trace elements that would remain in the coal after an exhaustive physical and chemical cleaning.

In a previous project funded by the ICCI, we generated data on trace elements in product coals from operating Illinois mines in addition to compiling existing data at the ISGS [Demir et al., 1993]. Thirty four product coal samples from operating Illinois mines were collected, processed, and analyzed for trace, minor, and major elements. These coal data will be useful in predicting the highest order of magnitude (worst scenario) for individual trace element emissions that would be derived from burning various Illinois coals. This current project characterizes the washability of 29 trace elements of some environmental concern in all 34 product coals collected during the previous project. Because of the proprietary nature of the data, average values will be reported for at least 4 multi-county regions of the Illinois coal field and individual mine results will be reported only with sequential sample numbers without identifying the mines.

## EXPERIMENTAL PROCEDURES

### Sample Preparation and Froth Flotation/Release Analysis Tests

**-100 mesh samples.** The details of the procedures for this task were included in the first quarterly report [Demir et al., 1993]. The froth flotation/release analysis (FF/RA) technique that was applied to -100 mesh, as well as -200 and -400 mesh, samples is illustrated in Figure 1.



\* all Denver cell runs were at 1200 rpm

**Figure 1.** Froth flotation and release analysis approach.

**-200 mesh samples.** About 700 grams of coal in each case was mixed with 700 grams of water and ground in a rod mill generally for 30 minutes. The coal slurry was filtered, split in half, and a one half fraction was cleaned using the FF/RA procedure shown in Figure 1 with a few reagent concentration changes. In general 0.7 ml kerosene was used along with 0.5 ml frother in the first flotation. In some cases additional kerosene had to be used in order to wet the coal and prevent excess amounts of tails. A split of each of the ground feed coal was tested on a Microtrac Particle Size Analyzer for particle size distribution.

**-400 mesh samples.** To achieve -400 mesh particle size, about 700 grams of each coal was mixed with 700 grams of water and ground in a rod mill generally for 60 minutes. The coal slurry was filtered, split in half, and a one half fraction was cleaned using the same procedure described in Figure 1 with a few changes in reagent concentration. In general 1.0 ml kerosene was used along with 0.5 ml frother in the first flotation. In some cases additional kerosene had to be used in order to wet the coal and prevent excess amounts of tails.

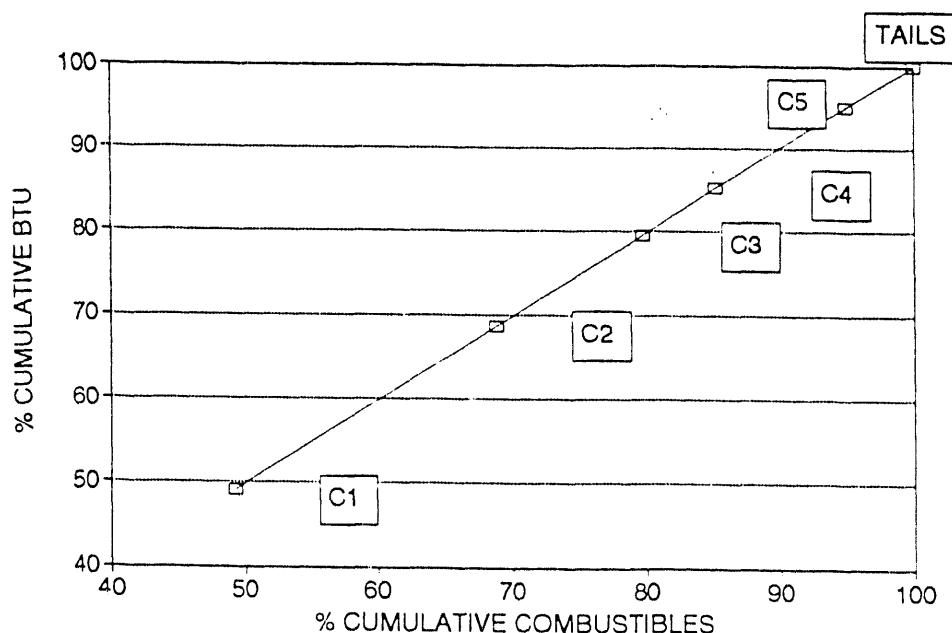
A split of each of the ground sample was analyzed for particle size distribution on a Microtrac Particle Size Analyzer.

All the concentrates and sinks from the FF/RA tests on the -200 and -400 mesh samples were submitted for ash, moisture, heating value (BTU), and total sulfur analyses in accordance with established ASTM procedures [ASTM, 1992].

## RESULTS AND DISCUSSION

### Progress on -100 Mesh Samples

During this reporting period, the preparation of composite samples of 80%-BTU (or combustibles) recovery from the products of FF/RA of all 34 -100 mesh feed coals was completed. Because there is a 1 to 1 linear relationship between BTU and combustibles, as indicated by an example in Figure 2, either value can be used to prepare the composites. The composite samples were prepared using computer plots indicating relationship between % cumulative BTU or combustibles and % cumulative weight of flotation products. This procedure was discussed in detail in the first quarterly report [Demir et al., 1993]. All composite samples were submitted for trace element analysis. The trace element data, expected to be available in 3 to 4 months, will be graphed in an appropriate manner as to show the degree of beneficiation for each element in each sample, as compared to the feed coal and also to corresponding data to be obtained on the -200 and -400 mesh FF/RA separations.



**Figure 2.** Relationship between heating value (BTU) and combustibles for the sample C32776, ground to -100 mesh.

### Progress on -200 Mesh Samples

Grinding the project samples in a rod mill for 30 minutes resulted in particle sizes of 90% -200 to -270 mesh (Table 1). The FF/RA separation testing was completed on all 34 -200 mesh feed samples. Eleven of the FF/RA tests had to be rerun because of low combustion yields due to partial non-wetting which resulted from the significantly smaller particle size and increased surface area of the coal particles during pulping. This was generally corrected by adding additional

**Table 1.** Particle size analysis of the 34 product coals ground in a rod mill for 30 minutes

Sample no.	Coal region*	90%passing micron size	90%passing U.S. Mesh size	Average micron size	80%passing U.S. Mesh size
C32773	1	55	270	25	325
C32774	1	54	270	24	325
C32777	1	73	200	31	230
C32778	1	52	270	23	325
C32782	1	61	230	29	325
C32783	1	64	230	31	325
C32785	1	63	230	30	325
C32797	1	73	200	34	230
C32814	1	76	200	34	230
C32779	2	58	230	27	325
C32794	2	59	230	30	325
C32798	2	48	270	22	400
C32800	2	62	230	30	325
C32813	2	56	230	27	325
C32815	2	65	230	30	325
C32784	3	61	230	32	325
C32795	3	76	200	36	230
C32796	3	46	270	31	400
C32799	3	53	270	25	325
C32801	3	71	200	33	230
C32802	3	46	270	23	400
C32803	3	57	230	27	325
C32661	4H	70	200	31	325
C32664	4H	64	230	31	325
C32665	4H	66	200	31	325
C32771	4H	61	230	30	325
C32776	4H	56	230	26	325
C32662	4S	59	230	28	325
C32663	4S	63	230	30	325
C32772	4S	59	230	27	325
C32775	4S	57	230	26	325
C32780	4S	52	270	23	325
C32781	4S	52	270	23	325
C32793	4S	58	270	26	325

\* 1, NW region; 2, SW region; 3, SC region; 4H, SE-Herrin Coal region; 4S, SE-Springfield Coal region. See also Demir et al. [1993].

kerosene and increasing the aeration rate during the initial separations. Analyses for ash and moisture of all concentrates and sinks from the FF/RA tests were completed. All 34 composite samples of 80%-combustibles recovery were prepared and submitted for trace element analysis.

Table 2 compares the %cumulative ash in 80% cumulative combustibles samples for -100, -200 and -400 mesh FF/RA runs. The results in Table 2 indicate much better beneficiation for the -200 mesh samples than for the -100 mesh samples. This relative beneficiation is consistent with the increased liberation of the mineral matter via grinding. It is anticipated that those trace elements associated with the liberated ash will be beneficiated accordingly.

**Table 2.** Percent cumulative ash for 80 percent cumulative combustibles for the -100 and -200 mesh composite samples

Sample no.	% ash in feed	% cum ash in -100 mesh composite	% cum ash in -200 mesh composite	% cum ash in -400 mesh composite
C32661	8.17	55	32	
C32662	7.00	52	41	
C32663	8.96	64	47	
C32664	9.87	68	48	
C32665	9.39	56	41	
C32771	12.57	46	29	
C32772	9.33	54	39	
C32773	8.72	51	38	
C32774	7.07	40	35* (25@77)	24
C32775	7.67	66	51	
C32776	9.27	45	30	
C32777	14.52	43	33	28
C32778	9.80	44* (34@70)	50* (26@70)	
C32779	9.63	50	34	
C32780	9.57	53	38	
C32781	9.71	63	52	
C32782	11.62	48	35	
C32783	12.86	51	34	
C32784	8.13	56	34	
C32785	9.75	52	36	
C32793	14.14	25	15	11
C32794	10.52	53	35	
C32795	5.76	57	39	
C32796	16.10	50	23	
C32797	10.29	46	29	
C32798	13.16	37	28	21
C32799	11.42	46	24	
C32800	11.39	50	33	
C32801	8.36	46	39	28
C32802	9.36	48	30	
C32803	9.19	49	34	
C32813	14.70	41	25	
C32814	6.00	54	42	
C32815	12.03	41	28	

\*tails had to be added to C1-C5 concentrates to attain 80% combustibles

### Progress on -400 Mesh Samples

The FF/RA separation testing was completed on six selected samples that had a particle size distribution of 80% to 90% -400 mesh (Table 3). The concentrates and sinks from the FF/RA tests were submitted for ash analysis. The six samples ground to -400 mesh were selected to represent each major coal seam, 4 coal regions, and low, medium, and high S and Cl coals.

**Table 3.** Particle size analysis of six product coals ground in a rod mill for 60 minutes.

Sample no.	Region	90%passing micron size	90%passing U.S. MESH size	Average micron size	80%passing U.S mesh size
C32774	1	38	400	17	500
C32777	1	46	325	21	500
C32798	2	38	400	17	500
C32796	3	40	400	17	500
C32801	3	43	325	21	500
C32793	4S	38	400	17	500

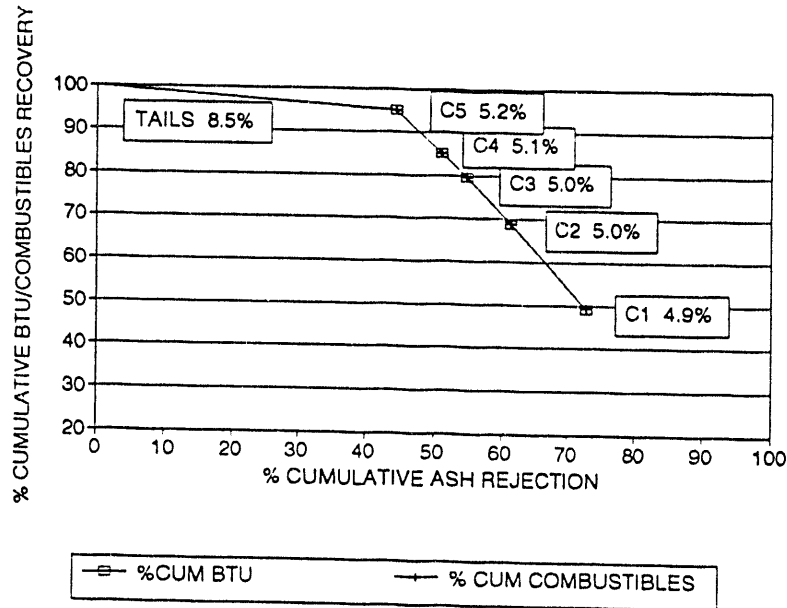
The results in Table 2 indicate better beneficiation for the -400 mesh samples than for the -100 and -200 mesh samples.

The FF/RA tests for -400 mesh size samples will be omitted for the remaining twenty eight product coals to save time and funds for the following additional work:

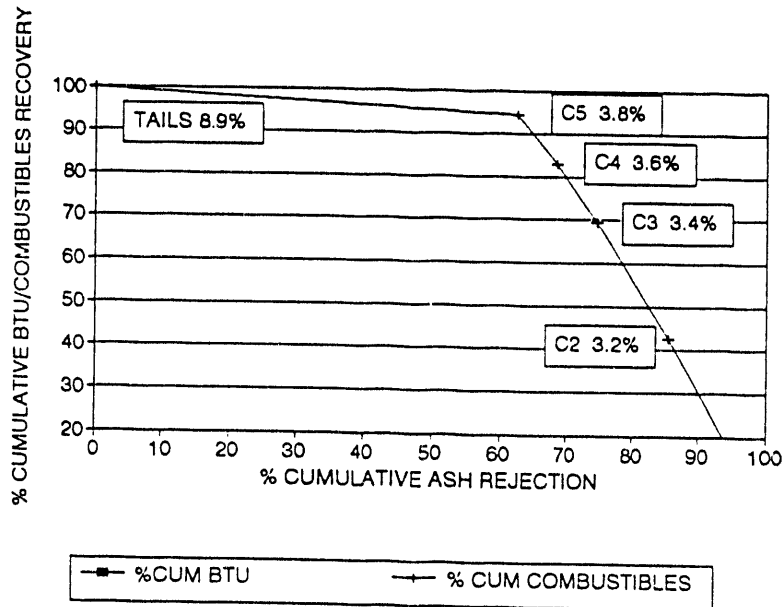
1. Complete mass balance for trace elements will be determined for two, instead one, of the flotation tests. For these two flotation tests, we will also determine the mass balance for total S, forms of S, and Cl.
2. The number of flotation concentrates were increased from four to five for all FF/RA tests to obtain better washability curves.
3. Heating value (BTU) determinations were increased from 68 to 128 to establish better empirical relationships among BTU, combustibles, and ash.

### Washability Curves

The data from the FF/RA tests are being used to construct a series of washability curves. As an example, Figure 3 indicates that for 80% of the BTU or combustibles recovered from a -100 mesh FF/RA test, about 55% of the ash was rejected. Cleaning the same coal similarly at -200 mesh size increased the ash rejection to 70% for the 80% BTU (combustibles) recovery composite (Figure 4).



**Figure 3.** Cumulative washability curve for ash rejection for sample C32776 ground to -100 mesh. Numbers on the plot are actual cumulative ash values in each concentrate (C) and tails.



**Figure 4.** Cumulative washability curve for ash rejection for sample C32776 ground to -200 mesh. Numbers on the plot are actual cumulative ash values in each concentrate (C) and tails.

**Progress on Mass Balance Study**

One sample with -100 mesh size and another with -400 mesh size were produced by grinding coal sample C32796. The ground

samples were cleaned using the established FF/RA procedures in an attempt to achieve mass balances for trace elements. The sample C32769 was selected because of its 1) high ash and high Cl contents, 2) relatively good beneficiation results achieved in the -200 vs the -100 mesh runs, and 3) the favorable distribution of weights in the FF/RA fractions. All the various solid float and sink fractions, representative samples of the water used for grinding and froth flotation, and representative feed coal for each mesh size run were prepared and submitted for analyses for trace elements, ash, total sulfur, forms of sulfur, and chlorine.

These data will indicate the degree of mass balance that is achievable under the test conditions. The data will be displayed as to indicate the beneficiation obtained in the progressive clean concentrates (C1, C2 etc.) and tails (T1, T2 etc.) fractions. The results will also be compared to the 80% composite data available from the previous tasks.

#### CONCLUSIONS AND RECOMMENDATIONS

Froth flotation/release analysis (FF/RA) tests on the 34 project samples were completed at three different particle sizes (-100, -200, -400 mesh). The FF/RA task on -400 mesh samples was modified to include six samples selected among the 34 product coals. Resources saved by omitting some of the -400 mesh runs were directed towards 1) more trace element mass balance characterization, 2) increasing the number of concentrates for each FF/RA test to obtain better washability curves, and 4) increasing the number of heating value (BTU) determinations to establish better empirical relationships among BTU, combustibles, and ash.

Flotation products from all FF/RA tests were analyzed for ash and moisture, and some of them were analyzed also for total S and BTU. Beneficiation of the samples through FF/RA were significant and increased from -100 mesh through -200 mesh to -400 mesh size. The ash, S, and BTU data are used to construct a series of washability curves that show various flotation results such as ash and S rejection or the recovery of BTU, combustibles, or weight of clean products.

Composite samples each having 80% of the total BTU or combustibles were prepared from flotation products from all FF/RA tests. All but six of the 80% BTU recovery composites were submitted for trace element analysis. The remaining six composite samples from the -400 mesh runs will be submitted soon. Trace element analyses for all composite samples will be available in 3 to 4 months. From the trace element data, beneficiation of each trace element for the -100, -200, and -400 mesh FF/RA tests relative to the feed coals will be determined.

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Chemical Coal Cleaning, Chapter in Chemistry of Physics of Coal Utilization, B. R. Cooper and L. Petrakis eds., AIP Conference Proceedings, No. 70, American Inst. of Physics, NY, pp. 357-387.

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PROJECT MANAGEMENT REPORT  
December 1, 1993 through February 28, 1994

Project Title: **WASHABILITY OF TRACE ELEMENTS IN PRODUCT COALS  
FROM ILLINOIS MINES**

DOE Grant Number : DE-FC22-92PC92521(YEAR2)  
ICCI Project Number : 93-1/6.2A-1P  
Principal Investigator: Ilham Demir, ISGS  
Co-investigators : Rodney R. Ruch, Richard D. Harvey,  
John D. Steele, and Saleem Khan, ISGS  
  
Project Manager : Ken K. Ho, ICCI

COMMENTS

The projected and estimated actual expenditures for the project through February 28, 1994 are given in the attached table and chart.

All tasks for the project are progressing as planned. The estimated actual labor cost for the first and second quarters is less than the projected amount due to shifting some of the personnel cost to later part of the project. The appointment of a half-time technical assistant, S. Khan, who was hired during the first quarter, ended January 31, 1994. The estimated actual cost for analytical services for the quarter is higher than the projected amount because we are somewhat ahead of schedule for this task.

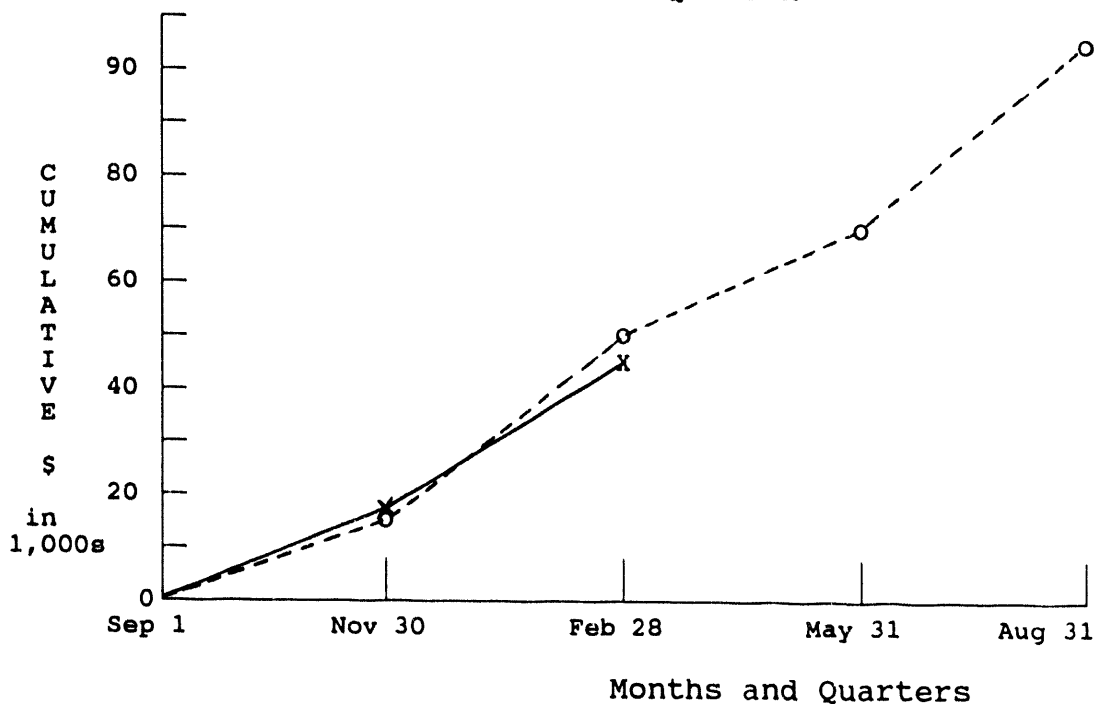
**EXPENDITURES**

## Projected and Estimated Actual Expenditures by Quarter

Quarter *	Types of Cost	Direct Labor	Fringe Benefits	Materials and Supplies	Travel	Major Equipment	Other Direct Costs	Indirect Costs	Total
Sept. 1, 1993	Projected	7,642	1,654	500	0	0	1,276	1,107	12,179
to									
Nov. 30, 1993	Estimated Actual	3,870	836	300	0	0	8,497	1,350	14,853
Sept. 1, 1993	Projected	19,789	4,234	1,100	0	0	17,730	4,285	47,138
to									
Feb. 28, 1994	Estimated Actual	10,309	2,202	533	0	0	26,862	3,991	43,897
Sept. 1, 1993	Projected	22,453	4,792	1,400	0	0	34,684	6,333	69,662
to									
May 31, 1994	Estimated Actual								
Sept. 1, 1993	Projected	25,243	5,655	1,400	0	0	51,256	8,355	91,909
to									
Aug. 31, 1994	Estimated Actual								

\*Cumulative by quarter

YEAR 1  
COSTS BY QUARTER

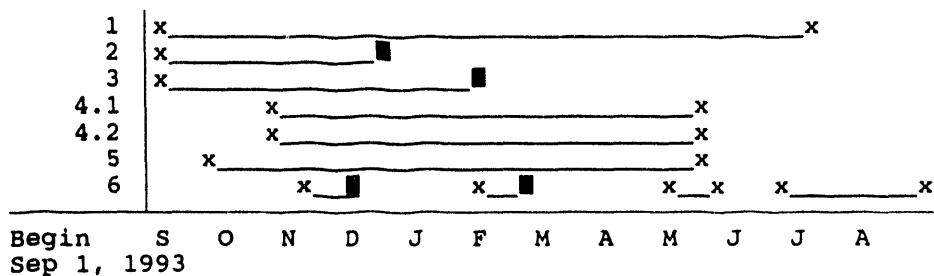


O = Projected Expenditures -----  
X = Actual Expenditures \_\_\_\_\_

Total ICCI Award \$ 91,909

**Schedule**

The schedule of project milestones for the project is as follows:



**Milestones**

- 1 Quality assurance/quality control
- 2 Sample preparation
- 3 Washability tests
- 4 Analyses of the samples
  - 4.1 Trace elements.
  - 4.2 Ash, Moisture, BTU, Total S
- 5. Mass balance calculations and washability curves
- 6. Compilation, evaluation and reports

**DATE**

**FILMED**

*6 / 29 / 94*

**END**