Energy

FOSSIL

DOE/PC/42295-T7
(DE84013743)

GRAVIMELT PROCESS DEVELOPMENT

Final Report

June 1983

Work Performed Under Contract No. AC22-81PC42295

TRW Energy Development Group
Redondo Beach, California

Technical Information Center
Office of Scientific and Technical Information
United States Department of Energy

DISCLAIMER

DISCLAIMER

Available from the National Technical Information Service, U. S. Department of Commerce, Springfield, Virginia 22161.

Price: Printed Copy A06
Microfiche A01

Codes are used for pricing all publications. The code is determined by the number of pages in the publication. Information pertaining to the pricing codes can be found in the current issues of the following publications, which are generally available in most libraries: *Energy Research Abstracts (ERA); Government Reports Announcements* and Index (*GRA* and I); *Scientific and Technical Abstract Reports (STAR);* and publication NTIS-PR-360 available from NTIS at the above address.

DOE/PC/42295-T7
(DE84013743)
Distribution Category UC-90b

Gravimelt Process Development

Contract No. DE-AC22-81PC42295

By

TRW Energy Development Group

Final Report

June 1983

Prepared for
U.S. Department of Energy
Pittsburgh Energy Technology Center
P.O. Box 10940
Pittsburgh, PA

LIST OF CONTENTS

		Page
1.0	INTRODUCTION	1
	1.1 THE GRAVIMELT PROCESS	1
	1.2 SUMMARY AND RECOMMENDATIONS	3
2.0	LABORATORY EXPERIMENTATION	7
	2.1 LABORATORY-SCALE REACTOR	7
	2.2 SEMI-CONTINUOUS OPERATION	8
	2.3 PITTSBURGH SEAM COAL PROCESSING	12
	2.4 PARTICLE SIZE EFFECTS	15
	2.5 UPPER FREEPORT COAL EVALUATION	17
	2.6 PITTSBURGH #8 REPOSITORY COAL EVALUATION	21
	2.7 OTHER REPOSITORY COALS	26
	2.8 SUMMARY OF LABORATORY RESULTS	32
3.0	BENCH-SCALE TESTING	38
	3.1 MOD 1 REACTOR	38
	3.1.1 Design of the Reactor System	38
	3.1.2 Testing of Mod 1	42
	3.1.3 Results of the Mod 1 Reactor Tests	48
	3.2 MOD 2 REACTOR	53
	3.2.1 Design	53
	3.2.2. Reactor Operation	56
APPENDIX A.	LABORATORY EXPERIMENTAL CONDITIONS AND RESULTS	A-1
APPENDIX B.	ANALYSIS OF UNPROCESSED COAL	B-1

LIST OF FIGURES

			Page
Figure	1.	Schematic Flow Diagram of the Gravimelt Process.	2
	2.	Semi-Continuous Processing of C.	10
	3.	Heat of Combustion of Semi-Continuously Processed Kentucky 11 Coal	11
	4.	Sieve Analysis of 45x200 Mesh Pittsburgh 8 Coals	14
	5.	Sulfur Removal from Upper Freeport Coal.	22
	6.	Heat of Combustion and Residual Sulfur for Processed Upper Freeport Coal.	23
	7.	Sulfur Removal from Pittsburgh No. 8 Coal.	27
	8.	Heat of Combustion and Residual Sulfur for Processed Pittsburgh No. 8 Coal.	28
	9.	Sulfur Removal from Kentucky 11 Coal	31
	10.	Heat of Combustion and Residual Sulfur for Processed Kentucky No. 11 Coal	33
	11.	Sulfur Removal from Illinois Coal.	34
	12.	Heat of Combustion and Residual Sulfur for Processed Illinois No. 6 Coal.	35
	13.	Summary of Sulfur Removal Results.	36
	14.	Mod 1 Reactor System Schematic	39
	15.	Mod 1 Reactor Weldment Assembly.	40
	16.	Mod 1 Reactor Complete Without Insulation.	41
	17.	Stirring Shafts for Mod 1 Reactor Inlet/Outlet	43
	18.	Coal Wash and Acid Treatment System.	44
	19.	SS Solid-Bowl Bird Centrifuge with Back-Drive.	45
	20.	Mod 2 Reactor, Baffle and Stirrer.	54

LIST OF FIGURES (Cont'd)

		Page
Figure 21.	Dasher Used to Assist Coal Movement.	55
22.	View of Reactor Through the Coal Removal Port.	57

LIST OF TABLES

		Page
TABLE 1.	PITTSBURGH 8 SAMPLE COMPARISONS.	13
2.	SIEVE ANALYSIS OF PITTSBURGH NO. 8 REPOSITORY COAL . . .	16
3.	PROCESSING OF PITTSBURGH 8 SIZE FRACTIONS.	18
4.	LABORATORY RESULTS WITH UPPER FREEPORT COAL.	20
5.	LABORATORY RESULTS WITH PITTSBURGH 8 REPOSITORY COAL . .	25
6.	LABORATORY RESULTS WITH KENTUCKY 11 AND ILLINOIS 6 COALS	30
7.	SUMMARY OF STEADY-FLOW REACTOR RESULTS	49
8.	COAL BALANCE	52
9.	MOD 2 REACTOR TEST SEQUENCE.	58
10.	MOD 2 REACTOR CAUSTIC AND COAL RECOVERY.	60
11.	RESULTS OF MOD 2 TESTING OF PITTSBURGH 8 COAL.	61
APPENDIX A		
A-1.	LABORATORY RESULTS	A-4
APPENDIX B		
B-1.	SUMMARY OF UNPROCESSED COAL ANALYSES	B-2
B-3.	ANALYSES OF THE REPOSITORY COALS TAKEN FROM FINAL REPORT NO. 1984-DY-061A - PREPARED BY CT&E, OHIO	B-3

1.0 INTRODUCTION

This final report contains the results of a bench-scale program to continue the development of the TRW proprietary Gravimelt Process for chemically cleaning coal. The development is under the sponsorship of the U.S. Department of Energy, Contract No. DE-AC22-81PC42295. The report introduction contains a brief description of the process, a summary of the report content and recommendations for further work.

1.1 THE GRAVIMELT PROCESS

The Gravimelt Process is a method for near-complete desulfurization and demineralization of coal by a series of three leaching operations. The process is proprietary to TRW Electronics and Defense of Redondo Beach, California. TRW conceived and tested the feasibility of both process leaching steps in 1978 and then approached the Department of Energy in 1979 for the funding of a laboratory effort. A contract was awarded to TRW (DE-AC-80PC30141) on April 1, 1980 for the continued laboratory testing of the Gravimelt Process. The success of that project resulted in award of the present effort.

TRW has, by agreement with DOE, retained its background patent rights and is in the process of obtaining patent protection on the Gravimelt Process.

TRW defines the Gravimelt Process as "treatment of crushed coal with molten alkali metal hydroxides to remove organic and pyritic sulfur, removal of the coal from molten media, washing with water to remove residual media from the coal and washing with dilute mineral acid to remove residual mineral matter to produce a unique coal product nearly free of sulfur and mineral matter". A simplified flow scheme is shown in Figure 1.

The complete process requires regeneration of both the alkali metal hydroxides and the neutralization of the dilute mineral acid wash streams to provide both sulfur-containing and mineral process products. The results of

TRW Gravimelt Process

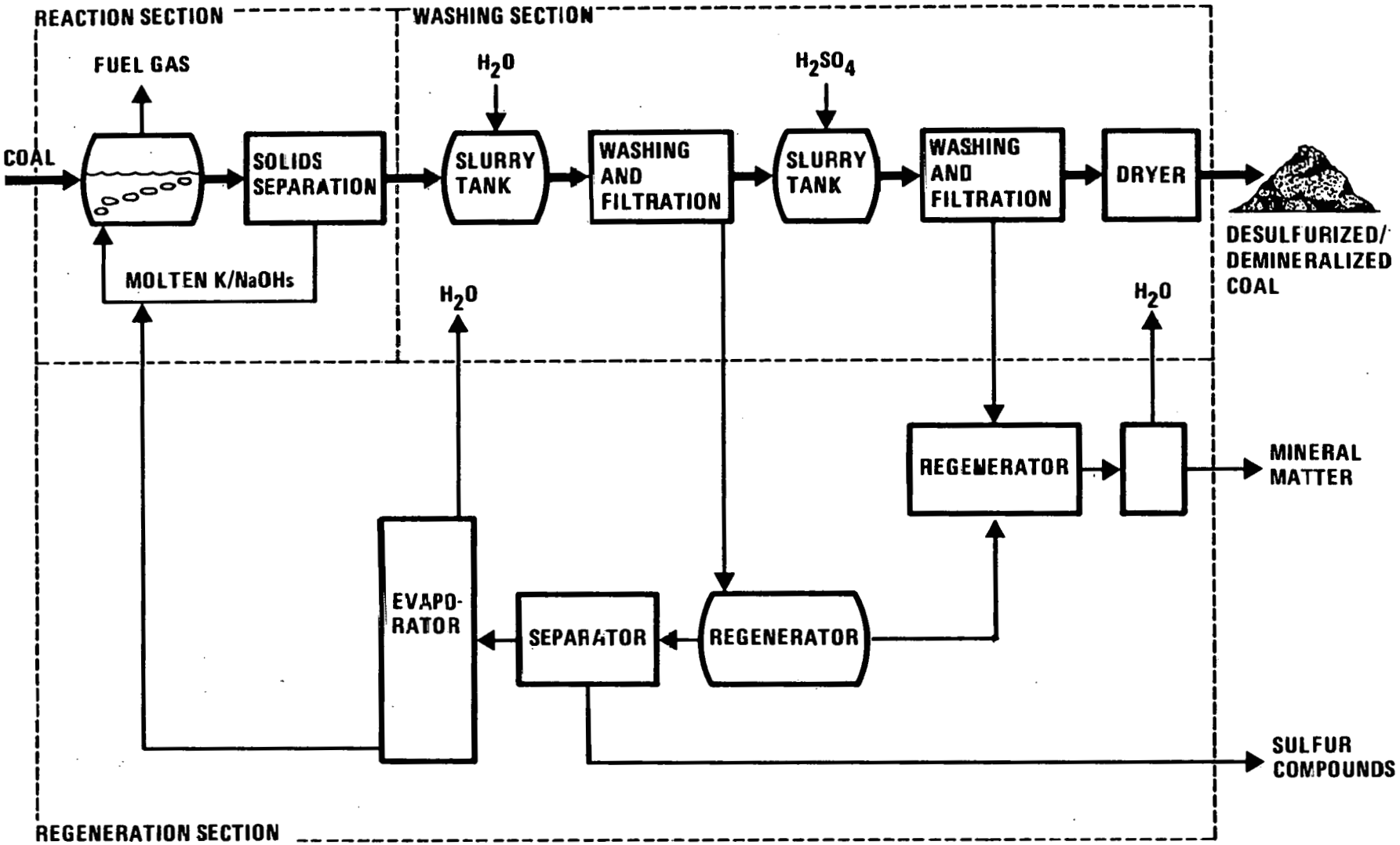


Figure 1. Schematic Flow Diagram of the Gravimelt Process

the DOE laboratory project have shown that a combination of normal mine cleaning followed by Gravimelt treatment can give a removal of more than 90% of the sulfur and about 99% of the mineral matter from coal. Sulfur reduction by this process produces product coal which meets current federal pollution control standards as well as state implementation standards for sulfur oxides control. The sulfur and ash content of the resulting coal product is similar to coal synfuels products and thus may be another method to produce boiler fuel of similar quality to imported or domestic residua.

The next step in development of the Gravimelt Process technology was to move to bench-scale testing. This involved working with larger quantities of coal in continuous and semi-continuous equipment. This type of testing provided improved material balance data, reactor configuration and equipment operation information and other data necessary to the further development of the process at test plant scale. In addition, it becomes possible to make increasingly accurate predictions as to the ultimate cost of the process as well as its applicability.

Recent preliminary conceptual design and economics studies performed by TRW indicate that the near-complete desulfurization and demineralization of coal can be accomplished at an upgrading cost of roughly $30 per ton of coal product ($1.20/$10^6$ Btu) with a 90% energy efficiency. If this forecast is accurate, the Gravimelt coal product could be quite advantageous over other methods of sulfur oxides control or methods for providing a fuel for switching of oil-fired boiler installations to coal.

1.2 SUMMARY AND RECOMMENDATIONS

This project consisted of two major efforts, a laboratory study aimed at identifying parameters which would influence the operation of a bench unit for desulfurization and demineralization of coal and the design, construction and operation of two types of continuous plug-flow type bench-scale fused caustic leachers.

One hundred and twenty-five laboratory size fused caustic extractions were performed and the analytical results were assessed defining the effects of retention time, temperature, coal to caustic ratio, ratio of sodium to potassium hydroxides, particle size distribution and coal type on sulfur and ash removal and the heat content of the product coal. Coals from four seams were tested as a part of this project. Illinois No. 6 and Kentucky No. 11, from the DOE coal repository, represented the eastern interior coal basin. Pittsburgh No. 8, also from the repository, and Upper Freeport, supplied by the DOE, represented the northern Appalachian region. The Upper Freeport coal had both mine-cleaned and run-of-mine (ROM) samples tested while the three repository coals were available only in mine-cleaned samples with about 10 percent ash.

A goal of the project was to identify Gravimelt reaction conditions that would result in a product coal meeting the EPA New Source Performance Standards (NSPS) for SO_2 emissions. These are four categories of coals based on their sulfur and heat content as mined. Category 1 applies to coal that is already quite clean as mined and would emit no more than 2 lb of SO_2 per million Btu without cleaning. The standards require than 70% of the potential SO_2 emission be removed. Category 2 is higher in sulfur and if not cleaned would emit between 2 and 6 pounds of SO_2 per million Btu. Coal in this range must be cleaned to yield a maximum of 0.6 pound per million Btu. This means 70% removal from the 2 pound per million Btu and increasing to 90% removal from the 6 pound per million Btu coal. For coal between 6 and 12 pounds of SO_2 per million Btu, the requirements of Category 3 are 90% removal. Finally, the highest sulfur coals are in Category 4 where the as-mined emission of SO_2 would exceed 12 pounds per million Btu. This must be cleaned to a level of 1.2 pounds of SO_2 per million Btu. These are summarized as follows:

Category	SO_2 Emission Potential for ROM Coal, ($lb/10^6$ Btu)	SO_2 Reduction Required
1	0-2	70%
2	2-6	to 0.6 (70-90%)
3	6-12	90%
4	> 12	to 1.2 $lb/10^6$ Btu

The four coals tested during this study have the following ROM analyses and SO_2 reduction requirements.

Seam	ROM Analysis (MF)* Ash	S	Btu/lb	SO_2 lb/10^6 Btu	EPA Category	Maximum SO_2 lbs/10^6 Btu
Upper Freeport	37.8	2.5	9039	5.5	2	0.6
Pittsburgh No. 8	30.2	3.7	8871	8.3	3	0.8
Kentucky No. 11	29.0	4.6	9092	10.1	3	1.0
Illinois No. 6	24.2	5.0	8272	12.1	4	1.2

Reaction conditions were found for each of these coals that produced a SO_2 compliant product with a concurrent reduction of ash by more than 99%.

Two fused caustic/coal leaching reactors were built and operated. The first, designated Mod 1, is a continuous flow, gravity displacement, stirred pipe reactor sized at up to 5 pounds per hour of coal feed. The unit was operated utilizing mine cleaned Illinois #6, Pittsburgh #8 and Upper Freeport seam coals as well as run of mine Upper Freeport seam coal. While the operation of this unit was found to be generally unsatisfactory due to foaming (which influenced the feed and discharge rates), 95% ash removal and better than 90% sulfur removal was obtained for Illinois #6 seam coal and 95% ash removal and near 90% sulfur removal for Pittsburgh #8 seam were obtained. It is believed that a larger diameter version of this reactor would not be impacted by foaming. Material balance data was obtained for the Pittsburgh #8 seam coal processing as well as for mine cleaned Upper Freeport coal indicating near 100% coal recovery.

The second reactor, designated Mod 2, is a continuous flow, open surface, spiral path reactor with sequential prewetting and reaction sections sized at 1 to 2 pounds of coal per hour. This reactor was operated successfully for a

---

*Each mine that had supplied coal for the repository was contacted and also supplied 6 to 36 months of ROM coal analyses. These were averaged by Dave Boron, (the TPO for this project) and provided to TRW for use in calculating the SO_2 removals throughout the report.

series of nine tests totalling 98 hours during one week of 24 hour per day operation on Pittsburgh #8 seam coal. The results showed that 80-90% of the sulfur and better than 90% of the ash was removed at steady state during the last two runs in the sequence where 50/50 ratio of potassium to sodium hydroxides was utilized at 370°.

This present bench scale project has demonstrated modes for the continuous operation of fused caustic leaching of coal at coal throughputs of 1 to 5 pounds per hour. The remaining process unit operations of leach solutions regeneration and coal washing and filtration should be tested at bench scale together with fused caustic leaching of coal to demonstrate the complete Gravimelt Process. The testing of these unit operations in a sequential mode will give data for assessment of major process questions such as:

1) Will regenerated caustic desulfurize and demineralize coal at the same rate and to the same degree as the fresh chemicals?

2) To what level do coal impurities build up in the various recycle streams and what effect do these impurities have on the process?

3) What is the purity of product reject mineral matter?

4) Can 99%+ of the sodium and potassium be recovered and recycled?

5) Can coal-derived sulfur be recovered and converted to sulfuric acid for internal process use?

6) How does microwave heating compare with thermal heating of the leach system?

7) Assuming that the process unit operations operate well in a sequential mode, what is the design basis and cost for a larger proof of concept test plant?

It is recommended that a bench scale project be initiated for the sequential testing of all Gravimelt Process unit operations at a roughly 20 pound per hour of coal design size. This size is selected to provide sufficient product for utilization of the smallest size commercial filtration and centrifuge equipment. The project should be initiated with a series of laboratory scale regeneration testing and scale up tasks followed by engineering design, construction and operation of the suggested 20 pound per hour size system.

2.0 LABORATORY EXPERIMENTATION

The purposes of the laboratory experimentation were to provide support to the design and operation of the bench scale reaction units and to conduct other studies needed to identify and investigate process parameters important to the development of the Gravimelt Process. Timely laboratory testing was vital to the successful scale up and transition to continuous operation of the reactor system. Laboratory-scale tests provided the quickest and least costly way to obtain large numbers of parametric data after the scale up effects were understood. Each of the general laboratory tests is listed in Appendix A and identified according to the assigned test designation. In the discussion of results, which follow, appropriate data have been abstracted from the Appendix and organized to demonstrate the specific parametric effect or conclusion. Special tests or analyses are occasionally presented in the discussion and not listed in the Appendix because they do not fit well into a standard format.

In the following sections, the laboratory-scale reactor will be briefly reviewed and results of parametric tests are presented.

2.1 LABORATORY-SCALE REACTOR

Experimentation during this effort used the lab scale apparatus developed on the previous program as described in detail in its final report*. Most of the present experiments were conducted in the 3-inch diameter by 12-inch long, 316L stainless steel reactor. Typically, 800 grams of caustic were melted and 80 grams of coal were added to the stirred reactor under a nitrogen purge. At the conclusion of the tests, the coal was either skimmed from the surface, or more frequently, the reactor was cooled and the surface of solid caustic containing the floating coal was removed. In either case, the coal/caustic mixture was dissolved in 0.8 to 1.0 liter of water, filtered and rewashed with water or aqueous acid using a similar volume of liquid, each time.

---

*Laboratory Study for Removal of Organic Sulfur from Coal," U.S. Department of Energy, Contract No. DE-AC22-80PC30141, dated July 1, 1981.

The water washed coal products were given one additional water wash, after the initial coal/aqueous caustic separation. The acid washed coal products were given one water wash, then a wash with 10% sulfuric acid and finally two additional water washes prior to drying. Drying was performed in an air oven at 70°C, usually overnight. The dried coal product was riffled into samples for analysis at TRW Laboratories and at Warner Laboratories, Cresson, Pennsylvania. In a few tests, the wet filter cake, after water washing, was split and about one-half was acid washed with a proportionately reduced quantity of liquid. In splitting wet cakes, a top-to-bottom slice of the cake from a filter paper was taken to minimize the effect of layering. Changes from the typical processing method are noted in the Appendix.

2.2 SEMI-CONTINUOUS OPERATION

Experimental investigations were initiated to support the development of the continuous bench-scale unit for coal leaching. A series of studies were performed in the laboratory reactor (3" diameter, 12" deep) and the TRW-owned large Gravimelt Batch Reactor* (18" diameter, 24" deep). The specific purpose of the experimentation was to investigate the processing of a semi-continuous flow of coal in a single stage reactor system. These data were compared with previous batch laboratory scale data to provide an indication of the importance of maintaining plug flow in the continuous reactor system.

Experimentation in the 3" diameter reactor consisted of two runs using NaOH/KOH (80/20) and Kentucky No. 11 coal (45 x 200 mesh) at 350°C. Coal (40g) was added to 400 grams of NaOH/KOH melt at 350°C. At the conclusion of 20 minutes, about 3 grams of reacted coal were removed from the reactor (grab sample) and 3 grams of fresh coal were added. This operation continued every 20 minutes (approximately) for 180 minutes, at which time the experiment was concluded and the final product was removed from the reactor. Duplicate runs

---

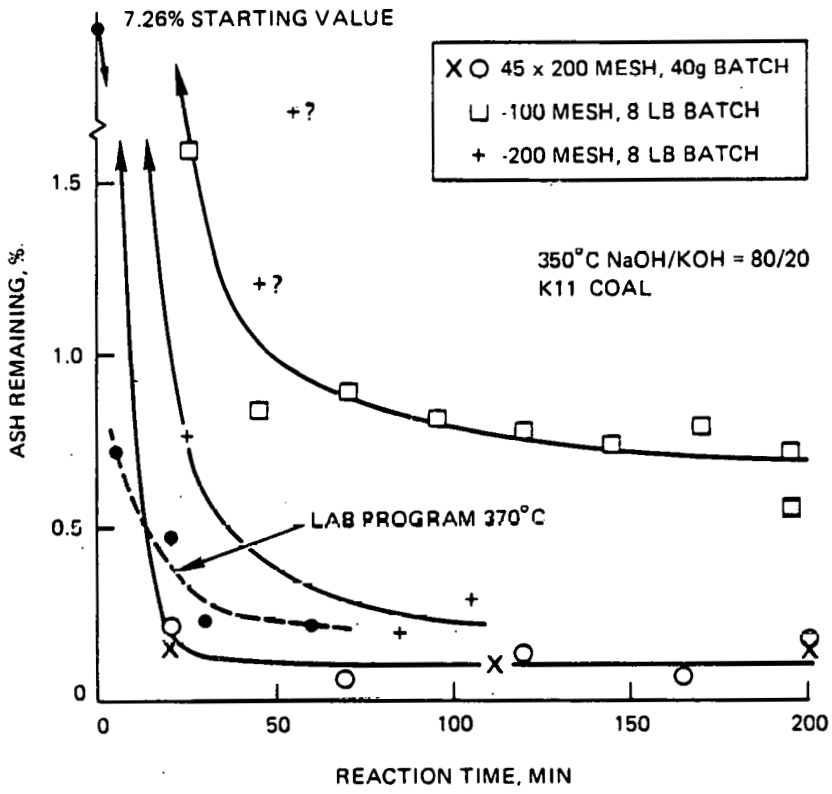
*This reactor was fabricated by TRW for Gravimelt evaluation of 8 to 10 pound batches of coal on Independent Research and Development Programs and was made available for use in these preliminary evaluation studies.

were conducted and representative samples were sent to Warner Laboratories for analyses. It is seen in Figure 2, that 20 minutes at 350°C is sufficient time to remove much of the sulfur (to a level of about 1%) and nearly all the ash (to a level of about 0.2%) from the 45 x 200 mesh Kentucky coal. Additional time, beyond 1 to 2 hours, appears to give steady state levels of both sulfur (0.7%) and ash (0.1%).

Laboratory batch reactor data with this coal, were reported in the previous final report. It was tested at 370°C rather than 350°C. The results of batches between 5 minute and 60 minute residence time are shown in Figure 2 for comparison. The sulfur removals are a little better, as expected from the higher temperatures, but the ash levels are slightly poorer (0.2% for the one hour batch versus 0.1% for the semicontinuous test).

Two experiments also were conducted in the large TRW-owned 18" diameter Gravimelt reactor. The experiments were conducted with a semicontinuous removal/addition of coal using particle coal sizes of 200 mesh x 0 and 100 mesh x 0. Both coal sizes,starting with 8 pound batches, were processed at 350°C for approximately three hours wherein every 20 minutes about 1/2 pound was removed and one pound was added. It is seen in Figure 2 that the large reactor product coal sulfur levels were similar to lab reactor results but the ash levels were higher even after 1 to 2 hours when an apparent steady state was reached. The effect is much less for the smaller particle size coal samples (0.7% ash at 100 mesh, 0.2% ash at 200 mesh versus 0.1% ash lab).

Heat of combustion data for these four tests (calculated on a moisture and ash free basis) tended to show small decreases with time. There were also small decreases with time in the residual sulfur, as discussed above. It was felt that the change in heat of combustion was probably more related to the severity of the reaction, as measured by the remaining sulfur, than it was related to time. All fourteen analyzed data points are shown in Figure 3 as a plot of the moisture and ash free (MAF) heat of combustion versus the remaining total sulfur in the process coal. Also shown is the starting value for the organic sulfur and heat content of the unprocessed coal.



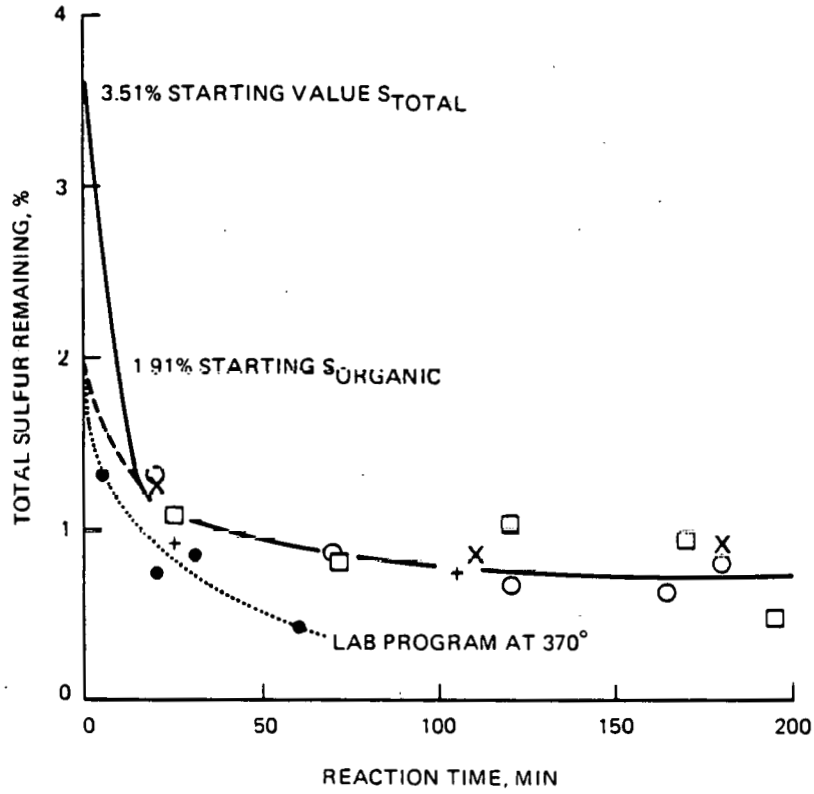


Figure 2. Semi-Continuous Processing of Coal

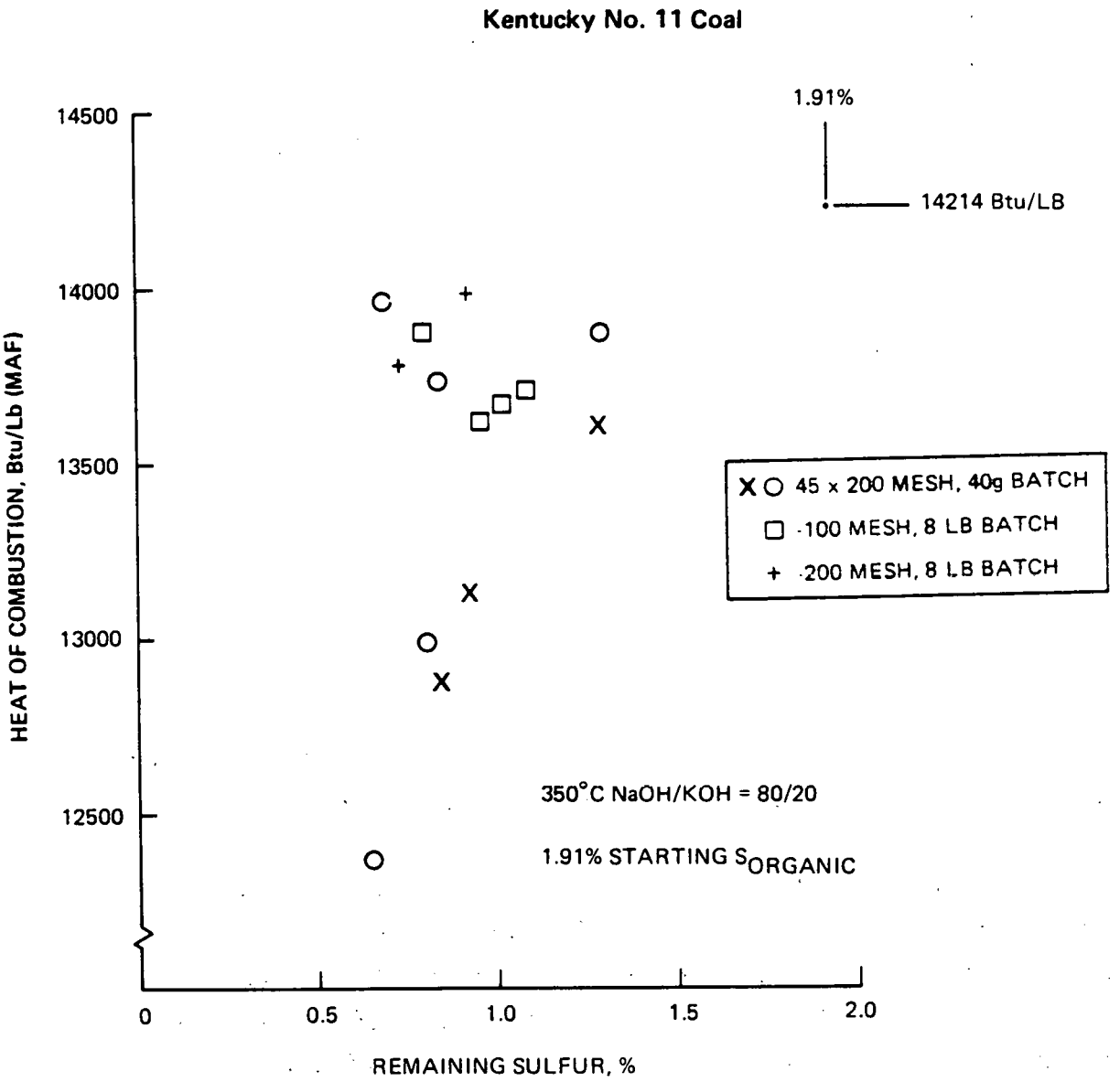


Figure 3. Heat of Combustion of Semi-Continuously Processed Kentucky 11 Coal

The data are scattered, but there is a weak trend toward lower heat of combustion at lower sulfur levels. An hypothesis, which is further examined throughout this report, is summarized as follows. When coal is contacted with molten caustic in the Gravimelt Process, the inorganic sulfur (pyrite and sulfate) is rapidly removed, but the organic sulfur is more slowly removed by reactions involving the organic matrix. Side reactions which reduce the heat of combustion of the organic matrix, may proceed concurrently with the desired reactions that remove sulfur from the organix matrix. This parallel reaction behavior is believed to produce the often observed correlation between heat of combustion and residual total (organic) sulfur. However, occasionally, an experimental run gives coal which analyzes very low in sulfur and has a high heat content.

2.3 PITTSBURGH SEAM COAL PROCESSING

Coals from the same seam cover large geographical areas and samples may vary in their response to chemical cleaning. In an effort to examine this parameter, four Pittsburgh #8 coals, available at TRW, were processed in a uniform manner. All coals were prepared as dried, 45 mesh by 200 mesh samples, then processed for one hour with a 50/50 NaOH/KOH caustic melt in the laboratory reactor. Table 1 shows the results of duplicate tests at 370°C and single or duplicate tests at 340°C. The coals are listed in the table in the order of their weight average particle sizes (Figure 4) which range from 150 microns for the laboratory coal to 200 microns for bagged coal.

The table shows that prior to processing, both ash and sulfur tend to be higher and the moisture-ash free (MAF) heat of combustion tends to be lower in the fine coals than they are in the coarser bag and barrel coals. As a result of 370°C caustic processing, the heat of combustion decreased by approximately 1000 Btu/lb (MAF basis) for each of the five processed coal samples.* The final ash and sulfur content of the processed samples showed no

---

*From the users point of view, the coal is improved by chemical cleaning. There is about 10% less ash and the chemically cleaned product has up to 1300 Btu/lb higher heat of combustion than the unprocessed, mine cleaned coal.

TABLE 1. PITTSBURGH 8 SAMPLE COMPARISONS

(Conditions: 45 x 200 mesh*, 50/50 caustic, 1 hour, acid washed)

	(MF) Ash,%	(MF) S_T,%	(MF) Btu/lb	(MAF) Btu/lb	% Btu Retained	lb/million Btu (MF) Ash	lb/million Btu (MF) SO_2	% Removal Ash	% Removal S
153μ, Untreated Lab-A (Control)	12.37	4.58	12826	14637	-	9.64	7.14	-	-
370°C GP206	.39	.34	13727	13781	94.1	.28	.50	97.1	93.1
370 GP207	.21	.33	13724	13752	94.0	.15	.48	98.4	93.3
340 **	.19	.45	14002	14029	95.8	.14	.64	98.5	91.0
340 **	.21	.31	13985	14014	95.7	.15	.44	98.4	93.8
158μ, Repository-C (Control)	9.19	4.45	13332	14681	-	6.89	6.68	-	-
370°C GP200	.05	.52	13760	13832	94.2	.38	.76	94.5	88.7
370 GP201	.15	.48	13868	13888	94.6	.11	.69	98.4	89.6
340 GP208	.34	1.13	14405	14454	98.5	.24	1.57	96.5	76.5
177μ, Repository-B (Control)	11.24	4.00	12981	14625	-	8.66	6.16	-	-
370°C GP198	.56	.80	13812	13889	95.0	.41	1.16	95.3	81.2
370 GP199	.06	.54	13694	13702	93.7	.04	.79	99.5	87.2
340 GP209	.34	1.05	14279	14327	98.0	.24	1.47	97.2	76.1
340 GP210	.47	1.27	14333	14401	98.5	.33	1.77	96.2	71.2
185μ, Barrel -D (Control)	8.78	3.67	13564	14870	-	6.47	5.41	-	-
370°C GP202	.36	.50	13941	13991	94.1	.26	.72	96.0	86.7
370 GP203	.29	.50	14557	14599	98.2	.20	.69	96.9	87.3
340 GP212	.73	1.08	14569	14677	98.7	.51	1.48	92.1	72.6
204μ, Bag -E (Control)	7.03	2.41	14007	15066	-	5.02	3.44	-	-
370°C GP204	.54	.42	13788	13863	92.0	.39	.61	92.2	82.4
370 GP225***	.48	.45	14293	14362	95.3	.34	.63	93.2	81.7
340 GP211	1.69	1.18	14734	14988	99.5	1.15	1.60	77.1	53.5

Notes

*The laboratory sample is 100% -45 mesh, 30% -200 mesh, sieved to 45 x 200. Repository coal nominally 14 x 0 (actually about 6 mesh x 0) was prepared: B sieved to -45 x +200 and C ground to -45 mesh, then sieved to +200 mesh. The Barrel (nominally 14 mesh x 0) was sieved to 45 x 200. The Bagged samples, purchased for Company Research nominally 50 mesh by 0 (actually about 14 mesh top size), was sieved to 45 x 200.

**These two 340°C tests were performed under the previous laboratory study Contract DE-AC22-80-PC30141 Final Report, Page 29, using a different natch of sieved coal with slightly different starting analysis. Results are not completely comparable.

***GP225 repeated test GP205 because the results of 205 were like a partially processed coal.

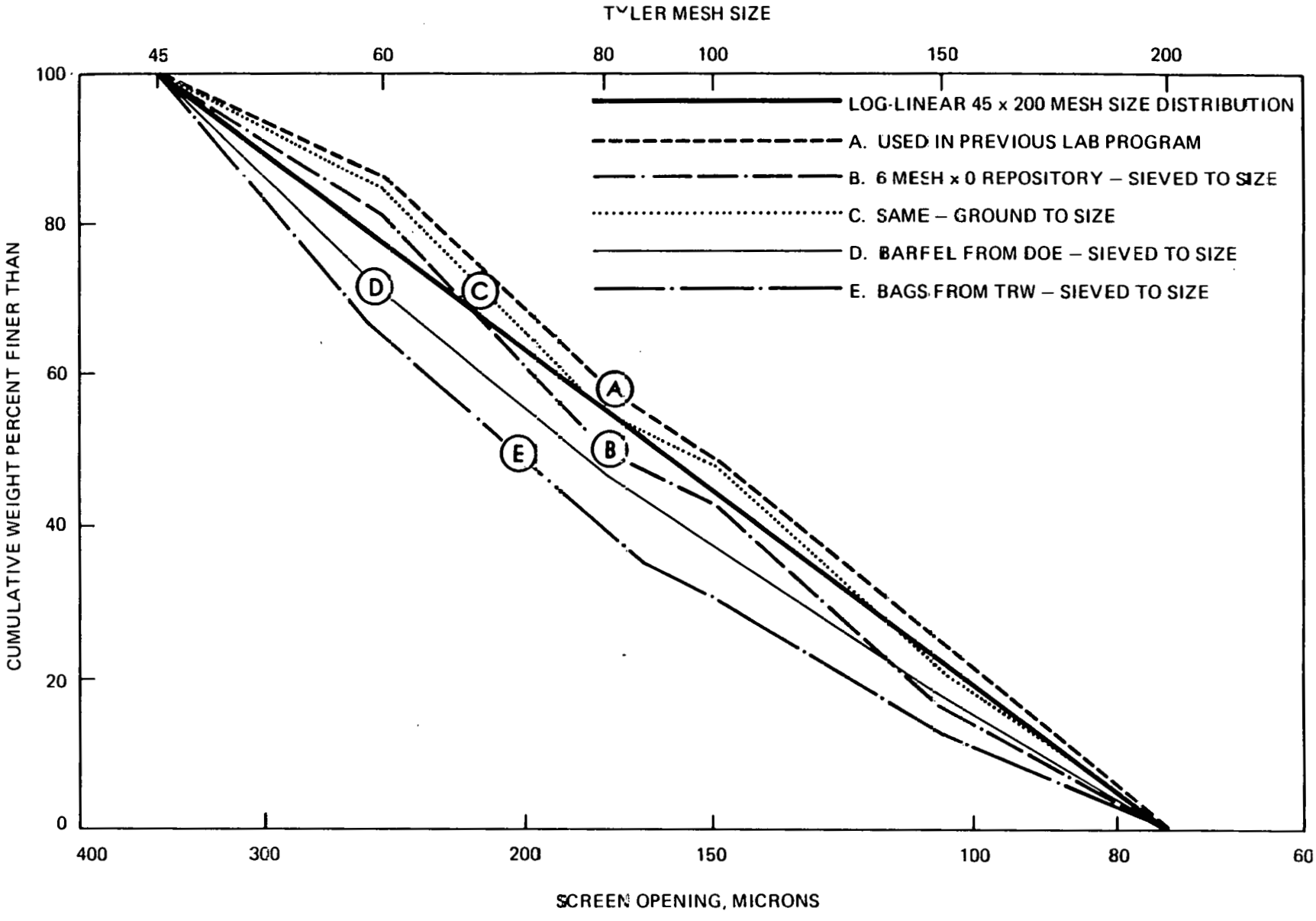


Figure 4. Sieve Analysis of 45x200 Mesh Pittsburgh 8 Coals

statistically meaningful trend with particle size. At 370°C, the ash was reduced to a mean value of .26 $\pm$ .13 pounds/million Btu and the residual sulfur to .70 $\pm$ .19 pounds SO_2/million Btu. Because the starting ash and sulfur of the mine-cleaned coal varied, the removal efficiency at 370° (starting with the mine-cleaned not run-of-mine values) ranged from about 92% to 99%+ for ash and 81% to 93% for sulfur.

Processing the coal samples at 340° results in a heat of combustion loss about one-fifth as large as 370° processing, but leaves about twice as much sulfur. Residual ash after 340° treatment, varies from about the same as at 370° to several times as much. In general, the lower temperature processing did not meet the project sulfur removal goals (1.2 pound SO_2/million Btu) whereas the 370° results all are below the target and also exceed 90% removal if the "as-mined" (ROM) starting levels of sulfur (typically 8 to 10 pounds of SO_2/million Btu are considered. However, it is obvious that processing the Pittsburgh #8 coal at 340° can give a product with extremely low ash (0.3% w/w) with only 1% to 2% loss of MAF heat content. At these mild conditions, probably all of the pyrite, but little of the organic sulfur, has been removed.

2.4 PARTICLE SIZE EFFECTS

The particle size of coal, processed by molten caustic to remove ash and sulfur, influences the rate or extent of removal. A limited, but systematic, study of particle size effects was undertaken for the Pittsburgh #8 coal from the repository. A sieve analysis, for the nominal 14 mesh coal, was provided by CT&E, from which four approximately equal quantities of coal were selected as shown in Table 2., After drying and sieving the coal at CTS, the actual yield of each size is also shown. The coarse, +14 mesh, cut was sieved as shown, to further define its particle size. Each size fraction was riffled into 80 gram samples for processing. Sampling variability was expected to be greater for the coarse samples; therefore, the 6 x 14 mesh and 14 x 24 mesh tests were run in duplicate. Mild processing conditions of 1-hour reaction time at 370° with an 80/20 NaOH to KOH ratio were selected in order to obtain a definitive spread of removal results. The effect of more aggressive process

TABLE 2. SIEVE ANALYSIS OF PITTSBURGH NO 8 REPOSITORY COAL

Size, mesh	CT&E (As Reported) wt,%	CTS (dried) wt,%
+14	22.9	15.3
14 x 28	26.6	27.1
28 x 100	28.2	30.0
100 x 0	22.3	27.6
	100.0	100.0

SIEVE SIZING OF THE +14 MESH FRACTION

Size, mesh	CTS wt,%
+6	0.0
6 x 10	31.6
10 x 12	31.8
12 x 14	36.1
14 x 0	0.5
	100.0

conditions (time, temperature, and caustic ratio) on the rate or extent of ash and sulfur removal was not determined.

Analytical results are presented in Table 3. Data for the four size fractions of unprocessed and processed coals are given in the top part of the table and are combined on a weighted average basis to compare with full-size range unseparated coal in the lower part of the table. The good agreement between the weighted average results and the unseparated coal results gives reasonable confidence in the individual size fraction results.

The table shows that the best removal of both sulfur and ash occurs during processing of the 24 x 100 mesh fraction. Ash and SO_2 were calculated on a pounds per million Btu basis, and the corresponding percent removals from the starting value are as follows:

Mesh Size	% Removal (lb/10^6 Btu basis)	
	Ash	S
6 x 14	70.6	54.5
14 x 24	78.1	57.0
24 x 100	90.9	68.2
100 x 0	90.7	65.8
6 x 0 - unweighted average	84.8	62.6
6 x 0 - unseparated	82.9	64.1

Ash removal is more sensitive to size than sulfur removal under the conditions and ranged from 70% in the coarse fraction to 90% in the two fine fractions. In the coarsest fraction the sulfur removal (55%) is only slightly more than the average inorganic sulfur for the Pittsburgh #8 coal (Appendix B shows 52%). In the finest fractions, the sulfur corresponds to removal of the inorganic sulfur plus about one-third of the organic.

2.5 UPPER FREEPORT COAL EVALUATION

Two samples of Upper Freeport coal were provided for program use by the Department of Energy. One barrel contained run of mine (ROM) coal analyzing about 40% ash. A typical analysis of the ROM coal was also provided by the

TABLE 3. PROCESSING OF PITTSBURGH 8 SIZE FRACTIONS

Test No.	Size, Mesh	Conditions			TRW	Warner		MF	MAF
		Temp	NaOH/KOH	Time	Ash,%	Ash,%	S_T,%	Btu/lb	Btu/lb
Control	6 x 14	unprocessed			-	10.40	4.11	13273	14814
GP171*	"	370°	80/20	1	3.12	3.21	2.25	13836	14234
GP185	"	370°	80/20	1	2.20	3.06	1.58	13425	13849
			mean value		2.66	3.14	1.92	13630	14072
Control	14 x 24	unprocessed			-	9.89	4.52	13207	14673
GP172	"	370°	80/20	1	2.09	2.45	2.22	14172	14534
GP186	"	370°	80/20	1	2.10	2.19	1.95	14083	14398
			mean value		2.10	2.34	2.08	14128	14467
Control	24 x 100	unprocessed			-	10.63	4.40	13124	14685
GP175	"	370°	80/20	1	1.19	.98	1.42	13335	13467
Control	100 x 0	unprocessed			-	13.44	4.79	12633	14595
GP177	"	370°	80/20	1	1.46	1.33	1.74	13437	13615
Calculated Results for Full-Size Range Using the Weighted Average Values Found Above									
Control	6x0	unprocessed			-	11.20	4.50	13034	14678
Processed	6x0	370°	80/20	1	1.74	1.78	1.76	13623	13872
Measured Results for Full Size Range Prior to Sieving into the Size Fractions									
Control	6x0	unprocessed			-	10.34	4.23	13265	14795
Control	6x0	unprocessed			-	10.34	4.20	13295	14832
			mean value			10.34	4.22	13280	14812
GP167	6x0	370°	80/20	1	1.67	1.86	1.59	13944	14208

* Analyses are after double acid washing.

DOE. The second barrel contained mine cleaned coal with about 10% ash. Two samples of each barrel were sent to Warner Laboratories for analysis. These results together with the typical DOE analysis provided by the DOE are as follows:

Upper Freeport	Mine Cleaned		ROM		
	#1	#2	#1	#2	Typical
Ash, % (MF)	9.90	9.53	37.48	38.03	40.00
S_t, % (MF)	1.25	1.23	2.48	2.49	1.09
Btu/lb (MF)	13923	14057	9169	8908	8065
Btu/lb (MAF)	15453	15537	14667	14374	13467
S_p, % (MF)	.93?	.50	1.98	1.93	-
S_s, % (MF)	.02	.01	.03	.02	-
S_o, % (MAF)	.30?	.72	.47	.55	-
SO_2, lb/10^6 Btu	1.80	1.75	5.41	5.59	2.70

Thirty-two laboratory-scale tests were conducted on the Upper Freeport coal. Twenty-seven tests used mine-cleaned coal and five tests used ROM coal. The complete results of each test are shown in Appendix A, but in this section, test results showing the sulfur data and heat of combustion data have been organized by test condition for greater clarity in observing the effect of key parameters.

In Table 4 all the 32 tests are listed, first by reaction temperature (370°C and 390°C), then by caustic ratio (which also identifies the use of commercial grade or laboratory grade) and finally by reaction time. Starting mesh size is shown and under the "Remarks" are the five tests using ROM coal, two tests in which hydrochloric acid rather than sulfuric acid was used, one test stirred slowly and one test stirred intermittently. The analyzed residual total sulfur is listed and the measured heat of combustion is given on a moisture and ash free basis. Table 4 also gives the potential emission of ash and sulfur dioxide calculated on a pounds per million Btu basis and the corresponding percent removal from the ROM value.

TABLE 4. LABORATORY RESULTS WITH UPPER FREEPORT COAL

	Temp °C	Caustic NaOH/KOH	Type	Time, hr	Mesh Size	MF S,%	MAF Btu/lb	lb/million Btu (MF) Ash	lb/million Btu (MF) SO_2	% Removal* Ash	% Removal* SO_2	Remarks
GP136	370	80/20	L	1/3	100x0	1.11	15450	4.28	1.53	89.7	72.1	
GP135	370	80/20	L	1/3	100x0	1.08	15444	2.86	1.46	93.2	73.4	
GP188	370	80/20	L	1	6x0	.357	15150	.36	.36	99.1	93.5	
GP174	370	80/20	C	2	6x0	1.01	15444	1.72	1.34	95.9	75.5	ROM
GP170	370	80/20	C	2	6x0	1.08	15497	1.67	1.38	96.0	75.0	
GP217	370	80/20	C	2	6x0	.90	15424	1.32	1.19	96.8	78.3	
GP178	370	80/20	C	4	6x0	1.06	15405	1.84	1.41	95.6	74.2	ROM
GP169	370	80/20	C	4	6x0	.90	15272	.96	1.20	97.7	78.3	
BSS15	370	50/50	L	1/3	6x0	.98	15432	1.80	1.31	95.7	76.3	ROM
BSS16	370	50/50	C	1/3	6x0	.9	15463	1.85	1.21	95.6	78.0	ROM
GP141	370	50/50	L	1	100x0	.97	15208	1.90	1.31	95.4	76.1	
GP142	370	50/50	L	1	100x0	.88	14729	1.66	1.22	96.0	77.7	
GP235	370	50/50	C	1	6x0	.75	15364	1.49	1.00	96.4	81.8	ROM
GP142	370	50/50	L	1	100x0	.79	15319	1.76	1.06	95.8	80.7	HCl
GP141	370	50/50	L	1	100x0	.68	15443	1.46	.90	96.5	83.6	HCl
GP152	370	50/50	L	2	100x0	.74	15237	.58	.98	98.6	82.2	
GP152A	370	50/50	L	2	100x0	.64	15164	.48	.85	98.9	84.5	
GP154	370	50/50	L	2	6x0	.62	15081	.64	.83	98.5	84.9	
GP230	370	50/50	C	4	6x0	.53	15034	.49	.71	98.8	87.1	Stir 1/2 speed
GP176	370	50/50	C	4	6x0	.47	14912	.36	.63	99.1	88.5	
GP231	370	50/50	C	4	6x0	.46	14815	.39	.62	99.1	89.6	Stir 1 min off 15 mins
GP191	370	50/50	C	6	6x0	.28	14808	.50	.38	98.8	93.1	
GP218	390	80/20	C	2	6x0	.77	15324	1.13	1.02	97.3	81.4	
GP236	390	80/20	C	2	12x0	.63	15193	.52	.83	98.7	84.8	
GP237	390	80/20	C	2	14x200	.68	15245	.81	.90	98.1	83.6	
GP151	390	50/50	L	1	100x0	.54	15058	.47	.72	98.9	86.9	
GP151A	390	50/50	L	1	100x0	.33	14511	.17	.46	99.6	91.7	
GP155	390	50/50	L	1	6x0	.53	15034	.67	.71	98.4	87.1	
GP192	390	50/50	C	2	6x0	.39	15009	.58	.52	98.6	90.5	
GP158	390	50/50	L	2	6x0	.28	14832	.24	.38	99.4	93.1	
GP193	390	50/50	C	4	6x0	.24	14547	.21	.33	99.5	94.0	
GP153	330	0/100	L	2	100x0	.38	13939	.34	.55	99.2	90.0	

*Removal calculated from ROM coal with ash 41.77, SO_2 5.50 lb/10^6 Btu. Physical cleaning reduced the ash by 83.4% and SO_2 by 67.8%

The sulfur data from Table 4 is plotted in Figure 5. The residual sulfur decreases with increasing reaction time and temperature and further decreases when the NaOH/KOH ratio is changed from 80/20 to 50/50. Except for one data point (GP185, S=0.25), the data follow generally smooth and orderly trends typical of previous laboratory results. The type of caustic (laboratory, chemically pure grade versus commercial grade obtained in drums) seems to have no effect. The data do not distinguish a difference between 6 mesh x 0 and 100 mesh x 0 particle size or between high ash ROM and mine cleaned coal. Half speed stirring (GP230) and intermittent stirring (GP231) gave substantially the same results as a normally stirred test (GP176). Tests GP141 and 142 involved splitting the water washed filter cake, washing one-half with hydrochloric acid and washing the other half normally with sulfuric acid. The hydrochloric acid wash produced coal about 20% lower in sulfur and 10% lower in ash than the sulfuric acid. This effect is small and nearly within the experimental variation, but should be noted in future acid wash studies.

Earlier in Section 2.2, the observed lower heat of combustion with lower residual sulfur for a Kentucky No. 11 coal was discussed. A similar effect is observed in Figure 6 which also shows the NSPS requirements for residual SO_2 for this coal. Even with the low sulfur starting coal, the SO_2 removal requirements are exceeded by 6* of the tests. To meet the standards, the caustic needs to be 50% KOH or more and the reactor conditions either at 390° for 2 hours or 370° for 6 hours. Nearly three-quarters of the results meet the older 1.2 lb of SO_2 million Btu level (23 of 32), but Category 2 coal must now meet the tougher 0.6 lb/million Btu level.

2.6 PITTSBURGH #8 REPOSITORY COAL EVALUATION

Section 2.3 compared four different lots of Pittsburgh #8 coal. This section of the report describes each of the additional tests conducted using

---

*One test, GP181, has an unexpectedly low sulfur analysis and may not be valid.

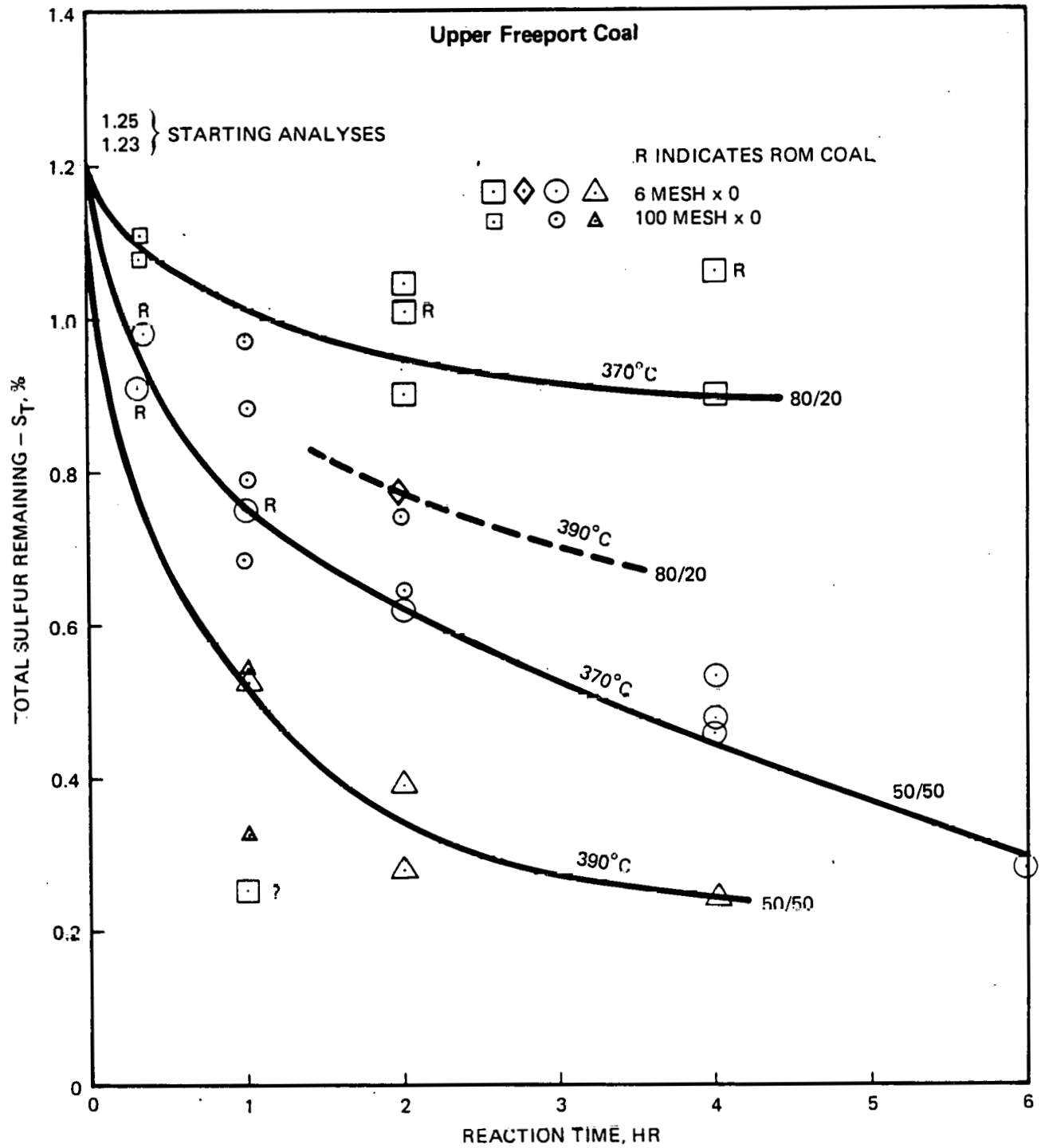


Figure 5. Sulfur Removal from Upper Freeport Coal

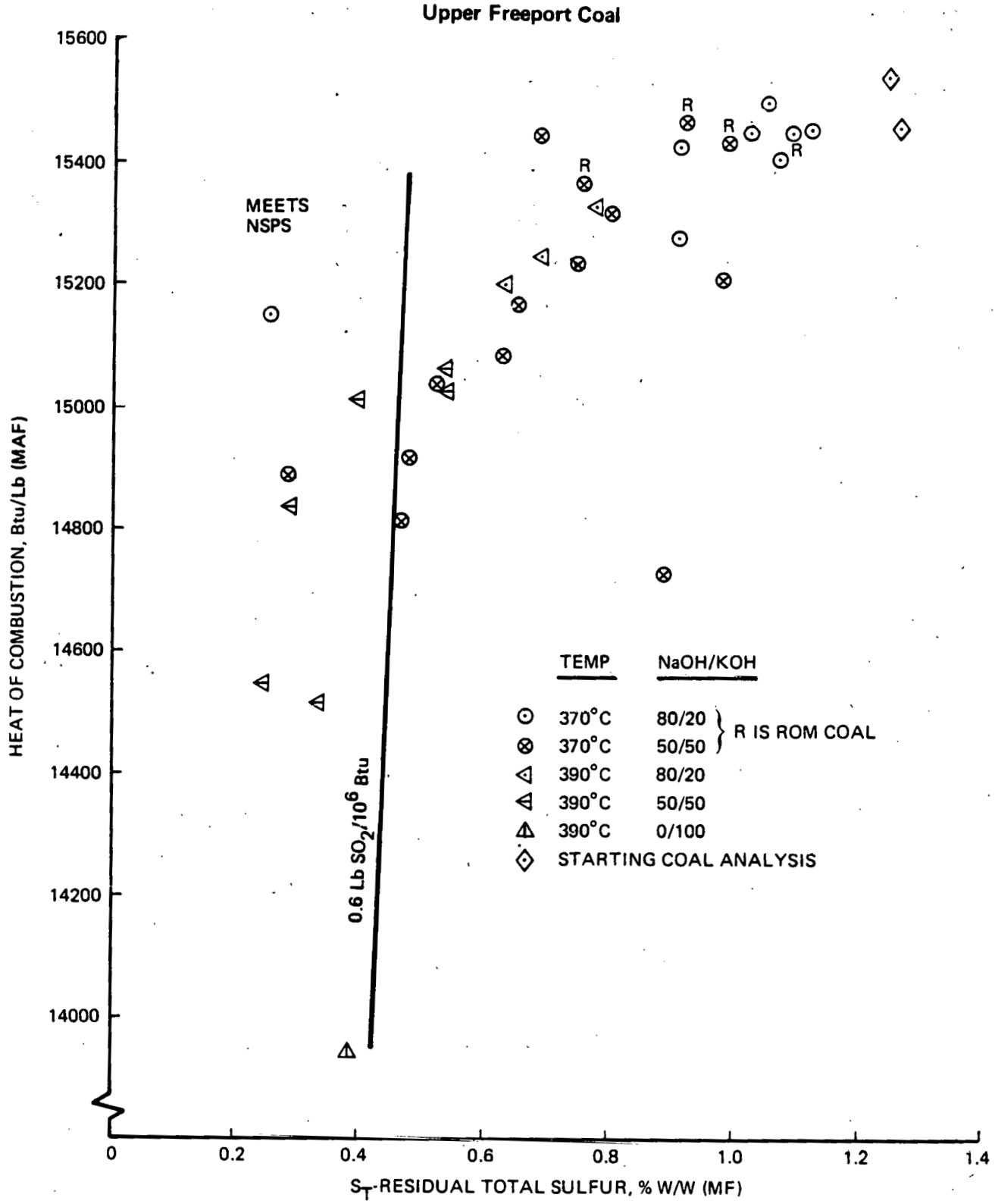


Figure 6. Heat of Combustion and Residual Sulfur for Processed Upper Freeport Coal

Pittsburgh #8 coal from the coal repository. Duplicate analyses were provided by Commercial Testing and Engineering, Chicago, for this 6 mesh x 0 coal and the average ROM values were provided by the DOE.

Pittsburgh No. 8	Mine Cleaned		ROM
	#1	#2	Average
Ash, % (MF)	10.34	10.34	30.2
S_t, % (MF)	4.23	4.20	3.7
Btu/lb (MF)	13265	13298	8871
Btu/lb (MAF)	14795	14832	12709
S_p, % (MF)	2.17	2.16	-
S_s, % (MF)	.03	.04	-
S_o, % (MF)	2.03	2.00	-
Moisture, %	5.87	5.91	-
SO_2, lb/10^6 Btu	6.38	6.32	8.34

A total of 28 laboratory tests of repository coal were conducted over a wide range of conditions. The test results, as abstracted from Appendix A, are shown in Table 5, in the same format as the Upper Freeport results. Compared with the Upper Freeport testing, the Pittsburgh coal tests involved a greater emphasis on the influence of particle size and method of preparation (sieving to size versus grinding to size), as discussed in Section 2.3 and 2.4. As a result, fewer tests were performed to show the effect of reaction time, temperature and caustic ratio on sulfur removal. Nine tests were conducted with 45 x 200 mesh particle size coal the same as was used during the previous laboratory program. All tests were one hour residence time and gave the following results:

Temp°, C	NaOH/KOH	No. of Tests	SO_2 lb/million Btu	Removal, % (from ROM)
340	50/50	3	1.60 ± .15	80.8
370	80/20	1	1.11	86.7
370	50/50	5	.84 ± .18	89.9

TABLE 5. LABORATORY RESULTS WITH PITTSBURGH 8 REPOSITORY COAL

Test No.	Temp °C	Caustic NaOH/KOH	Caustic Type	Time, hr	Mesh Size	MF S,%	MAF Btu/lb	lb/million Btu (MF) Ash	lb/million Btu (MF) SO_2	% Removal* Ash	% Removal* SO_2	Remarks
GP165	330	80/20	C	1	6x0	2.30	14347	2.06	3.30	93.9	60.4	
GP166	330	80/20	C	2	6x0	2.02	14624	1.77	2.83	94.8	66.0	
GP208	340	50/50	C	1	45x200	1.13	14454	.24	1.57	99.3	81.2	ground to -45, sieved to +200
GP209	340	50/50	C	1	45x200	1.05	14327	.24	1.47	99.3	82.4	sieved from 6x0
GP210	340	50/50	C	1	45x200	1.27	14401	.33	1.77	99.0	78.8	sieved from 6x0
GP171	370	80/20	C	1	6x14	2.30	14668	2.54	3.25	92.5	61.0	
GP171	370	80/20	C	1	6x14	2.25	14294	2.32	3.25	93.2	61.0	double acid wash
GP185	370	80/20	C	1	6x14	1.58	13849	2.28	2.35	93.3	71.8	
GP172	370	80/20	C	1	14x24	2.22	14534	1.76	3.13	94.8	62.4	
GP186	370	80/20	C	1	14x24	1.95	14398	1.56	2.77	95.4	66.8	
GP175	370	80/20	C	1	24x100	1.42	13467	.73	2.13	97.8	74.5	
GP177	370	80/20	C	1	100x0	1.74	13619	.99	2.59	97.1	69.0	
GP167	370	80/20	C	1	6x0	1.59	14208	1.33	2.28	96.1	72.7	
GP180	370	80/20	L	1	45x200	.79	14314	.46	1.11	98.6	86.7	
GP168	370	80/20	C	2	6x0	.99	14171	1.24	1.42	96.4	83.0	
GP213	370	80/20	C	2	6x0	.98	14300	.86	1.39	97.5	83.4	
GP173	370	80/20	C	4	6x0	.55	14034	.37	.79	98.9	90.5	
GP179	370	50/50	L	1	45x200	.56	13799	.20	.81	99.4	90.2	sieved from 6x0
GP198	370	50/50	C	1	45x200	.80	13889	.41	1.16	98.8	86.1	sieved from 6x0
GP199	370	50/50	C	1	45x200	.54	13702	.04	.79	99.9	90.5	sieved from 6x0
GP200	370	50/50	C	1	45x200	.52	13767	.04	.76	99.9	90.9	ground to -45, sieved to +200
GP201	370	50/50	C	1	45x200	.48	13888	.11	.69	99.7	91.7	Ground to -45, sieved to +200
GP190	370	50/50	C	2	6x0	.42	13815	.47	.61	98.6	92.7	
GP194	370	50/50	C	2	6x0	.58	13636	1.06	.86	96.9	89.7	
GP195	370	50/50	C	4	6x0	.54	13672	.36	.79	98.9	90.5	
GP197	370	50/50	C	4	6x200	.41	13583	.00	.60	100.0	92.8	
GP214	390	80/20	C	2	6x0	.31	13871	.10	.45	99.7	94.6	
GP196	390	50/50	C	2	6x0	.48	13207	.35	.73	99.0	91.2	

*Removal calculated from ROM coal with ash 34.04, SO_2 8.34 lb/10^6 Btu. Physical cleaning reduced the ash by 77.1% and the sulfur by 23.9%.

These results show that 90% removal of SO_2 was obtained for five samples at 370° with a 50/50 caustic ratio and one hour of reaction time. The process coal had an average of 0.2% ash (99+% removal) and a 13,780 Btu/lb heat of combustion on a moisture free basis.

Eleven tests were performed with the full size range coal (6 mesh x 0) with sulfur removal results as shown in Figure 7. Also included in Figure 7 are two fine grind coal results. The sulfur removal trends are relatively smooth and as expected, except that the single test (GP196) at 390°C with a 50/50 caustic ratio, appears to be about 0.3% higher in sulfur than would be predicted. The curves appear to extrapolate to values above the 2.0% organic sulfur level at zero time. With the relatively large amount of pyritic sulfur (2.2%) it is supposed that complete pyrite removal requires longer than one hour. Therefore, the short residence time sulfur values are believed to include pyritic sulfur as well as organic sulfur. This assumption should be verified by additional testing in which sulfur forms are obtained on samples processed over a range of reaction times, but with other parameters such as temperature and caustic ratio held constant.

Figure 8 shows the residual sulfur level and the corresponding heat of combustion for each of the tests listed in Table 5. It can be seen that 11 of the 28 samples produced coal that meet NSPS for SO_2 removal. This included six samples of the full size range (6 mesh x 0) repository coal which averaged 0.6% ash (98+% removal) and 13,720 Btu/lb (MF). To meet the NSPS, the coarser, full-size range Pittsburgh No. 8 coal requires about two hours of processing at 370°C rather than one hour needed for the 45 x 200 mesh size fraction. The resulting coarse coal contains about 0.6% ash rather than 0.2% for the finer coal. This is consistent with the particle size effect reported in Section 2.4.

2.7 OTHER REPOSITORY COALS

In addition to the Pittsburgh #8 repository coal discussed in the previous section, two additional repository coals were tested. For each coal,

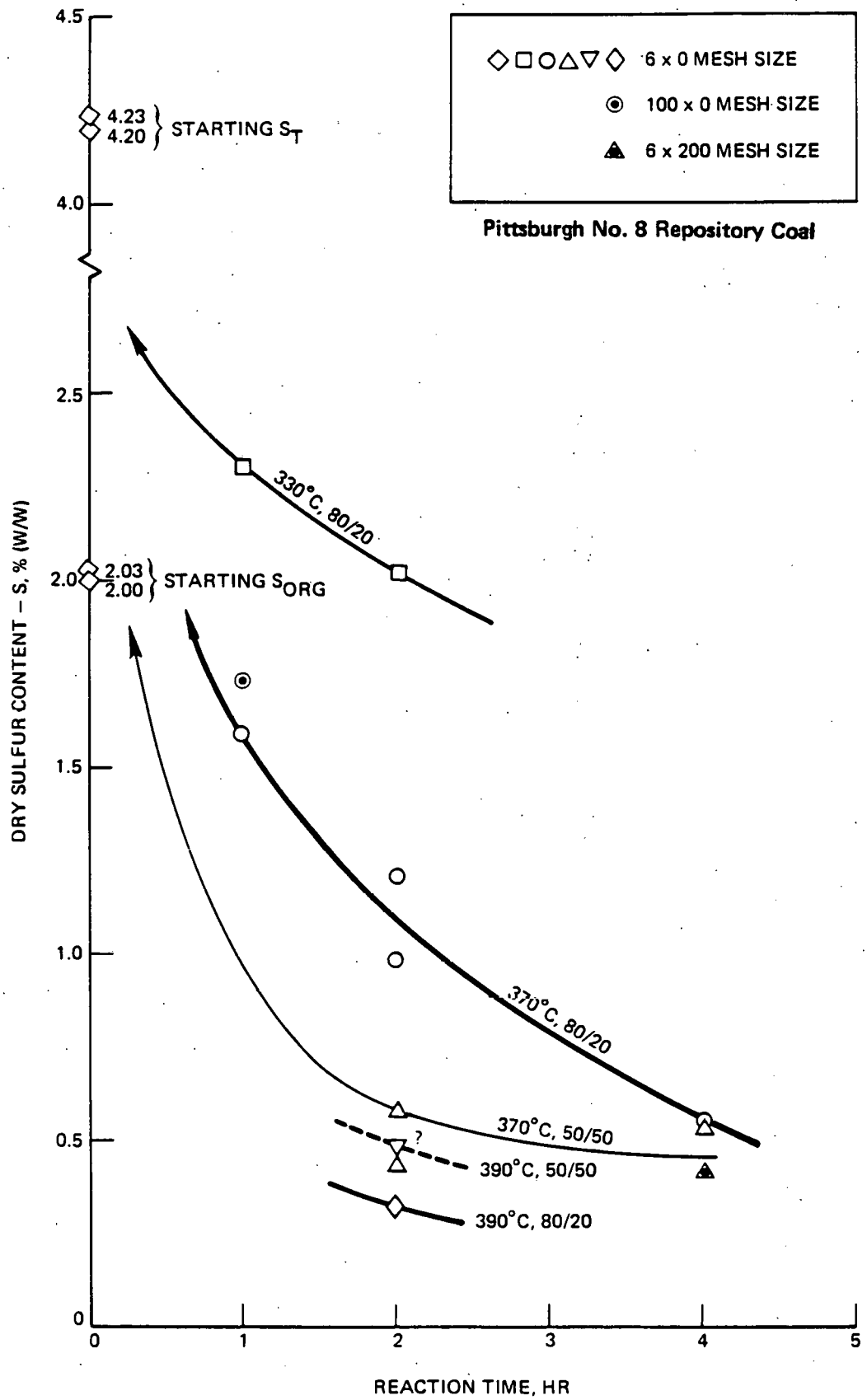


Figure 7. Sulfur Removal from Pittsburgh No. 8 Coal

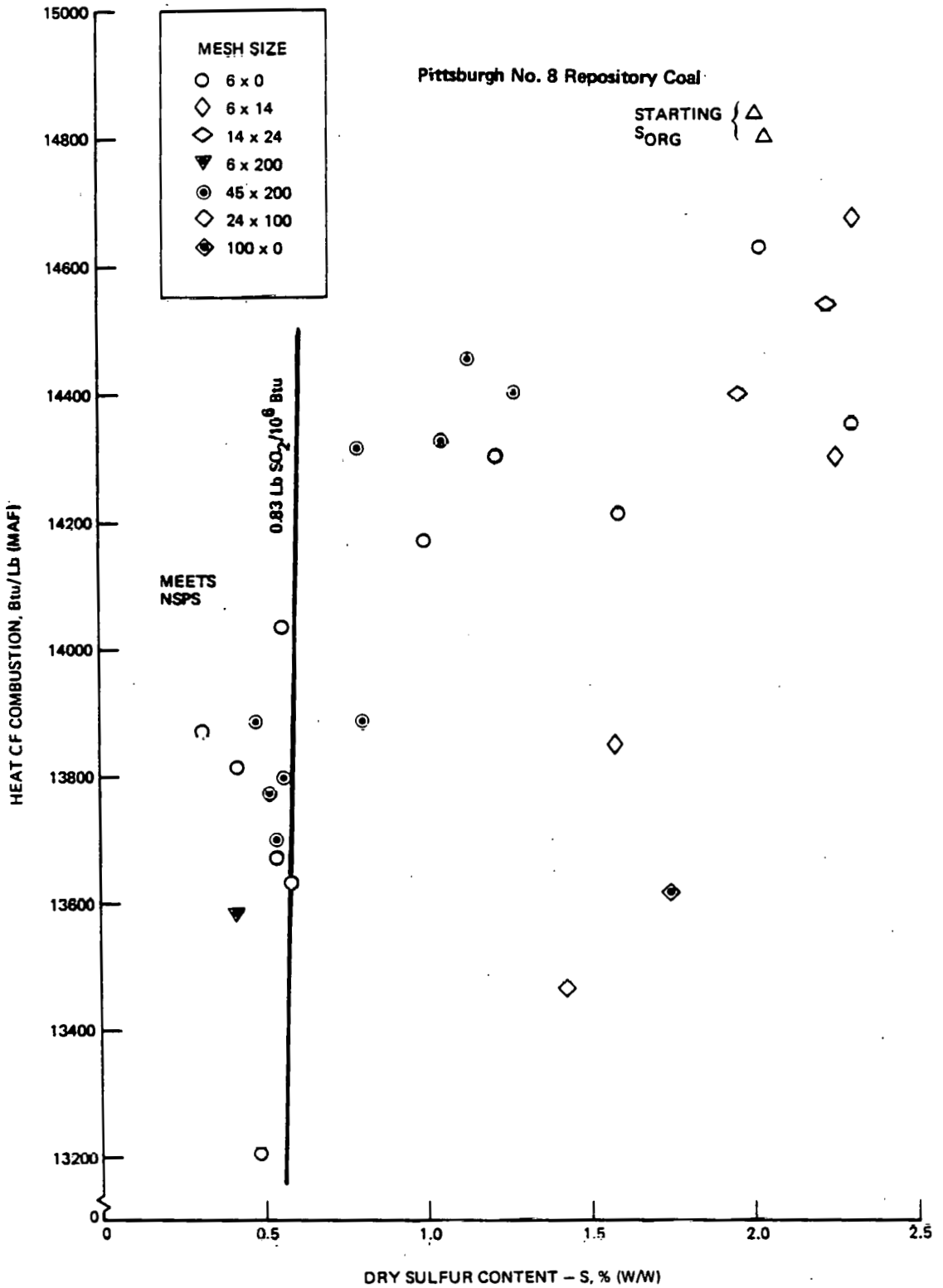


Figure 8. Heat of Combustion and Residual Sulfur for Processed Pittsburgh No. 8 Coal

duplicate analyses of the nominal 14 mesh size (actually 6 mesh x 0) were supplied with the coal by Commercial Testing and Engineering and the average ROM values were provided by the DOE.

	Kentucky No. 11			Illinois No. 6		
	Mine Cleaned		ROM	Mine Cleaned		ROM
	#1	#2	Average	#1	#2	Average
Ash, % (MF)	10.72	10.76	29.0	10.03	9.91	24.2
S_t, % (MF)	3.30	3.37	4.6	4.18	4.23	5.0
Btu/lb (MF)	12985	12996	9092	12790	12755	8272
Btu/lb (MAF)	14544	14563	12806	14216	14158	10913
S_p, % (MF)	1.53	1.53	-	1.35	1.36	-
S_s, % (MF)	.06	.07	-	.04	.05	-
S_o, % (MF)	1.71	1.77	-	2.79	2.82	-
Moisture, %	9.52	9.57	-	15.99	15.99	-
SO_2, lb/10^6 Btu	5.08	5.19	10.12	6.54	6.63	12.09

A total of twelve laboratory tests were conducted using the Kentucky coal and ten tests using the Illinois coal. These tests are given in Table 6 as abstracted from Appendix A. The Kentucky tests were conducted at 350°C with an NaOH/KOH ratio of 80/20, and at 370°C with both an 80/20 and a 50/50 ratio.

Figure 9 shows the sulfur removal results for the Kentucky coal. With an 80/20 NaOH/KOH ratio, sulfur reaches its minimum value for the coarse 6 mesh coal (shown as open symbols) at about three hours and is not further reduced by processing for seven hours. For both the 350°C, 80/20 tests and the 370°C, 50/50 tests, residual sulfur in the finer, 45 mesh, coals (one test point at each condition) was lower than in the coarse 6 mesh coal. At the other test condition (370°C, 80/20), the three fine coal results (filled square symbols) are as expected but the single coarse coal result shows about one-half of the expected residual sulfur level. The small influence of the two particle size distributions on sulfur removal from the Kentucky coal is similar to the weak effect reported for the Pittsburgh coal in Sections 2.4 and 2.6. For the Kentucky coal, the ash removal is affected about the same as the sulfur removal. Additional fine coal testing is needed to determine the final sulfur level and the corresponding minimum reaction time obtainable with fine coal.

TABLE 6. LABORATORY RESULTS WITH KENTUCKY 11 AND ILLINOIS 6 COALS

Test No.	Temp., °C	Caustic NaOH/KOH	Caustic Type**	Time, hr	Mesh Size	MF S,%	MAF Btu/lb	lb/million Btu (MF) Ash	lb/million Btu (MF) SO_2	% Removal* Ash	% Removal* SO_2
Kentucky No. 11 - Repository											
GM198	350	80/20	U	2	45x0	.76	14225	.30	1.07	99.1	89.4
GM199	350	80/20	U	2	6x0	1.04	14241	.46	1.47	98.6	85.5
GM201	350	80/20	U	3.5	6x0	.52	13952	.24	.75	99.3	92.6
GM202	350	80/20	U	7.2	6x0	.51	13840	.32	.74	99.0	92.7
GM200	370	80/20	L	.5	45x0	1.22	14344	.41	1.71	98.7	83.1
GM200A	370	80/20	L	.5	45x0	1.14	13947	.43	1.64	98.6	83.7
GM197	370	80/20	L	.5	45x0	1.51	14324	.54	2.12	98.3	79.0
GP183	370	80/20	L	1	6x0	.66	13287	.29	1.00	99.1	90.1
GP234	370	50/50	C	.5	45x200	.58	13489	.21	.86	99.3	91.5
GP224	370	50/50	C	.5	6x0	1.31	13843	.68	1.91	97.9	81.1
GP223	370	50/50	C	1	6x0	1.26	13883	.83	1.84	97.4	81.9
GP233	370	50/50	C	1	6x0	1.11	13593	.61	1.65	98.1	83.7
Illinois No. 6 - Repository											
GM194	330	80/20	U	2	6x0	.93	14582	.67	1.29	97.7	89.3
GP215	350	80/20	C	2	6x0	1.43	13861	.64	2.08	97.8	82.8
GP187	350	50/50	C	2	6x0	2.03	13871	2.237	3.01	92.4	75.0
GP189	350	50/50	C	4	6x0	.57	13423	.25	.85	99.1	93.0
GP226	370	80/20	C	.5	6x0	2.06	13918	1.93	3.04	93.4	74.9
GP182	370	80/20	L	1	6x0	1.08	13287	1.12	1.65	96.2	86.4
GP228	370	80/20	C	1	6x0	1.71	13800	1.37	2.53	95.3	79.1
GP216	370	80/20	C	2	6x0	.79	13784	.81	1.16	97.2	90.4
GP229	370	80/20	C	2	6x0	1.08	13885	.67	1.57	97.7	87.0
GP188	370	50/50	C	2	6x0	.34	12460	.31	.55	99.0	95.5

*Removal calculated From RGM coals, Kentucky #11 ROM has ash 31.90, S 10.13 lb/10^6 Btu. Mine cleaning reduced the ash by 74.1% and sulfur by 49.3%. Illinois ROM has ash 29.26, S 12 09 lb/10^6 Btu. Mine cleaning reduced ash by 72.2% and sulfur by 45.6%.

**Caustic type in addition to laboratory grade (L) and commercial grade (C), includes reused commercial grade (U) to evaluate the effect of mineral buildup.

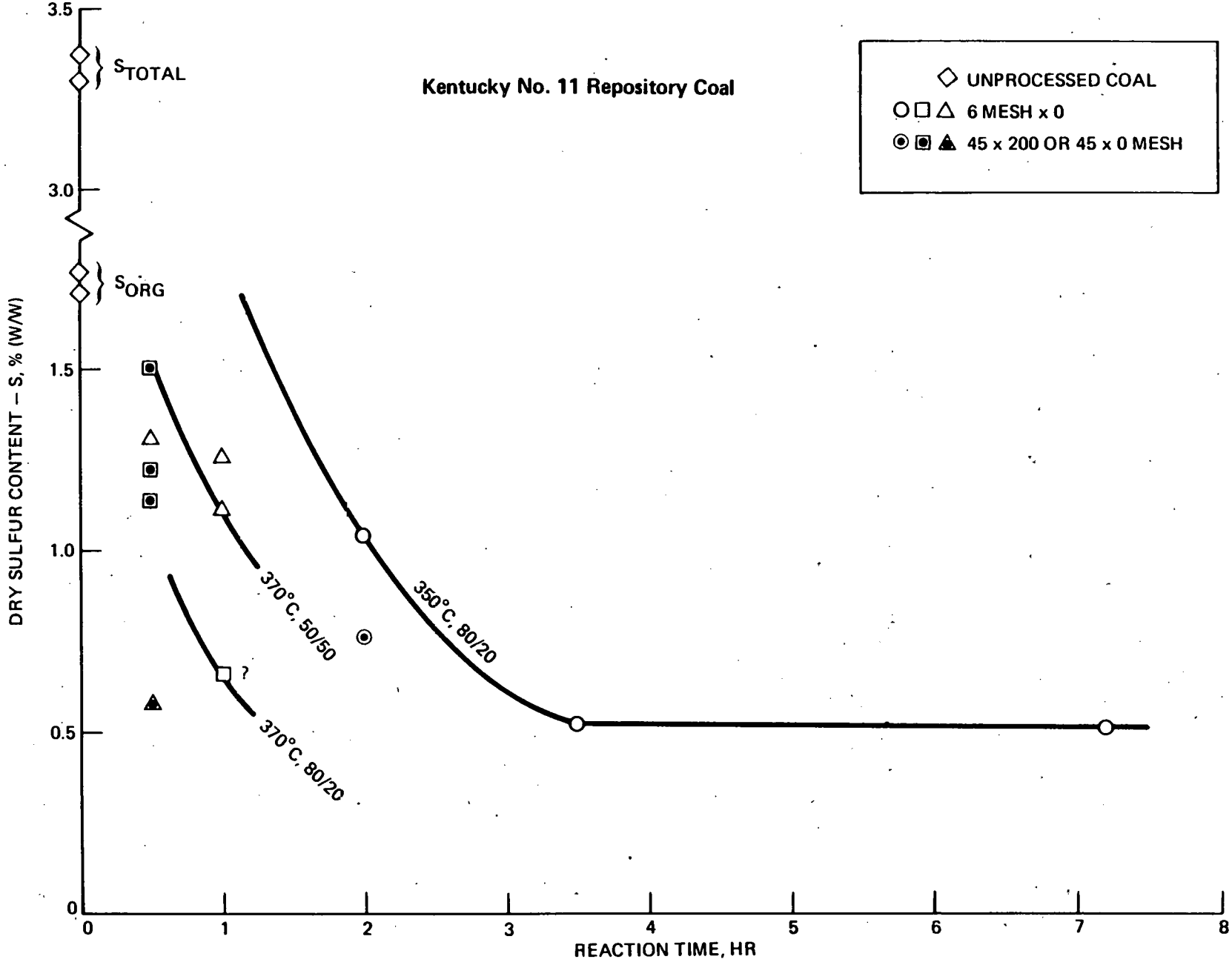


Figure 9. Sulfur Removal from Kentucky 11 Coal

The residual sulfur and corresponding heat of combustion on a moisture free basis are shown in Figure 10 for the Kentucky coal tests. Four of the twelve tests meet the NSPS. These data show that sulfur is removed from the Kentucky coal under milder conditions than required by the Upper Freeport and Pittsburgh coals. For the 45 x 200 mesh, one-half hour at 370° with a 50/50 caustic ratio gave 92% removal. The full size range (6 mesh x 0) processed with a milder 80/20 caustic ratio needed one hour at 370° to give 90% removal or about 3 hours at 350°.

The final repository coal tested as a part of the program was Illinois #6 coal. The observed sulfur removal as a function of time is shown in Figure 11. All of the results follow the trends that are expected except for one test (GP-187) at 350°C, 50/50 which had 2 to 3 times the sulfur value expected for two hours of processing. The corresponding high heat of combustion and high ash value are consistent with the high sulfur and would tend to indicate the effective processing time was about one-half an hour instead of two hours. This test result is currently unexplained.

The residual sulfur and corresponding heat of combustion (MF) for the Illinois coal tests are shown in Figure 12. Three of the ten tests yield coal which meets the NSPS. In general, it has been found that the Illinois coal requires processing conditions about the same as Kentucky coal to meet the same percent removal. At 370° and an 80/20 caustic ratio, two hours gave a compliant product. At the more aggessive 50/50 caustic ratio, two hours at 370° (96% removal) and four hours at 350° (93% removal) gave greater sulfur removal than required and probable standards could be met at about one-half the residence time of the tests.

2.8 SUMMARY OF LABORATORY RESULTS

The goal of the TRW Gravimelt Process development effort is to provide a chemical coal cleaning technique that can produce compliance coal to meet the NSPS. Each of the four coals tested during the program were cleaned to the required levels. As a benefit of sulfur removal, ash was also lowered, typically by about 99% of its starting ROM value. Figure 13 shows the residual sulfur levels for the tests reported in Sections 2.4 and 2.7.

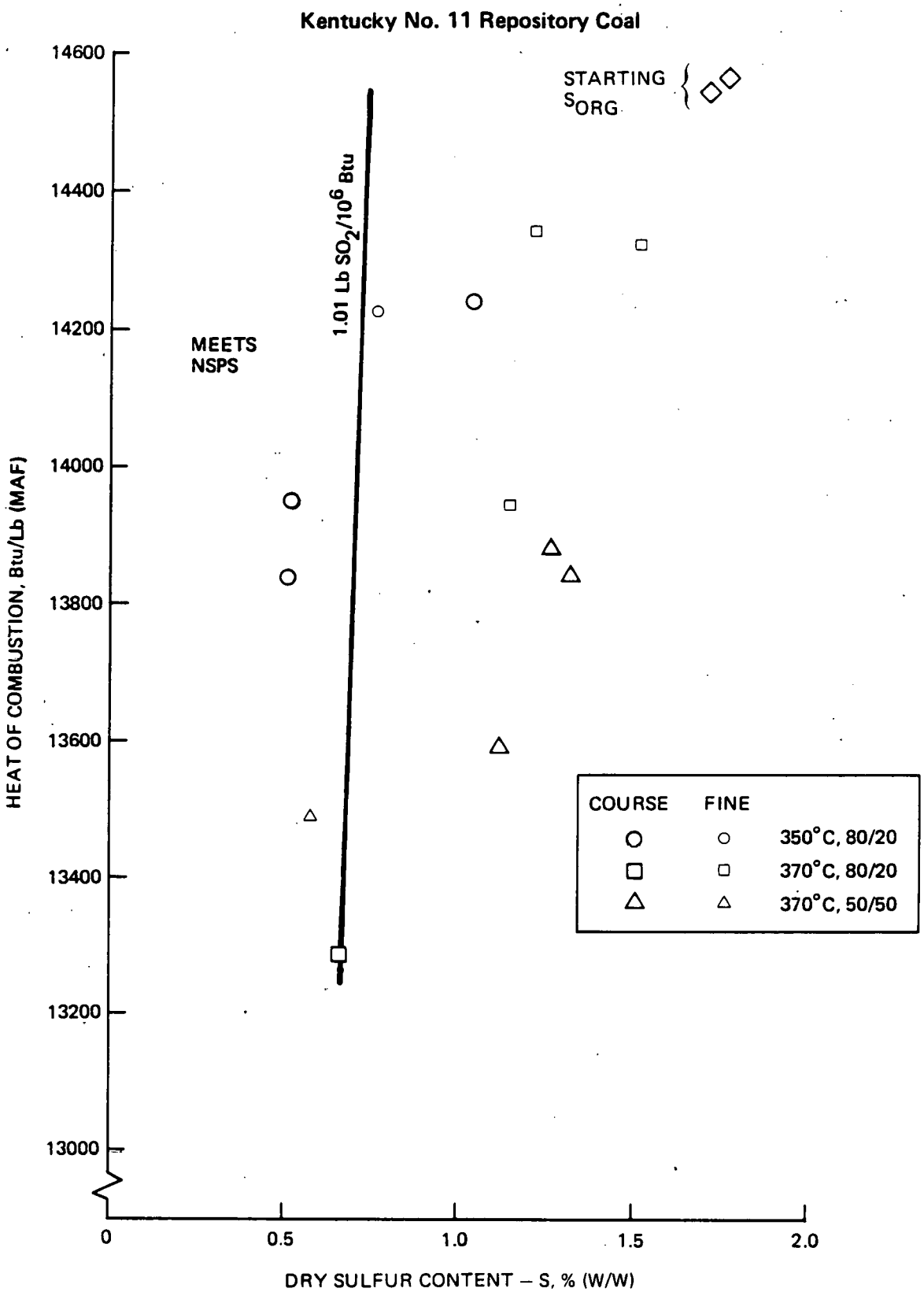


Figure 10. Heat of Combustion and Residual Sulfur for Processed Kentucky No. 11 Coal

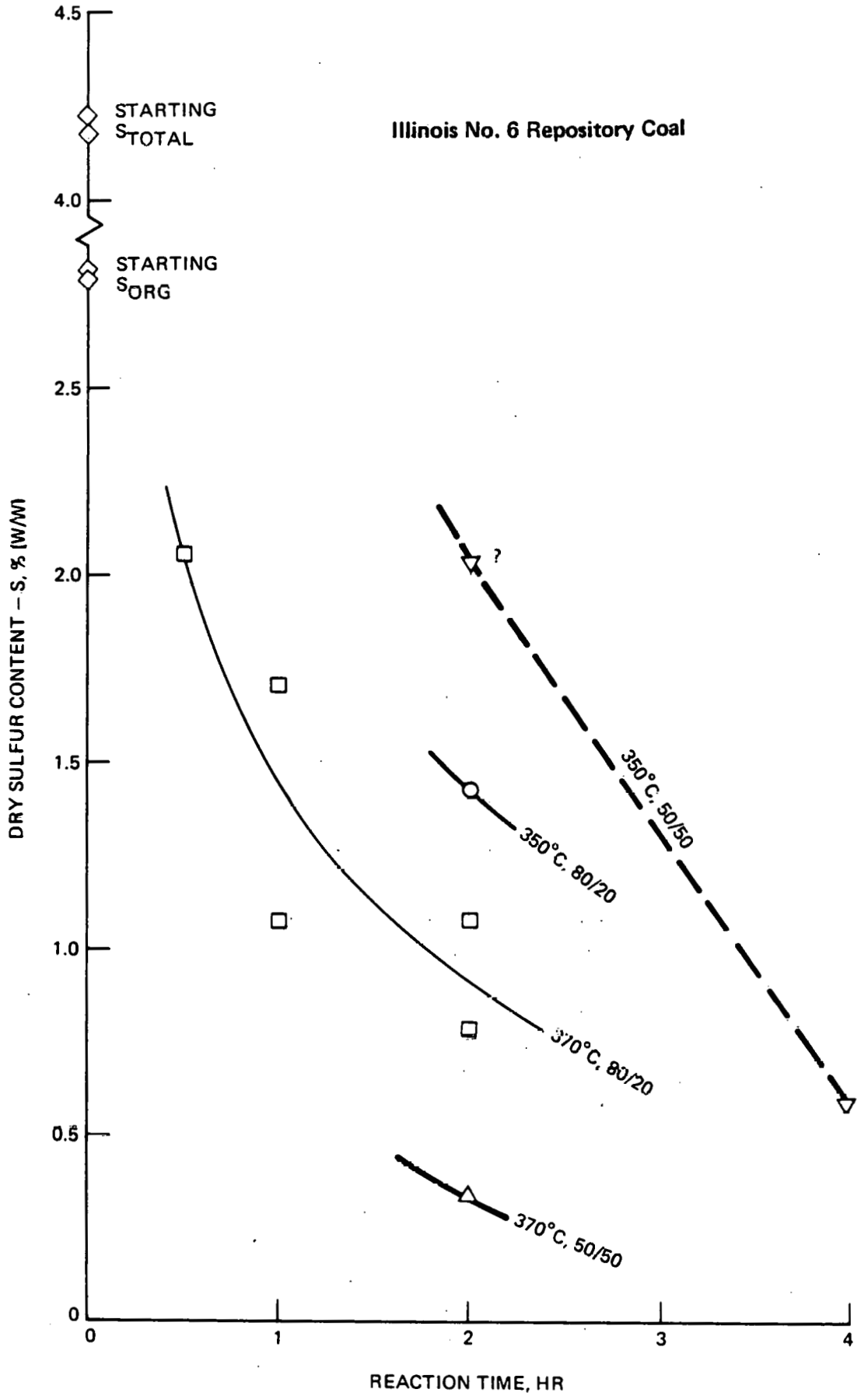


Figure 11. Sulfur Removal from Illinois Coal

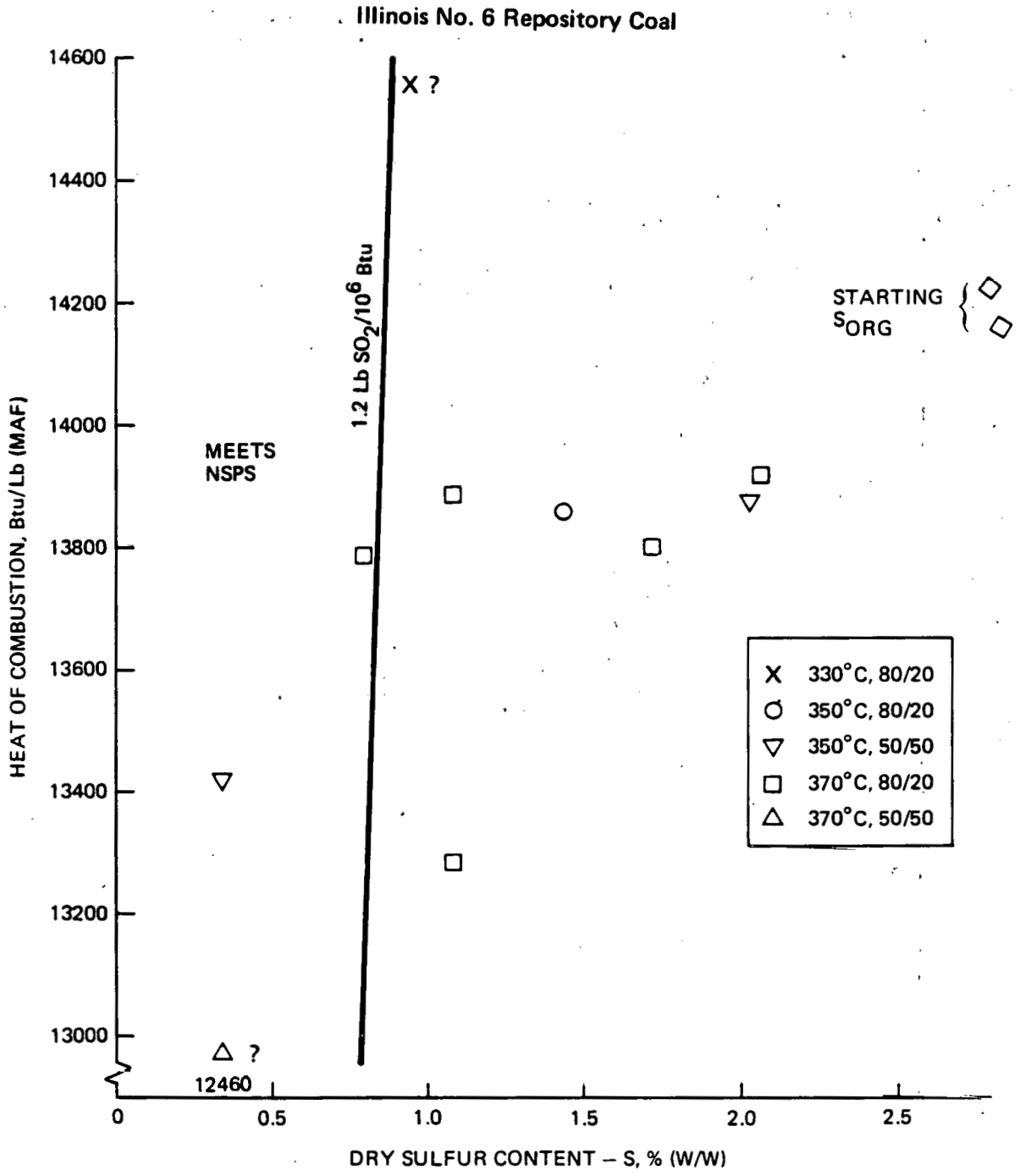


Figure 12. Heat of Combustion and Residual Sulfur for Processed Illinois No. 6 Coal

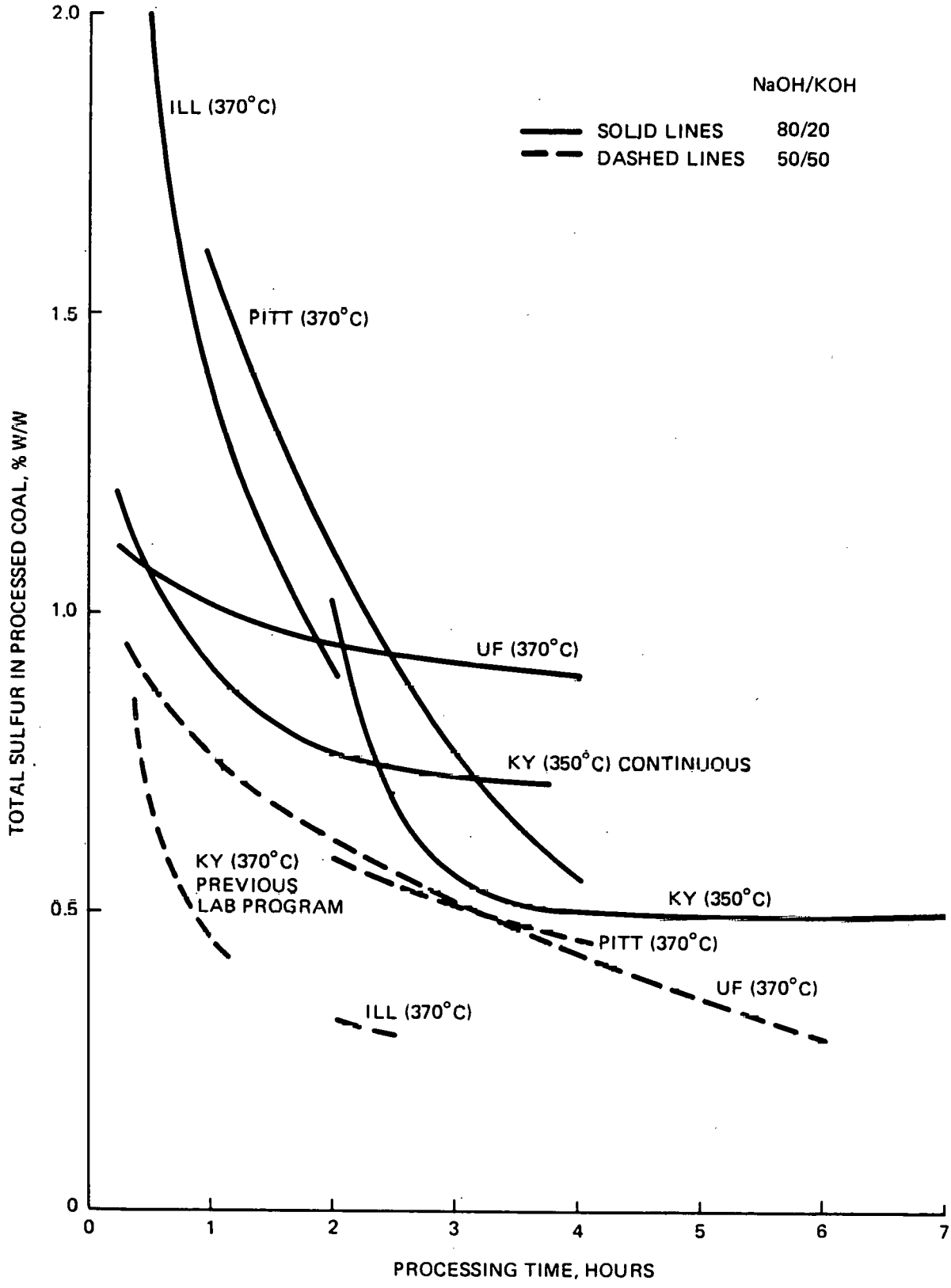


Figure 13. Summary of Sulfur Removal Results

In summarizing the cleaning results from each of the four coals, it is clear that coal processed in an 80/20 NaOH/KOH melt at 350°C to 370°C, to reach the target sulfur level usually requires a residence time of about four hours or more, if it can be reached at all. However, at 370°C and a 50/50 caustic ratio, the NSPS sulfur levels can be reached in times in the vicinity of two hours.

	Reaction Conditions to Meet NSPS	
Coal Seam	370°, 50/50	other, 50/50
Upper Freeport	4 to 5 hrs	390°, 2 hrs
Pittsburgh 8	2 hrs	-
Illinois 6	1 to 2 hrs	-
Kentucky 11	.5 to 1 hrs	350°, 2 hrs

Further trade-offs of time, temperature and caustic ratio with process economics will be required to select the best conditions for each particular feed coal. Before much additional laboratory data is obtained, it is suggested that actual or simulated recycled caustic be examined as a parameter. Processed coal must be hot separated from the melt and the melt repeatedly reused to build up impurities. Caustic which stays with the coal also should be washed from the coal and reconcentrated for reuse. Several tests with previously used caustic did not have either the moisture or the sulfur (sodium sulfide) content of recycled material. No effect was found and no major effect is expected, but impurities could influence the rate or ultimate extent of sulfur removal. If a difference is found, the offending material should be identified and the effect examined quantitatively in suitable laboratory experiments.

3.0 BENCH-SCALE TESTING

A key objective of this project was to begin the transitions from process studies performed at laboratory scale to reaction studies conducted in bench scale reactors capable of continuous, steady state operation. Most of the design concerns related to producing a near plug flow of the coal through caustic and avoiding caking of the dry feed coal, especially during the times just before and immediately following caustic contact.

The first design to be fabricated and tested is designated the Mod 1 reactor. Based on experience gained during operation of the Mod 1 reactor, a second (Mod 2) reactor was designed and operated. These reactors and the test results are described in the sections which follow.

3.1 MOD 1 REACTOR

The first reactor consised of an upright feed tube partially filled with caustic connected to the top of a large caustic filled reservoir which also had a vertical discharge tube partially filled with caustic. The reactor discharge tube was connected to a partly water filled quench tank by a scraper box as shown schematically in Figure 14. The design details and the test results are provided in the subsections which follow.

3.1.1 Design of the Reactor System

The reactor system at two stages of installation is shown in Figures 15 and 16. In the first photograph, the welded stainless steel reactor parts and the coal feeder are attached to show their relative locations. The second photograph shows the system with heaters, mixers, coal feed funnel and quench vessel (coal receiver) attached.

The principle of operation consists the following sequence of events. Caustic is added to and melted in the left side vessel heated by strip heaters until the desired liquid level is reached. Band heaters, attached to temperature controllers, cover all parts of the reactor and scraper box to

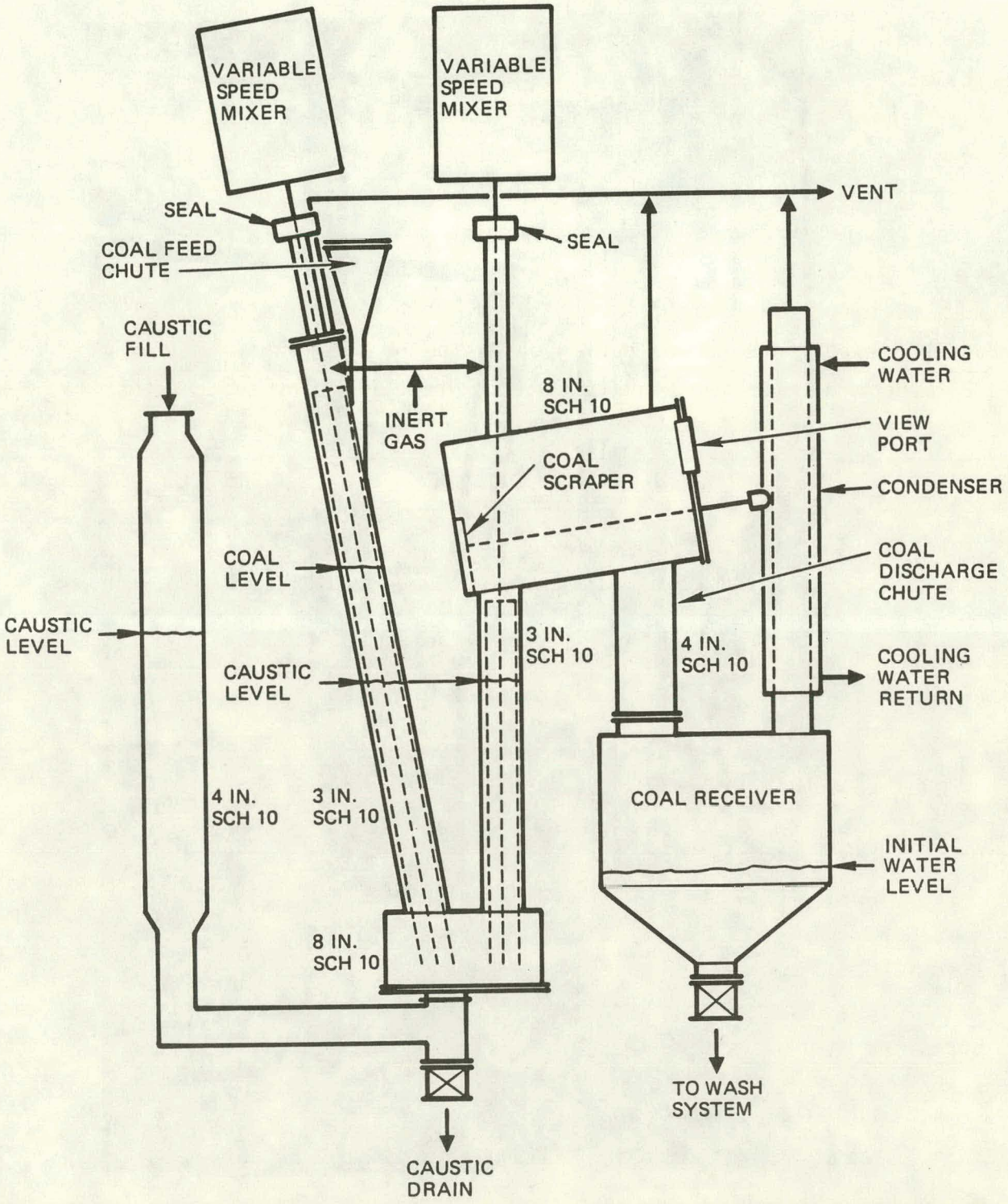


Figure 14. Mod 1 Reactor System Schematic

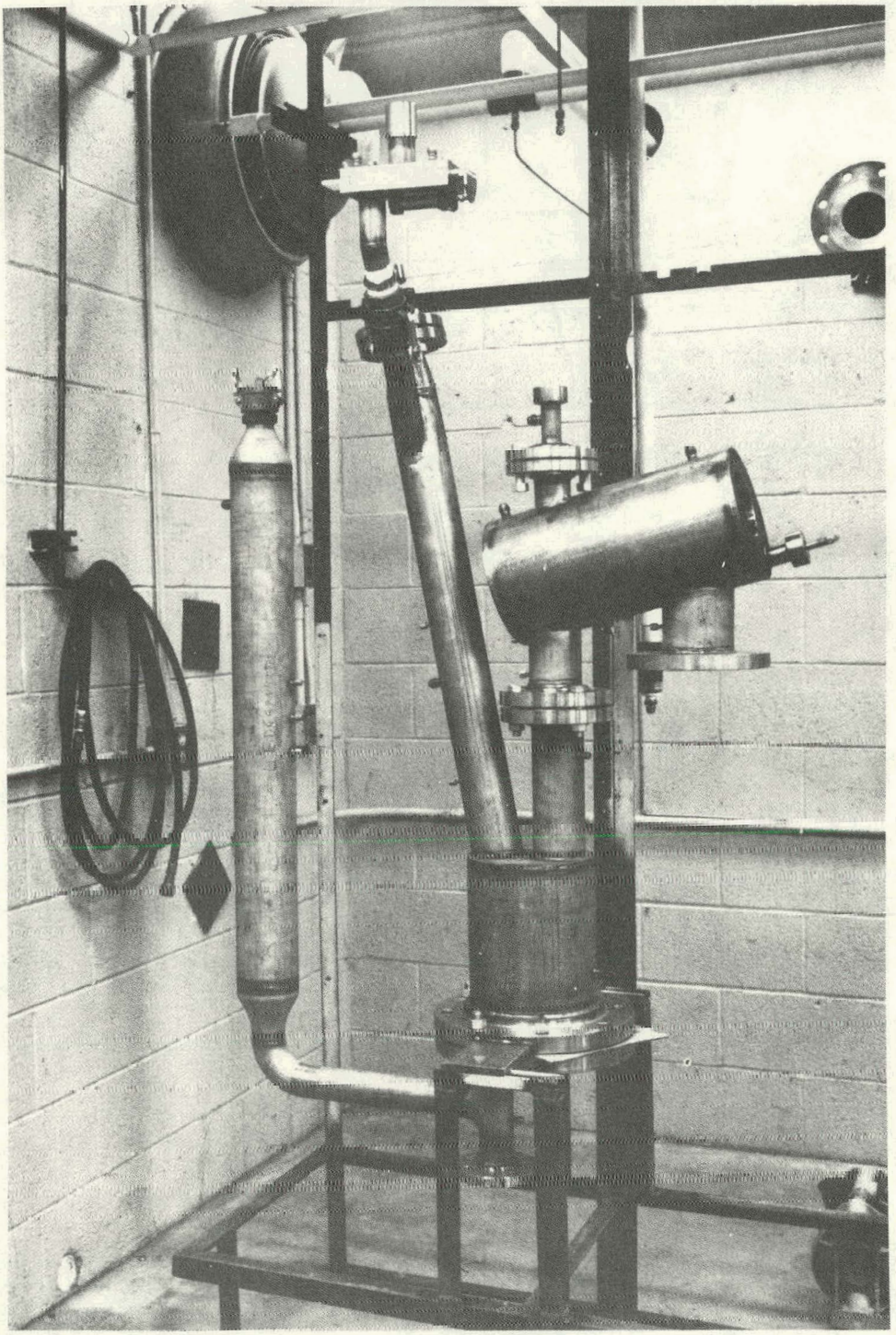


Figure 15. Mod 1 Reactor Weldment Assembly

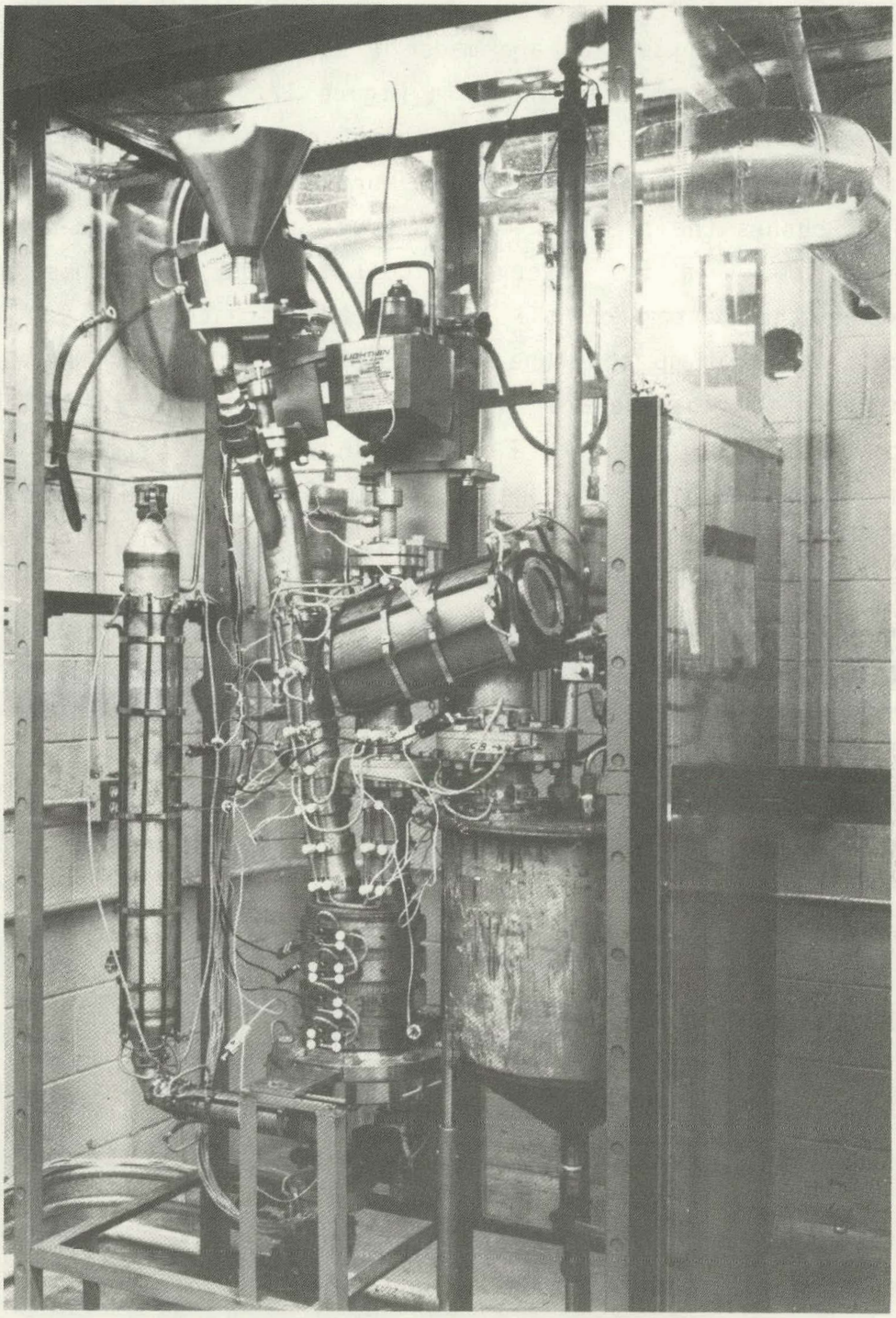


Figure 16. Mod 1 Reactor Complete Without Insulation

obtain the desired temperatures throughout the reactor system. Coal is added to the funnel at the top of the left side feed tube. Both the inlet and the outlet mixers are set at the desired speed.

The stirring shafts are made of stainless steel rods drilled to accept stainless steel pins as shown in Figure 17. These stirrers are to prevent coal from caking and thereby to assist the coal in passing down the feed tube and into the molten caustic. As more coal is added to the feed side, its weight pushes the previous coal downward until coal reaches the reservoir at the bottom. In the reservoir, coal spreads out across the top until it reaches the bottom of the right side discharge tube. The buoyant coal particles float up the tube and reach the surface of the caustic. As coal continues to float upward in the discharge tube, it raises the previous coal above the liquid level where excess caustic drains from the floating coal. In time, enough coal enters the tube that floating coal reaches the entrance to the scraper box. As often as needed, the coal is scraped from the exit of the discharge tube to the entrance of the coal discharge chute and dropped into quench water in the coal receiver. Steam generated by the hot coal and caustic is condensed and returned to the receiver.

At the conclusion of a test, the coal, mixed with aqueous caustic, is pumped to one of the three wash tanks shown in Figure 18. Here water or acidified water is added as desired and the resultant slurry is separated in a centrifuge shown in Figure 19. The centrifuge cake may be reintroduced into the wash tanks for further treatment or dried as product coal.

3.1.2 Testing of Mod 1

Illinois No. 6 Coal Testing

Shakedown testing of the reactor system, using Illinois #6 coal, was initiated with checkout runs of 2 hour duration at 350°C at a 5 pound per hour (pph) coal feed rate. It was found necessary to add heaters and gas purges to remove moisture from the feed side of the reactor. Without moisture control, plugging was experienced in the gas filter and coal feed system. Pressure relief was also added to prevent reactor pressure from rising higher than a few inches of water.

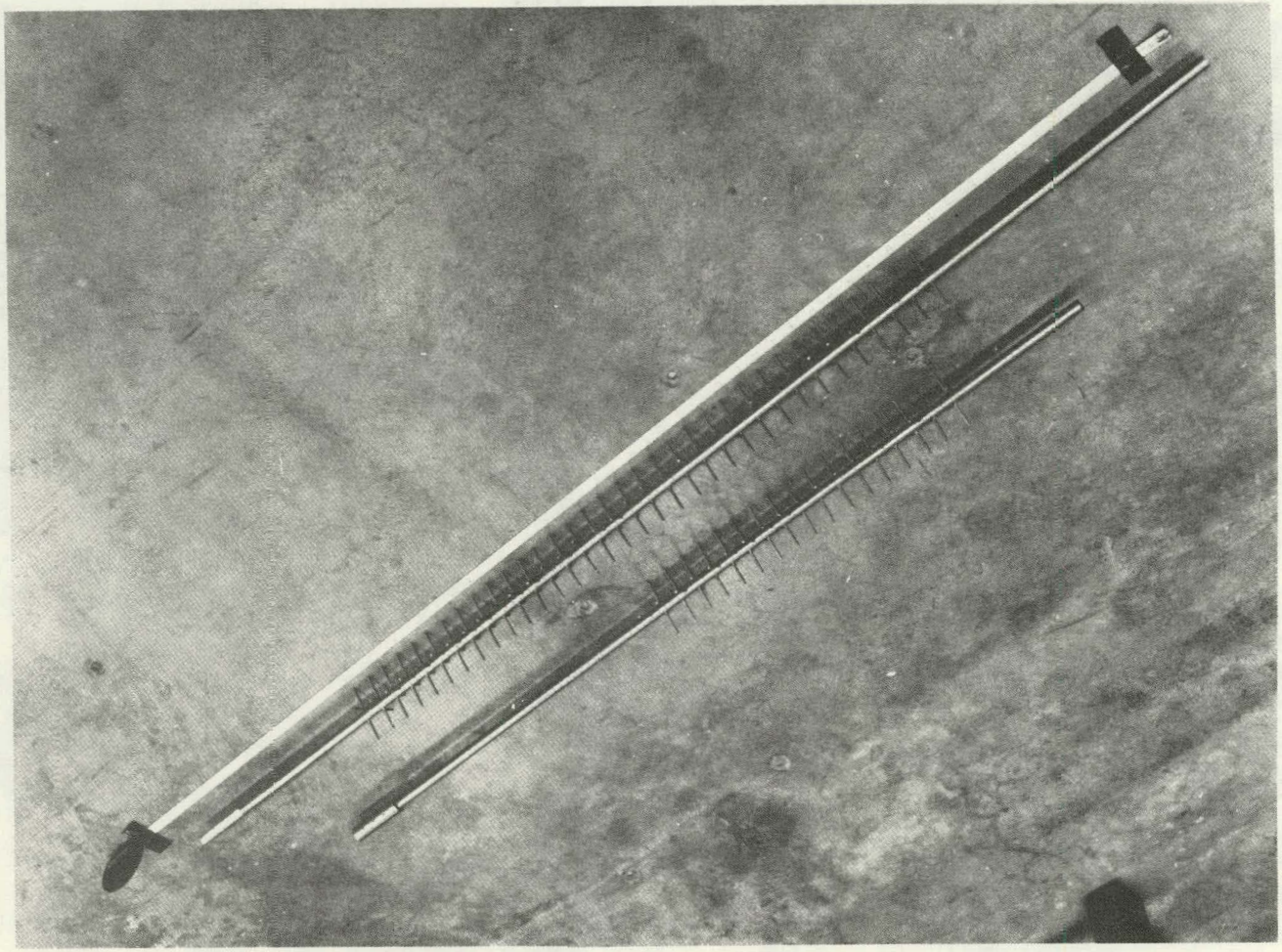


Figure 17. Stirring Shafts for Mod 1 Reactor Inlet/Outlet

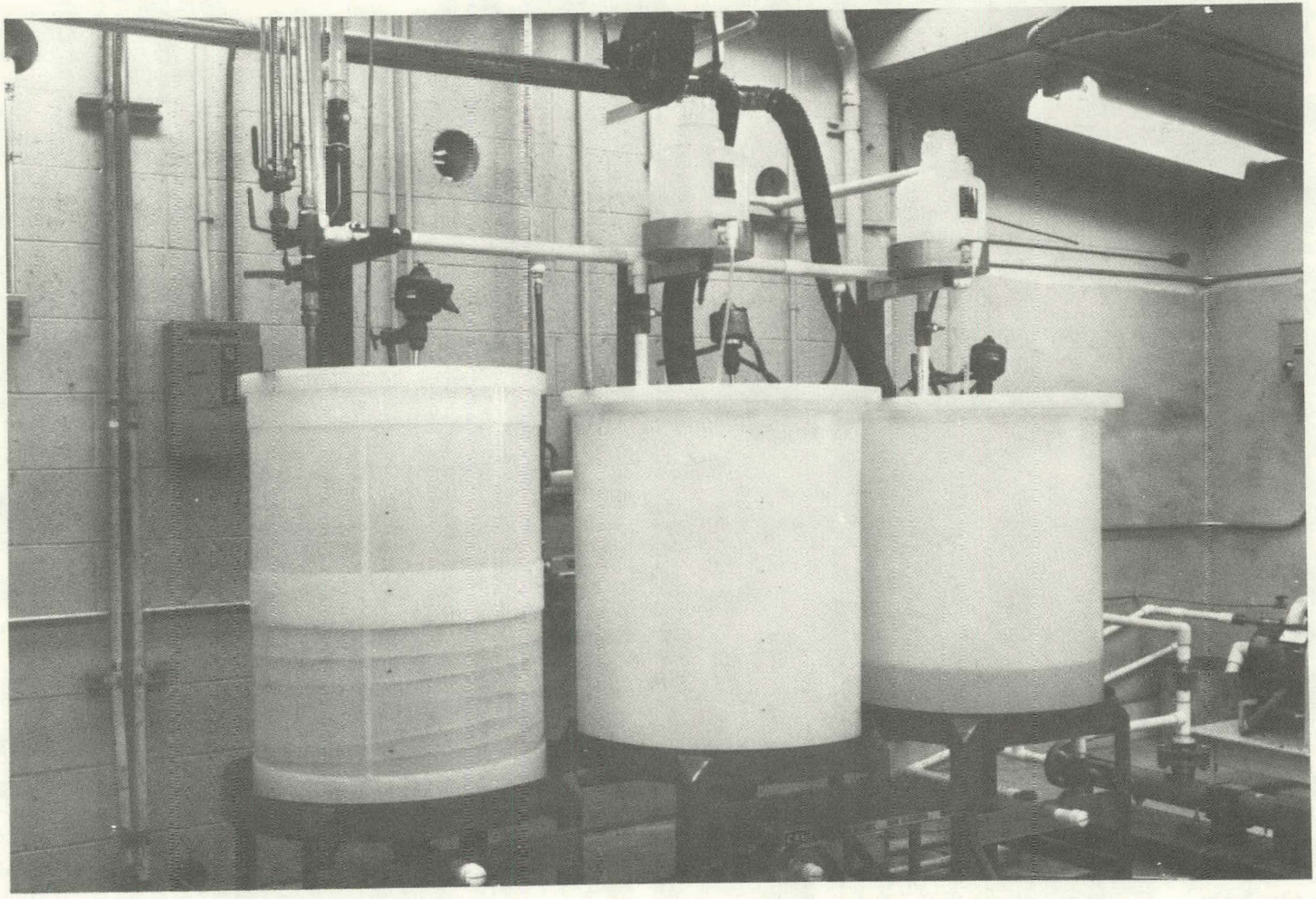


Figure 18. Cca' Wash and Acid Treatment System

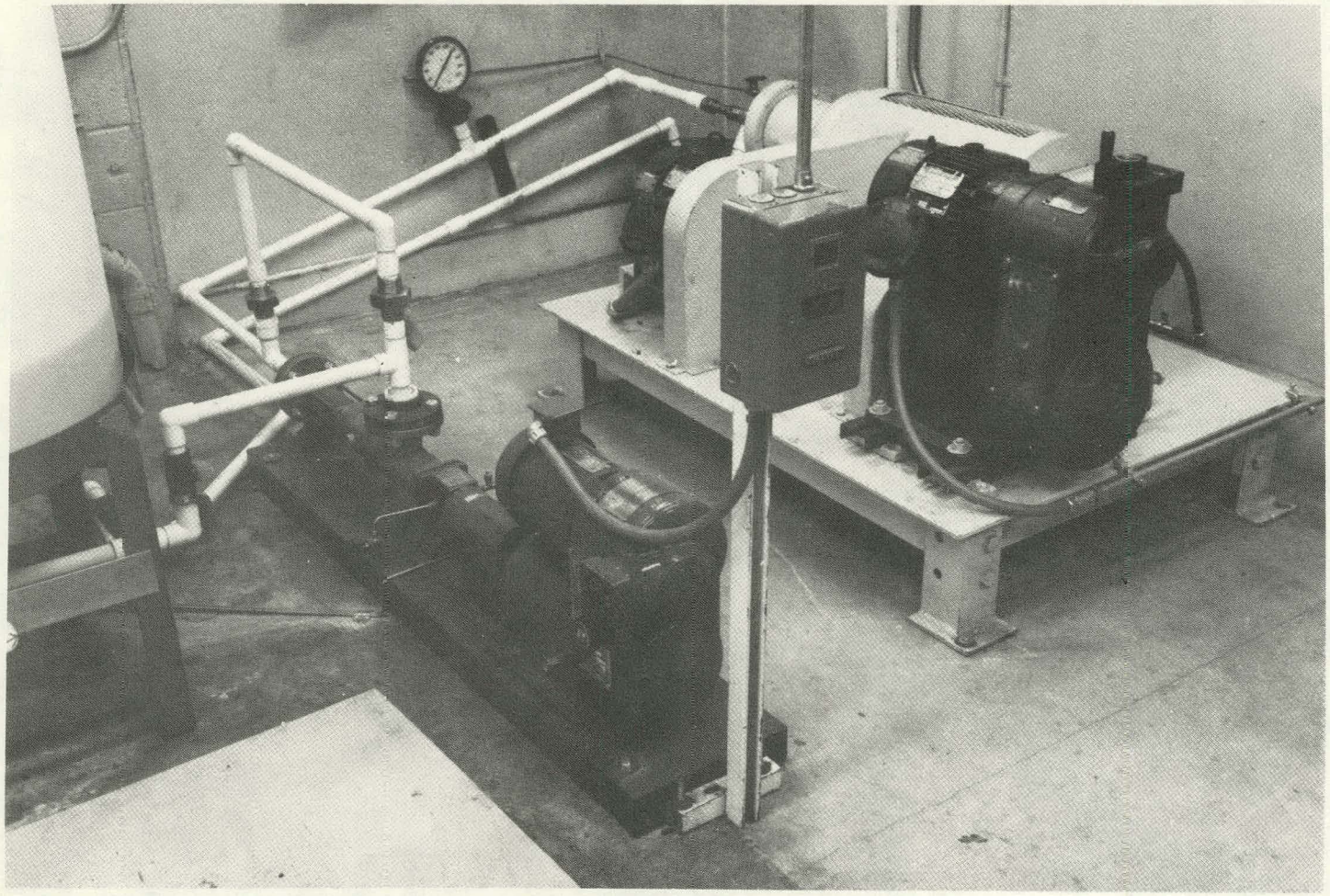


Figure 19. SS Solid-Bowl Bird Centrifuge With Back-Drive

During a shakedown test run, to confirm the suitability of the new heater arrangement, it was discovered that the feed-side stirrer shaft had failed. Metallographic and chemical testing showed that chloride crevice attack from residual zinc chloride, used in the hydraulic flow test prior to shakedown was a likely contributor to the failure. The reactor walls were examined by an ultrasonic thickness meter with no indication of general corrosion. Die penetrant studies identified some weld zone cracks above the caustic level that were repaired.

After the reactor was rechecked, Run 1 was started with 80/20 caustic at 370°C with 14 mesh x 0 Illinois #6 coal from the repository. Beginning about an hour into the run, plugs formed from moisture/coal mixtures and intermittent operation continued for about 5 hours. The following day, Run 1A was started, but plugging by moist coal fines prevented a useful test.

Dried repository coal was used for Run 1B and again about 5 hours of operation was obtained. A third test, Run 1C, was conducted at conditions similar to Run 1B and was terminated at about 5 hours when the coal feeder became sticky and erratic.

Upper Freeport Coal Testing

The first test of Upper Freeport coal used the 14 mesh x 0 sample that had been mine cleaned to about 10% ash at about 1.6 specific gravity. This test, Run 2, was at 350°C with a nominal coal feed rate of 2.5 pph. The run was terminated at three hours when a heater failed.

Run 3 was similar to Run 2, but at 370°C. The test was terminated at the end of the day with about 5 hours of operation. The following morning, Run 4 was initiated but plugging occurred in the feed side of the reactor and the product coal is believed to be residual coal from Run 3.

Pittsburgh No. 8 Coal Testing

Experience with previous reactor operation indicated that coal plugging tended to occur in the feed side of the reactor following lengthy reactor shutdowns (overnight). To overcome this behavior, 24-hour operation was planned. Runs 5 and 6 tested 14 mesh x 0 Pittsburgh #8 coal from the repository that had been vacuum dried. Run 5 was made at 370°C with a nominal coal feed rate of 2.5 pph. After about 7 hours of operation, the product coal receiver was changed for the next run.

Run 6 was conducted at 330°C but otherwise like Run 5. During both runs, the coal foamed vigorously and bubbled over into the quench receiver without assistance. The run was terminated after about 6 hours and the coal changed for the next test.

Upper Freeport High Ash (ROM) Testing

Run 7 was performed with the 38% ash ROM Upper Freeport coal sample. This test was conducted at 370°C under conditions similar to Run 5. Foaming did not occur and the caustic level was so low that most of the coal collected in the reactor reservoir and was not discharged to the quench received. The test was terminated after 6 hours of operation. The next planned test was not started because of a coal feed stoppage. The stoppage was found to be coal bridging in the feed funnel; however, the reactor was shut down because it was thought that the feed side of the reactor was plugged.

These seven tests constitute the testing that was conducted with the Mod 1 steady flow reactor. Several general comments can be made about its operation and the need for a Mod 2 steady flow reactor.

1) The 316SS reactor suffered metallurgical damage probably from chloride attack as a result residuals from the zinc chloride hydraulic simulation testing.

2) The Mod 1 reactor and heaters were limited to about 370°C. Even at 350°C to 370°C some heater failures occur. Testing was needed at temperatures up to 390°C.

3) Level control in this reactor design is a difficult task. When coal foaming occurs, the low density foam rapidly rises to the caustic surface and overflows into the receiver carrying caustic with it. When the surface of the caustic drops to the reservoir level, then coal short circuits between the feed leg and the discharge leg. Even when the caustic level is above the reservoir, it is far below the planned depth and the coal residence time is greatly reduced. Small diameter tubes, which hinder caustic disengagement from the foam, are not suitable for use with foaming coals.

3.1.3 Results of the Mod 1 Reactor Tests

Three coals: Illinois #6, Pittsburgh #8 and Upper Freeport (run-of-mine and cleaned) were tested at temperatures from 330°C to 370°C. Coal analyses obtained during each of the test series are given in Table 7. Interpretation of the data is difficult because of coal foaming which influences feed and discharge rates and coal residence time.

As a generalization, the first test data from each run showed lower sulfur and ash levels than later samples. The probable explanation for this result is that the caustic level was at the planned depth at the start of testing and gave more nearly the nominal residence time. Because steady state flow conditions had not been fully established at the time of this first sample, these first results from each run probably are not as good as steady state results. However, as the run proceeded, much of the caustic was carried out in foam sharply reducing the residence time and even permitting coal to short circuit the caustic. Thus, as the run progressed, the results tended to show less reaction.

Considering both the operation of the equipment and the timing of the samples, the following results are considered most typical of the coals tested at the specific test conditions. All tests used an 80/20 NaOH/KOH ratio.

TABLE 7. SUMMARY OF STEADY-FLOW REACTOR RESULTS

Run No.	Coal	Temp, °C	Analysis, moisture free		
			Ash %	S_T, %	Btu/lb
1	16 14x0 moist	370	.54	.39	13340
1B	16 14x0 dry	370	.41	.62	13039
1C	16 14x0 dry	370	1.17	.93	13275
2	UF 14x0 1.6 float	350	1.72	.97	15158
3	UF 14x0 1.6 float	370	1.20	.69	15025
3-2	UF 14x0 1.6 float	370	1.57	.86	14964
3-3	UF 14x0 1.6 float	370	1.15	.65	14796
4-1	UF 14x0 1.6 float	370	1.01	.65	14612
5-1	P8 14x0 repos	370	.48	.55	13684
5-2	P8 14x0 repos	370	.98	1.18	14087
6-1	P8 14x0 repos	330	1.79	2.50	13997
6-2	P8 14x0 repos	330	3.06	2.44	14061
7-1	UF 14x0 ROM	370	.87	2.56	13792
7-2	UF 14x0 ROM	370	1.63	3.38	14281
7-R	UF 14x0 ROM	370	2.51	1.03	14571

Illinois No. 6 - (370°, 1 hr nominal residence time*)

	Analysis (moisture free basis)		
	Ash	S_t	Btu/lb
Starting coal	9.97	4.21	12773
Run 1	.54	.39	13440

Pittsburgh No. 8 - (370° and 330°, 2 hrs nominal residence time**)

	Analysis (moisture free basis)		
	Ash	S_t	Btu/lb
Starting coal	10.34	4.22	13282
Run 5-1 (370°)	.48	.55	13684
GP173 (370°, 80/20, 4 hr)	.51	.55	13962
Run 6-2 (330°)	3.06	2.44	14061
GP165 (330°, 80/20, 1 hr)	2.87	2.30	13935

Upper Freeport - ROM (370°, 1-6 hr residence time***)

	Analysis (moisture free basis)		
	Ash	S_t	Btu/lb
Starting coal	38.00	2.50	8908
Run 7-R	2.51	1.03	14571
GP174 (370°, 80/20, 2 hr)	2.60	1.01	15042

---

*Coal feed problems caused flow interruptions which lengthened the residence time while foaming reduced it. Actual residence time is thought to be more than 2 hours. Extrapolating the laboratory data of Figure 11 indicates and effective residence time of about 4 hours.

**The coal feeder was set to give 2 hr residence time, but for about 2/3 of Run 5-1 the actual weighed coal input was equivalent to about 4 hr residence time. Probably foaming and the low caustic level made Run 6-2 nearer 1 hr residence time.

***Most of the coal collected in the reservoir which was washed and sampled at the end of the run to give sample 7-R. The coal was added over a 6-hr period so that the first coal had about 6 hrs of residence time and the last coal probably had 1 hr or less.

Upper Freeport-Cleaned (350° and 370°, 2 hr nominal residence time*)

	Analysis (moisture free basis)		
	Ash	S_t	Btu/lb
Starting coal	9.53	1.22	14057
Run 2 (350°)	1.72	.97	15158
(No comparative laboratory tests were conducted at 350°C)			
Run 3 (370°)	1.20	.69	15025
Run 3-3 (370°)	1.15	.65	14796
Run 4-1 (370°)	1.01	.65	14612
GP169 (370°, 80/20, 4 hr)	1.44	.90	15052
GP154 (370°, 50/50, 2 hr*)	.95	.62	14938

Coal Recovery

Test series 5 to 7 which were from a continuous operation included sufficient samples to calculate the overall coal recovery. During tests 5 and 6, 24.1 pounds of Pittsburgh #8 coal with 10.34% ash was fed to the reactor. During the tests, four product samples were removed with a total weight of 18.5 pounds. The 5.6 pound difference is about equal to the six pounds calculated to be the reactor holdup, but the actual holdup was not determined. Test 7 followed immediately and input 11.0 pounds of dry, ROM Upper Freeport coal containing 38.0% ash. The two product samples had a total coal product weight of 4.8 pounds. After Test 7, the reactor was drained and 7.4 pounds of coal were recovered from the caustic. The total input and output of solids will not balance because ash (actually mineral matter) was removed by the process. A meaningful approach to obtaining a proper balance is to reduce all inputs and outputs to a moisture and ash free (MAF) basis. This eliminates most of the problems associated with mineral matter dissolving into the leach solution. Table 8 shows the MAF coal balance for Runs 5-7. The overall closure is reasonable showing 28.4 pounds in and 29.3 pounds out.

---

*Using a 50/50 caustic ratio matches the results which laboratory tests (see Figure 5) indicate would need a temperature near 390°C to reach at an 80/20 ratio.

TABLE 8. COAL BALANCE

	Sample	Wt, lb	$\%H_2O$	% Ash	MAF Coal, lb	MAF Btu/lb
Input :	P-8	24.1	nil	10.34	21.6	-
	UF, ROM	11.0	nil	38.00	6.8	
					28.4	
Output:	5-1	3.95	3.74, 3.12	.48, .85	3.78, 3.79	13750, 12669
	5-2	4.30	2.22, 2.90	.99, 1.20	4.16, 4.13	14227, 12824
	6-1	6.44	2.56, 3.85	1.79, 1.49	6.16, 6.10	14251, 13121
	6-2	3.77	1.54, 3.21	3.06, 1.43	3.60, 3.60	14503, 13361
	7-1	3.84	3.33, 3.29	.87, 1.68	3.68, 3.65	13914, 13267
	7-2	.97	2.24, 2.92	1.63, 2.55	.93, .92	14517, 14075
	7R	7.42*	1.97	2.51	7.09	14947
					29.34 ± .06	
	Overall coal recovery				103.3%	

Approximate Pittsburgh 8 recovery

$$\frac{\text{Sum of 5-1 through 7-2}}{\text{Input P-8}} = \frac{22.25}{21.6} \text{ or } 103\%$$

Approximate Upper Freeport recovery

$$\frac{\text{Sum of 7R and wash}}{\text{Input UF, ROM}} = \frac{7.09}{6.8} \text{ or } 104\%$$

*The coal recovered from the equipment after shutdown, Sample 7R, is 7.3 pounds from the caustic and .12 pounds washed from the hardware. Analysis is only for the 7.3 pound portion; the wash part is assumed to be similar.

At the bottom of the table is an estimate of the recovery of each coal. Because the Pittsburgh coal foamed and was readily carried out of the reactor into the quench receivers even when the caustic level was low, it is believed that most of the coal obtained during Test 7 (Samples 7-1 and 7-2) was residual Pittsburgh coal held up in the reactor. The non-foaming Upper Freeport sank into the reservoir and was recovered from the caustic as sample 7R and the small amount of coal washed from the equipment. This explanation is also supported by the heat of combustion (MAF) of coals from 7-1 and 7-2 which are more like Pittsburgh coal (14000 Btu/lb) than Upper Freeport coal (15000 Btu/lb).

3.2 MOD 2 REACTOR

3.2.1 Design

Coal foaming posed a major problem relating to the movement of coal in the 3" diameter feed and discharge tubes of the Mod 1 bench scale reactor. Based on the test experience gained during the Mod 1 reactor operation, a design concept was developed and implemented which greatly enlarged the coal flow path. The Mod 2 reactor made use of an existing electrically heated, nickel vessel 20" in diameter and 28" deep. Into this vessel a baffle arrangement was inserted as shown in Figure 20.

Dry coal was introduced into the central section, equipped with a stirrer to contact the coal with the molten caustic. As the coal became wetted, it passed through a slot in the lower portion of the cylinder amd floated to the surface of the annular reactor zone in the region between the tall divider and the short baffle. A coarse screen over the slotted opening prevented large unwetted coal agglomerates from leaving the mixing zone.

In the annular zone, coal floated to the surface, passed over the short baffle and worked its way around the reactor to the opposite side of the tall divider. Movement was assisted by an occasional use of a screen dasher (Figure 21) that was moved up and down against the surface of the floating coal. At the end of its circuit, coal was periodically removed to maintain a

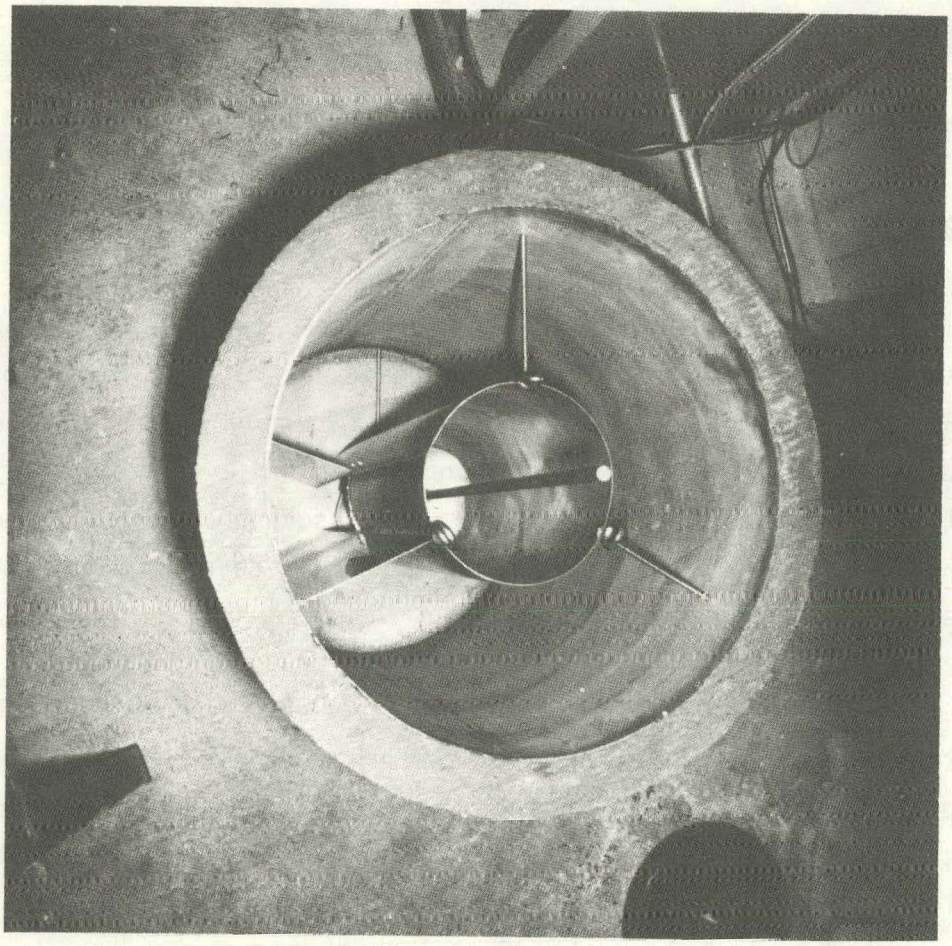


Figure 20. Mod 2 Reactor, Baffle and Stirrer

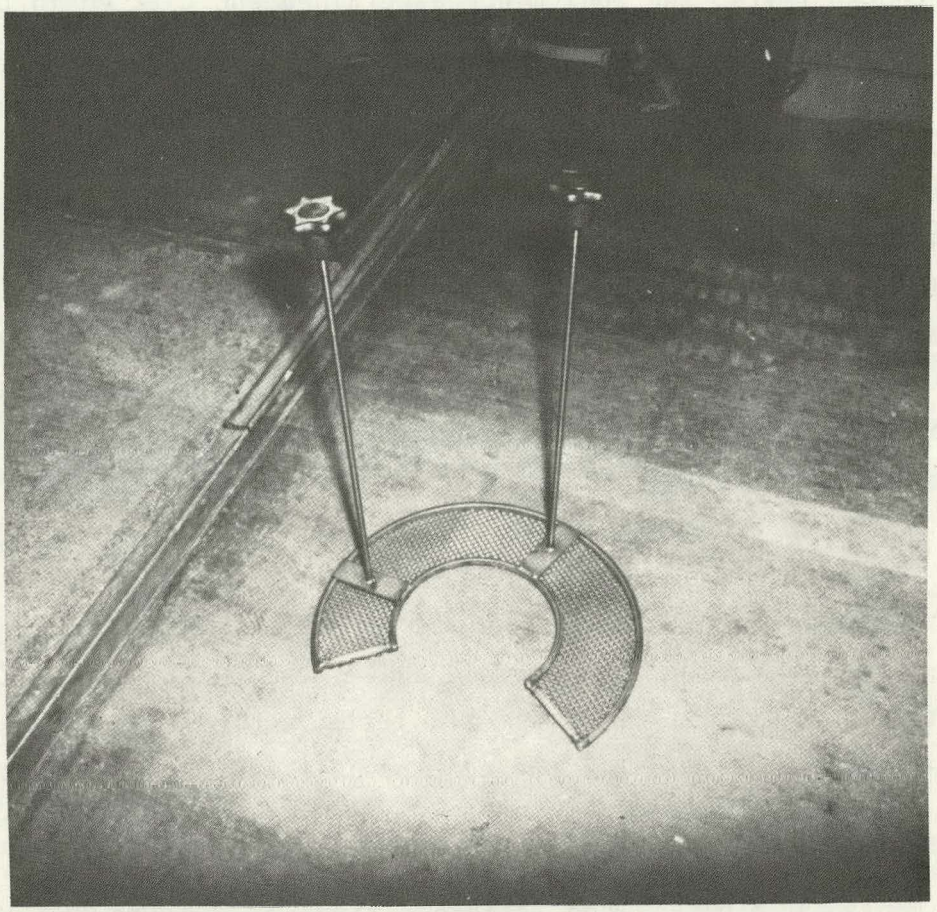


Figure 21. Dasher Used to Assist Coal Movement

relatively constant quantity of coal in the reactor. Figure 22 shows the reactor, its cover and the screen scoop used to removal coal from the annular discharge area.

Shakedown tests showed no major operational difficulties in feeding and removing dry Pittsburgh coal over the range of temperatures and feed rates planned. Moist coal would not properly feed through the automatic feeder system and required hand feeding. The single-shift shakedown runs provided an initial estimate of the caustic/coal ratios removed by the scoop. Following the shakedown tests, the equipment was drained and cleaned in preparation for the week-long, round-the-clock test.

3.2.2 Reactor Operation

The steady flow, Mod 2 reactor was used successfully for a series of nine tests totalling 98 hours during one week of 24 hour per day operation. All tests as shown in Table 9, used the 6 mesh x 0 Pittsburgh #8 repository coal* with a sieve analysis as reported previously. In Test 4, the 100 mesh x 0 fraction (about 28%) was removed and only the +100 fraction was processed. The coal as reported in the repository analysis has 5.9% moisture and on a dry basis has 10.34% ash and 4.22% sulfur.

Coal was fed to the reactor at a rate of 1 lb/hr (4 hours nominal residence time) during Tests 2, 3 and 9 and at 2 lb/hr (2 hour nominal residence time) during the other six tests. In all tests except the moist coal test (Test 6), the coal was continuously fed by a calibrated feeder. It was planned that 4 pounds of coal would be contained in the reactor and that about 1/6 of the coal would be scooped out at each sample time. The sampling interval was 20 minutes when residence time was 2 hours and 40 minutes when residence time was 4 hours.

---

*This coal has generally been referred to as 14 mesh coal. It actually is 6 mesh top size and is referred to as 6 mesh x 0 in these discussions.

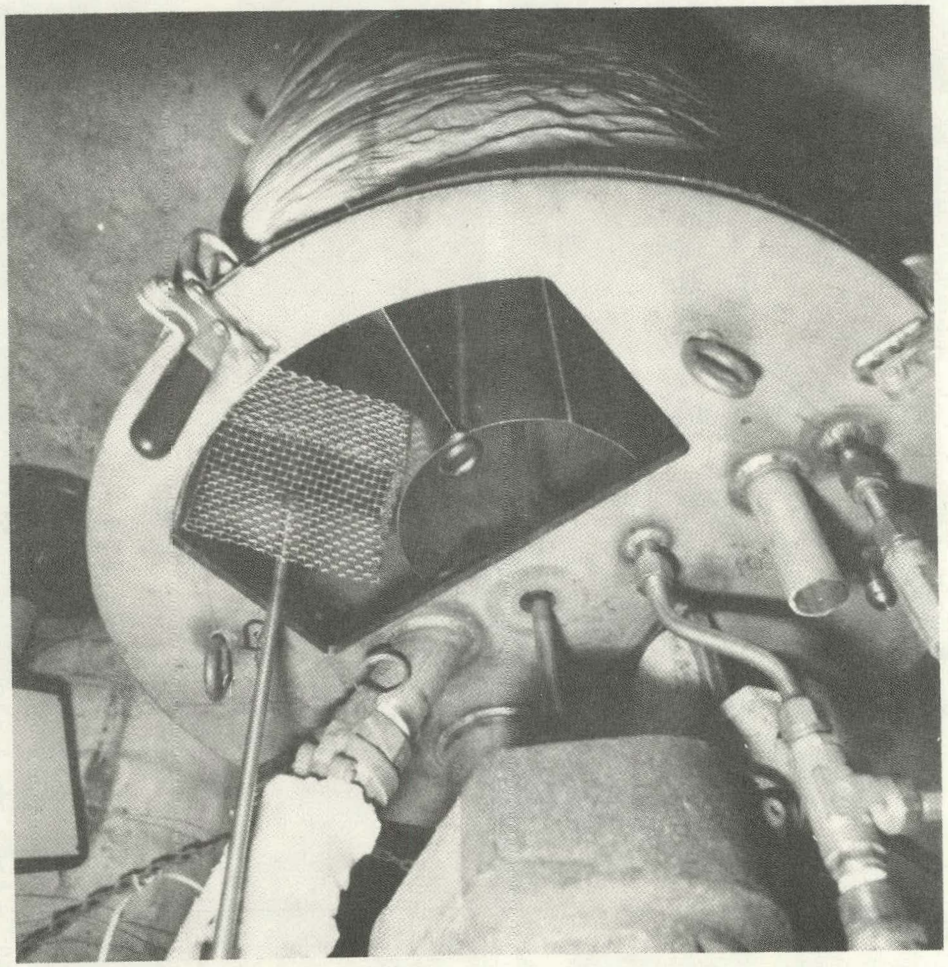


Figure 22. View of Reactor Through the Coal Removal Port

TABLE 9. MOD 2 REACTOR TEST SEQUENCE

Test	Temp °C	NaOH/ KOH	Res. Time hr	Test Duration, hr	Coal Feed	Notes
Start Up	370	80/20	-	2	none	
1	370	80/20	2	12	6 x0, dry	Jan. 17 02:00-14:00; coal fed, no samples first 2 hr
2	370	80/20	4	12	6 x0, dry	Jan. 17 14:00 - Jan. 18, 02:00
Cool down	-	80/20	4	4	6 x0, dry	continued to add/remove coal
3	350	80/20	4	12	6 x0, dry	Jan. 18 06:00-18:00
Heat up	-	80/20	2	4	6 x0, dry	no coal added; sampling continued
4	370	80/20	2	8*	6 x100 dry	Jan. 18 22:00 - Jan. 19 06:00
5	370	80/20	2	8*	6 x0, dry	Jan. 19 06:00-14:00
6	370	80/20	2	8*	6 x0, moist	Jan. 19 14:00-22:00; coal hand fed
Add KOH	-	67/33	2	4		temp dropped to 304°C, 362° at start of #7
7	370	67/33	2	12*	6 x0, dry	Jan. 20 02:00-14:00
Add KOH		50/50	2	4		temp dropped to 297°C, 361° at start of #8
8	370	50/50	2	16*	6 x0, dry	Jan. 20 18:00 - Jan. 21 10:00; feeder off 1 hr
9	370	50/50	4	10	6 x0, dry	Jan. 21 10:00-20:00
Shutdown	-	-	-	4	none	skim total reactor surface

*First 2 hours used to purge the reactor of coal from prior test period.

The overall material recovery is given in Table 10 which shows agreement between the total weight of material removed from the reactor and the sum of caustic and coal added to the reactor. A total of 563 pounds of caustic-wet coal samples were removed from the reactor. The starting and ending caustic levels were within an inch (20 lb/in) so that the overall balance is free of significant error.

The solidified product removed at 20 to 40 minute intervals from the reactor consisted of several lumps of coal mixed with caustic. Every fifth of these products was sampled and the sample, weighing 100 to 200 grams was laboratory washed and dried to obtain a processed coal sample for analysis. The results are given in Table 11 which shows the ash value obtained at the CTS laboratory and the weight yield of dry coal from each sample. Usually the last two samples from each run also were submitted to Warner Laboratories for a short proximate analysis.

The ash and sulfur data obtained from the Mod 2 reactor samples generally show slightly less removal than was obtained in the laboratory batch reactor tests (Section 2.6, Table 5). Sulfur comparisons are more important since ash removal remains well above the 90% target.

Laboratory		Mod 2		
Tests	SO_2 Removal,%	Tests	SO_2 Removal,%	Conditions
GP168,213	83.0,83.4	1,5	79.1,81.3,80.2 83.6	(370°,80/20,2hr)
GP173	90.5	2	87.1,90.4,90.4 87.1,86.9,85.9	(370°,80/20,4hr)
GP190,194	92.7,89.7	8	85.8,84.8	(370°,50/50,2hr)
GP195,197	90.5,92.8	9	85.9,91.8,88.0	(370°,50/50,4hr)

In three of the four conditions, the best Mod 2 sample is about the same as the laboratory sample. This suggests that a sampling error may exist. The laboratory analyses are based on a riffle cut of coal after working up the complete batch. It is more likely that the sampling error is from the grab sample technique used on the Mod 2 product. A total of three samples, all at 4 hour residence time, did meet the NSPS levels.

TABLE 10. MOD 2 REACTOR CAUSTIC AND COAL RECOVERY

Event	Duration, hr	Coal In lb	Caustic In lb	Samples Out lb
Initial fill	-	4.1	-	-
Test 1	12	19.8	29.1	46.6
Test 2	12	12.7	20.4	31.5
Cool down	4	3.5	9.0	13.9
Test 3	12	11.4	26.7	41.1
Heat Up	4	-	8.9	12.0
Purge	2	6.3	7.0	10.7
Test 4	6	8.3	25.8	39.7
Purge	2	6.2	9.6	14.7
Test 5	6	10.8	21.5	33.2
Residual (est)	-	-1.8	-	-
Purge	2	3.5	8.3	12.8
Test 6	6	12.0	26.3	42.9
Add KOH, heat	4	-	60.0	-
Purge	2	7.0	-	14.0
Test 7	10	19.0	29.7	56.8
Add KOH, heat	4	-	90.0	-
Purge	2	2.2	-	19.8
Test 8	14	24.7	26.8	86.2
Test 9	10	6.9	8.3	31.9
Residual (est)	-	-1.4	-	50.2
		155.2	407.4	558.0
12 small bottle samples at about 200g each				5.3
			Total Out	563.3
			Total In	562.6

TABLE 11. RESULTS OF THE MOD 2 TESTING OF PITTSBURGH 8 COAL

Sample	CTS,Ash	% Coal	Ash,%	S_t,%	Btu/lb (MF)
RUN 1 (370°C, 80/20, 2 hr nom., dry 6 x 0)					
1-1	1.35	21.6			
1-6	1.01	23.3			
1-11	1.26	24.9			
1-16	1.28	23.1			
1-21	1.15	24.2	1.11	1.23	14084
1-26	1.12	22.9	1.10	1.10	14100
RUN 2 (370°C, 80/20, 4 hr nom., dry 6 x 0) see note at end of table					
2-1	1.00	23.0			
2-6	.64	22.1	.62	.76	14106
2-7	.94	21.5	1.02	.55	13695
2-8+9	-	-	1.37	.50	12504
2-11	.68	21.0	.53	.76	14087
2-12+13+15	-	-	1.26	.75	13780
2-16	.67	18.0	.67	.83	14110
RUN 3 (350°C, 80/20, 4 hr nom., dry 6 x 0)					
Cool down	.85	19.6			
3-1	.78	19.9			
3-6	.79	19.6			
3-11	1.09	20.6	1.10	1.03	14151
3-16	.96	18.9	.99	.93	14232
RUN 4 (370°C, 80/20, 2 hr nom., dry 6 x 100)					
Heat up	.93	19.8			
Pre 4	1.23	22.6			
4-1	1.12	24.3			
4-6	1.45	22.0			
4-11	1.36	20.5	1.45	1.23	14098
4-16	1.29	21.9	1.34	1.20	14180

TABLE 11. (Cont'd)

Sample	CTS, Ash	% Coal	Ash, %	S_T, %	Btu/lb (MF)
RUN 5 (370°C, 80/20, 2 hr nom., Repeat #1)					
Pre 5	.96	23.3			
5-1	1.13	22.3			
5-6	.78	23.7			
5-11	1.46	23.7	1.34	1.17	14136
5-16	.96	23.1	1.14	.97	14187
RUN 6 (370°C, 80/20, 2 hr nom., moist 6 x 0)					
Pre 6	1.21	22.5			
Pre 6-1	1.00	22.1			
6-1	.89	21.2			
6-6	1.17	18.6			
6-11	1.09	19.6	1.13	1.17	14177
6-16	.91	21.3	.83	1.04	14103
RUN 7 (370°C, 67/33, 2 hr nom., dry 6 x 0)					
Pre 7	.59	18.6			
7-1	1.21	20.1			
7-6	1.27	20.1			
7-11	1.13	19.9			
7-16	.89	17.7			
7-21	1.38	18.4	1.17	1.16	14243
7-26	1.34	19.6	1.21	1.14	14160
RUN 8 (370°C, 50/50, 2 hr nom., dry 6 x 0)					
Pre 8	.45	18.6			
Pre 8-1	.52	17.8			
8-1	.58	14.4			
8-6	.79	15.2			
8-11	1.06	15.7			
8-16	.81	16.9			

TABLE 11. (Cont'd)

Sample	CTS,Ash	% Coal	Ash,%	S_T,%	Btu/lb (MF)
RUN 8 (Cont'd)					
8-21	.79	16.8			
8-26	.82	11.7			
8-31	.64	15.2			
8-36	.86	14.3	.78	.82	13889
8-41	.94	14.5	1.14	.88	13881
RUN 9 (370°C, 50/50, 4 hr nom., dry 6 x 0)					
9-1	1.49	16.0			
9-6	1.14	18.0			
9-7	.20	16.9			
9-11	.73	16.6	.76	.81	13766
9-13	.84	14.2	.91	.44	12909
9-14	.69	13.2	.90	.67	13335

Notes on Run 2

Samples from this run were sent to Ames Laboratories for detailed study as a part of their on going coal research. Sample 2-7 was sent, as removed from the reactor, mixed with frozen caustic. A small sample of the material was processed by the usual washing method at CTS to provide the sample for analysis as shown. Sample 2-8+9 was prepared by water washing 2-8 and 2-9 and blending the product. A sample of the blend was acid washed by the usual method at CTS to provide the sample for analysis. Sample 2-12+13+15 is the complete washing of these three samples by the usual CTS laboratory procedure. The washed material was blended and sampled for analysis.

In order to gain additional insight into the location of the ash, three samples from Run 2 were further treated. Sample 2-11 which had 0.68% ash (CTS) or 0.53% ash (Warner) was sieved into 3 size-fractions and a CTS ash obtained on each fraction as follows:

Size, mesh	wt, %	Ash, %
+ 12	5	1.34
12 x 100	51	.47
100 x 0	44	.33
	100	.45

Sample 2-16 which had .67% ash (CTS and Warner) was also sieved into a coarse and a fine fraction:

Size, mesh	wt, %	Ash, %
+ 100	62	.52
100 x 0	38	.27
	100	.42

Since both sieved samples gave ash values after sieving that were substantially below the starting value, sample 2-6 also was ground to 100 mesh top-size and analyzed without separation. This reduced the measured ash from 0.64% CTS (0.62% Warner) to 0.32%. These results show that sieving reduces the ash, as analyzed, from .68% to .45% and .67% to .42% and that grinding reduces it from .64% to .32%. These differences exceed the usual reproducibility of the ASTM ash analysis. Therefore, a more definitive investigation of ash determination for low ash processed coal is needed*.

Material from Run 1 and Run 7 were used to check out the wash tanks and centrituge. The throughput of the wash system is so high that each run produces slurry for only about 5 minutes of centrifuge operation. Useful

---

*Subsequent to the completion of this contract study, TRW discovered that the ASTM ash method yields incorrect values when bound or free alkali is present with the coal. The error can be removed by increasing the ashing temperature. The excess ash appears to be related by incomplete carbon combustion in the presence of alkali. It has not been checked, but it is possible that fine particles may reduce the amount of unburned carbon.

mass balances could not be obtained because end effects, such as feed line equipment holdup, were too great a fraction of the total. For future testing of the wash system, it is recommended that about 100 pounds of more of processed coal can be prepared at a single processing condition to provide the large quantity of uniform material needed to verify the operation of the washing and centrifuge operation.

APPENDIX A

LABORATORY EXPERIMETAL CONDITIONS AND RESULTS

APPENDIX A

This section contains a listing of experimental conditions and analysis results for tests conducted as a part of the laboratory task. The tests are listed in sequence of their laboratory notebook test designation. Tests include BSS 1 to 16, GM 190 to 202 and GP 135 to 240. Within each series the tests were performed chronologically. Test numbers lower than those listed were performed prior to the initiation of this project and missing numbers are laboratory tests for other programs running concurrently with this effort.

The following is a description of the meaning of the column headings:

Coal- - P8 rep, K11 rep and I6 rep are dried samples of the 3 repository coals with properties given in greater detail in Appendix B. K11 lab is the 45 x 200 mesh coal used in the previous laboratory program.

- UF ROM and UF cln are run-of-mine and mine cleaned samples of Upper Freeport coal provide as a drummed sample by the DOE.

- P8 bbl and P8 bag are other samples of Pittsburgh #8 coal originally received in barrels and bags respectively.

Size - The approximate mesh size of the unprocessed coal is given. Each of the repository coals is nominally 14 mesh but sieve analysis shows it is about 6 mesh x 0 in size. In a few tests, the "g" following 45 x 200 indicates that the large particle size coal (6 x 0 or 14 x 0) was ground to pass the top size (45 mesh) and the fines were removed by sieving to give the 45 x 200 mesh size. In the others the 45 x 200 is a sieve cut of the coarse coal.

Caustic - The weight ratio of NaOH to KOH is given along with a designation of the source of caustic. L and C represent laboratory (CP or AR) grade and commercial grade received in drums. Both have very similar purity. NaOH is about 99% to 100% caustic with a few-tenths CO_3 and moisture. KOH is about 88% caustic with about 12% moisture. "CU" is the commercial caustic first used in the large reactor to process multiple batches of coal, then sampled for use in lab testing of the effect of impurities.

Temp - This represents the controller temperature (°C) at which the test was conducted.

Time - The residence time in hours starting with the introduction of coal into the preheated reactor containing the molten caustic and ending when the floating coal is skimmed from the surface or the reactor is rapidly cooled to freeze the caustic.

Wash - The skimmed coal or the top layer of the solidified caustic is digested in water to dissolve the caustic. The coal is filtered and washed again with water and dried to yield a water washed product "W". Acid washed coal involves treating the water washed, wet product with 10% sulfuric acid, filtering, followed by two additional water washes and drying. This is designated "A". The "2A" sample had the acid washing steps repeated. The "HCl" results are from substituting hydrochloric acid for sulfuric acid. In a few instances the moist filter cake was split to permit two difference treatments.

H_2O, Ash, S, Btu - Analyses of the processed coal sample obtained from Warner Laboratories, Inc., Cresson, Pennsylvania. The as-received moisture of the sample is in weight percent. The ash (wt %) total sulfur (wt %) and heat of combustion (Btu/lb) are calculated on a dry, moisture-free (MF) basis.

MAF - The heat of combustion is also reported by Warner, calculated on a moisture and ash free basis to correct for the influence of ash dilution.

CTS Ash - Many of the coals had small samples removed, dried and ashed in the test lab to get rapid evaluation of the process conditions. These results are generally from riffled samples and performed much like the ASTM procedure and can be used to cross-check the Warner analyses.

Notes - Represent numbers of the footnotes at the end of the final table.

TABLE A-1. LABORATORY RESULTS

No.	Coal	Size	Caustic	Temp	Time	Wash	H_2O	Ash	S	Btu/lb	MAF	CTS-Ash	Notes
BSS Series													
1	P8 bbl	14 x 0	50/50 L	370	.33	A	1.12	2.19	1.31	14435	14760		
2	P8 bbl	14 x 0	50/50 C	370	.33	A	1.38	3.07	1.48	14298	14751		
3	P8 bbl	14 x 0	50/50 L	370	1	A	2.17	1.08	.67	14145	14300		
4	P8 bbl	14 x 0	50/50 C	370	1	A	1.68	1.32	.84	14207	14397		
5	P8 bbl	14 x 0	50/50 L	350	.33	A	1.15	2.81	1.52	14490	14909		
6	P8 bbl	14 x 0	50/50 C	350	1	A	2.05	1.23	.85	14244	14422		
7	P8 bbl	14 x 0	50/50 C	350	.33	A	1.36	2.16	1.34	14387	14706		
8	P8 bbl	14 x 0	50/50 L	350	1	A	1.25	1.93	1.19	14310	14591		
9	P8 bbl	14 x 0	80/20 C	350	1	A	.96	3.03	1.47	14397	14847		
10	P8 bbl	14 x 0	50/50 L	325	.33	A	1.02	3.46	1.66	14434	14951		
11	P8 bbl	14 x 0	50/50 C	325	.33	A	1.05	3.31	1.76	14472	14966		
12	P8 bbl	14 x 0	50/50 L	325	1	A	1.06	2.41	1.41	14378	14733		
13	P8 bbl	14 x 0	50/50 C	325	1	A	.77	2.65	1.49	14532	14928		
14	P8 bbl	14 x 0	80/20 C	325	1	A	1.12	1.49	1.20	14559	14778		
15	UF ROM	14 x 0	50/50 L	370	.33	A	.97	2.70	.98	15015	15432		
16	UF ROM	14 x 0	50/50 C	370	.33	A	.92	2.78	.91	15034	15463		

TABLE A-1 (Cont'd)

No.	Coal	Size	Caustic	Temp	Time	Wash	H_2O	Ash	S	Btu/lb	MAF	CTS-Ash	Notes
GM Series													
190	P8 bag	50 x 0	80/20 C	305	2	A	.90	3.00	1.45	14551	15001	3.07, 2.40	
191	P8 bag	50 x 0	80/20 L	330	2	A	1.10	2.36	1.29	14822	15181	2.35, 2.47	
192	P8 bag	50 x 0	80/20 CU	330	2	A	.98	2.08	1.14	14652	15964	1.48, 1.52	
193	P8 bag	50 x 0	80/20 CU	370	.5	A	1.30	2.76	1.30	14647	15062	2.12, 2.09	
194	16 rep	6 x 0	80/20 CU	330	2	A	1.74	.97	.93	14440	14582	1.25, 1.12	
195	P8 bag	45 x 200	80/20 CU	330	2	A	1.11	1.97	1.32	14822	15119	1.78, 1.85	
196	K11 lab	45 x 200	50/50 L	370	.5	A	3.49	.21	.61	13573	13602	.22, .22	
197	K11 rep	45 x 0	80/20 L	370	.5	A	4.50	.77	1.51	14214	14324	.39, .39	
198	K11 rep	45 x 0	80/20 CU	350	2	A	2.09	.42	.76	14166	14225	.26, .23	
199	K11 rep	6 x 100	80/20 CU	350	2	A	1.66	.65	1.04	14148	14241	.39, .34	
200	K11 rep	45 x 0	80/20 L	370	.5	A	1.42	.58	1.22	14261	14344	.14, .20	
200A	K11 rep	45 x 0	80/20 L	370	.5	A	2.41	.60	1.14	13864	13947		
201	K11 rep	6 x 100	80/20 CU	350	3.5	A	2.56	.33	.52	13906	13952	.12, .12	
202	K11 rep	6 x 100	80/20 CU	350	7.2	A	N/A	.44	.51	13779	13840	.11, .08	

TABLE A-1 (Cont'd)

No.	Coal	Size	Caustic	Temp	Time	Wash	H_2O	Ash	S	Btu/lb	MAF	CTS-Ash	Notes
GP Series													
135	UF cln	100 x 0	80/20 L	370	.33	A	.79	4.23	1.08	14791	14544	4.6	
136	UF cln	100 x 0	80/20 L	370	.33	A	.67	6.21	1.11	14490	15450	6.9	
141	UF cln	100 x 0	50/50 L	370 (split)	1	A	1.49	2.81	.97	14781	15208		
						HCl	1.60	2.20	.68	15103	15443		
142	UF cln	100 x 0	50/50 L	370 (split)	1	A	1.60	2.38	.88	14378	14729		
						HCl	1.51	2.63	.79	14915	15319		
151	UF cln	100 x 0	50/50 L	390	1	A	2.11	.71	.54	14950	15058		
151A	UF cln	100 x 0	50/50 L	390	1	A	3.19	.25	.33	14475	14511		
152	UF cln	100 x 0	50/50 L	370	2	A	1.97	.87	.74	15105	15237		
152A	UF cln	100 x 0	50/50 L	370	2	A	2.76	.72	.64	15054	15164		
153	UF cln	100 x 0	KOH L	390	2	A	3.89	.47	.38	13873	13939		
154	UF cln	14 x 0	50/50 L	370	2	A	1.87	.95	.62	14938	15081	.92	
155	UF cln	14 x 0	50/50 L	390	1	A	1.97	.99	.53	14884	15034	.92	
158	UF cln	14 x 0	50/50 L	390	2	A	2.18	.35	.28	14780	14832	.24	
161	16 rep	45 x 200	50/50 L	370	2x.5	W	Not analyzed					10.7	
162	P8 rep	45 x 200	50/50 L	370	2x.5	W	Not analyzed					10.6	
163	16 rep	45 x 200	50/50 L	370	2x.5	Not well separated							
164	16 rep	45 x 200	50/50 L	370	2x1	Not well separated							
165	P8 rep	6 x 0	80/20 C	330	1	A	1.50	2.87	2.30	13935	14347	3.08	
166	P8 rep	6 x 0	80/20 C	330	2	A	1.59	2.52	2.02	14255	14624	2.27	
167	P8 rep	6 x 0	80/20 C	370	1	A	1.84	1.86	1.59	13944	14208	1.67	
168	P8 rep	6 x 0	80/20 C	370	2	A	2.42	1.727	.99	13927	14171	.73, .93	
169	UF cln	14 x 0	80/20 C	370	4	A	1.61	1.44	.90	15052	15272	1.32	
170	UF cln	14 x 0	80/20 C	370	2	A	1.46	2.52	1.04	15106	15497	2.05	

TABLE A-1 (Cont'd)

No.	Coal	Size	Caustic	Temp	Time	Wash	H_2O	Ash	S	Btu/lb	MAF	CTS-Ash	Notes
GP Series (cont'd)													
171	P8 rep	6 x 14	80/20 C	370	1	A	1.85	3.59	2.30	14142	14668		
						2A	1.82	3.21	2.25	13836	14294	3.12	
172	P8 rep	14 x 24	80/20 C	370	1	A	2.14	2.49	2.22	14172	14534	2.09	
173	P8 rep	6 x 0	80/20 C	370	4	A	2.88	.51	.55	13962	14034	.50	
174	UF ROM	14 x 0	80/20 C	370	2	A	1.22	2.60	1.01	15042	15444	2.54	
175	P8 rep	24 x 100	80/20 C	370	1	A	3.68	.98	1.42	13335	13467	1.19	
176	UF cln	14 x 0	50/50 C	370	4	A	1.99	.53	.47	14833	14912	.50	
177	P8 rep	100 x 0	80/20 C	370	1	A	3.23	1.33	1.74	13437	13619	1.46	
178	UF ROM	14 x 0	80/20 C	370	4	A	1.89	2.75	1.06	14982	15405	2.29	
179	P8 rep	45 x 200	50/50 L	370	1	A	4.48	.27	.56	13762	13799	.07	
180	P8 rep	45 x 200	80/20 L	370	1	A	3.19	.66	.79	14220	14314	.44	
181	UF cln	14 x 0	80/20 L	370	1	W	4.13	4.47	.16	13768	14412	4.85	(4)
						A	3.99	.51	.25	14079	14150	0.42	(5)
182	16 rep	6 x 0	80/20 L	370	1	W	6.79	12.59	1.13	12032	13764	10.66	(4)
						A	3.75	1.47	1.08	13092	13287	1.33	(5)
183	K11 rep	6 x 0	80/20 L	370	1	W	8.94	10.42	.53	12174	13589	9.44	(4)
						A	5.01	.39	.66	13235	13287	.42	(5)
185	P8 rep	6 x 14	80/20 C	370	1	A	2.76	3.06	1.58	13425	13849	2.20	
186	P8 rep	14 x 24	80/20 C	370	1	A	1.89	2.19	1.95	14083	14398	2.10	
187	16 rep	6 x 0	50/50 C	350	2	A	2.07	3.00	2.03	13455	13871	2.66	
188	16 rep	6 x 0	50/50 C	370	2	A	5.46	.38	.34	12412	12460	.30	
189	16 rep	6 x 0	50/50 C	350	4	A	3.33	.34	.57	13378	13423	.34	

TABLE A-1 (Cont'd)

No.	Coal	Size	Caustic	Temp	Time	Wash	H_2O	Ash	S	Btu/lb	MAF	CTS-Ash	Notes
GP Series (cont'd)													
190	P8 rep	6 x 0	50/50 C	370	2	A	3.50	.64	.42	13727	13815	.15	
191	UF cln	14 x 0	50/50 C	370	6	A	2.13	.74	.28	14778	14888	.30	
192	UF cln	14 x 0	50/50 C	390	2	A	2.12	.86	.39	14980	15009	.78	
193	UF cln	14 x 0	50/50 C	390	4	A	2.23	.30	.24	14504	14547	.19	
194	P8 rep	6 x 0	50/50 C	370	2	A	3.39	1.43	.58	13441	13636	.40	
195	P8 rep	6 x 0	50/50 C	370	4	A	4.75	.49	.54	13605	13672	.44	
196	P8 rep	6 x 0	50/50 C	390	2	A	4.97	.46	.48	13146	13207	.32	
197	P8 rep	6 x 200	50/50 C	370	4	A	4.23	.007	.41	13583	13583	.04	
198	P8 rep	45 x 200	50/50 C	370	1	A	3.22	.56	.80	13812	13889	.18	
199	P8 rep	45 x 200	50/50 C	370	1	A	4.13	.06	.54	13694	13702	.03	
200	P8 rep	45 x 200g	50/50 C	370	1	A	3.32	.05	.52	13760	13767	.07	
201	P8 rep	45 x 200g	50/50 C	370	1	A	3.67	.15	.48	13868	13888	.16	
202	P8 bbl	45 x 200	50/50 C	370	1	A	3.55	.36	.50	13941	13991	.18	
203	P8 bbl	45 x 200	50/50 C	370	1	A	2.53	.29	.50	14557	14599	.14	
204	P8 bag	45 x 200	50/50 C	370	1	A	2.65	.54	.42	13788	13863	.21	
205	P8 bag	45 x 200	50/50 C	370	1	A	1.80	1.99	1.00	14334	14624	2.00	
206	P8 lab	45 x 200	50/50 C	370	1	A	3.38	.39	.34	13727	13781	.15	
207	P8 lab	45 x 200	50/50 C	370	1	A	3.67	.21	.33	13724	13752	.17	
208	P8 rep	45 x 200g	50/50 C	340	1	A	2.11	.34	1.13	14405	14454	.28	
209	P8 rep	45 x 200	50/50 C	340	1	A	2.21	.34	1.05	14279	14327	.18	
210	P8 rep	45 x 200	50/50 C	340	1	A	2.39	.47	1.27	14333	14401	.32	

TABLE A-1 (Cont'd)

No.	Coal	Size	Caustic	Temp	Time	Wash	H_2O	Ash	S	Btu/lb	MAF	CTS-Ash	Notes
GP series (cont'd)													
211	P8 bag	45 x 200	50/50 C	340	1	A	1.58	1.69	1.18	14734	14988	1.19	
212	P8 bbl	45 x 200	50/50 C	340	1	A	2.28	.73	1.08	14569	14677	1.00	
213	P8 rep	6 x 0	80/20 C	370	2	A	2.93	1.21	.98	14127	14300	.92	
214	P8 rep	6 x 0	80/20 C	390	2	A	4.03	.14	.31	13851	13871	.13	
215	16 rep	6 x 0	80/20 C	350	2	A	2.68	.88	1.43	13739	13861	1.04	
216	16 rep	6 x 0	80/20 C	370	2	A	4.29	1.11	.79	13630	13784	.79	
217	UF cln	14 x 0	80/20 C	370	2	A	1.20	2.00	.90	15116	15424	1.55	
218	UF cln	14 x 0	80/20 C	390	2	A	1.42	1.70	.77	15064	15324	1.30	
219	KII lab	45 x 200	80/20 C	350	2	A	2.97	.33	.84	14028	14075	.17	
220	KII lab	45 x 200	80/20 C	370	2	A	2.95	.21	.37	13632	13661	.06	(1)
221	KII lab	45 x 200	50/50 C	370	2	A	3.05	.41	.51	13938	13885	.18	
222	KII lab	45 x 200	50/50 C	370	1	A	4.29	.22	.56	13461	13491	.05	
223	KII lab	6 x 0	50/50 C	370	1	A	3.52	1.14	1.26	13724	13883	1.17	
224	KII lab	6 x 0	50/50 C	370	.5	A	3.91	.93	1.31	13714	13843	.48	
225	P8 bag	45 x 200	50/50 C	370	1	A	1.16	.48	.45	14293	14362	.16	
226	16 rep	6 x 0	80/20 C	370	.5	A	1.43	2.61	2.06	13555	13918	1.70	
SF	UF cln	12 x 0	50/50 C	370	1	W	1.31	4.14	.43	14698	15332		
227	UF cln	12 x 0	80/20 C	370	.4	W	.77	4.40	.76	14941	15629		
228	16 rep	6 x 0	80/20 C	370	1	A	1.78	1.86	1.71	13543	13800	1.69	
229	16 rep	6 x 0	80/20 C	370	2	A	1.55	.92	1.08	13757	13885	.49	

TABLE A-1 (Cont'd)

No.	Coal	Size	Caustic	Temp	Time	Wash	H_2O	Ash	S	Btu/lb	MAF	CTS-Ash	Notes
GP Series (cont'd)													
230	UF cln	14 x D	50/50 C	370	4	A	.67	.73	.53	14924	15034		(2)
231	UF cln	14 x [illegible]	50/50 C	370	4	A	.87	.58	.46	14729	14815		(3)
233	KII rep	14 x [illegible]	50/50 C	370	1	A	3.26	.82	1.11	13483	13593	.64	
234	KII rep	45 x 200	50/50 C	370	.5	A	4.37	.28	.58	13451	13489	.17	
235	UF ROM	14 x 0	50/50 C	370	1	A	1.04	2.24	.75	15021	15364	2.54	
236	UF cln	12 x 0	80/20 C	390	2	A	1.33	.79	.63	15073	15193	.75	
237	UF cln	14 x 200	80/20 C	390	2	A	1.11	1.22	.68	15059	15234	.54	
238	P8 rep	6 x 0	50/50 C	370	1	W	5.55	11.61	.79	12577	14160	8.57	
240	P8 rep	6 x 0	50/50 C	370	1	W	5.24	10.62	.77	12703	14212		

Notes

(1) Ash analysis by Warner 18.97% Na_2O, 8.92% K_2O

(2) Like GP176, but stirred 1/2 speed (225 rpm)

(3) Like GP 176, but stirred 1 min, unstirred 15 min, cycles

(4) Poured from reactor and filtered hot, water washed, dried and riffled into samples

(5) Sample of riffled, water washed dry coal was acid washed, dried and riffled for analysis

APPENDIX B

ANALYSES OF UNPROCESSED COAL

APPENDIX B

ANALYSIS OF UNPROCESSED COALS

Table B-1 summarizes the available analytical data for the four coals studied as a part of the program. The Upper Freeport coal was examined both in the high ash ROM form and as a mine cleaned 10% ash coal.

Analyses were performed by Commercial Testing and Engineering Company (CTE) for the three repository coals for which more complete analyses are given in the final 24 pages of this appendix. Most of the other analyses were performed at Warner Laboratories, Inc. Two barrels of coal were analyzed by Ames Laboratory, Iowa State University prior to shipment to TRW and were later sampled and analyzed by Warner.

Standard units for the analyses are weight percent. Oxygen is obtained as the difference of the sum of C, H, N, Cl, S and Ash from 100% and thereby includes losses and errors as well as oxygen.

TABLE B-1. SUMMARY CF UNPROCESSED COAL ANALYSES

	H_2O %	Ash %(MF)	ST %(MF)	Btu/lb (MF)	Btu/lb (MAF)	Sulfur Forms (MF) S_p,%	S_S,%	S_O,%	Composition (MF) C,%	H,%	N,%	Ci,%	O,%
Pittsburgh 8 Repos (CTE) #1	5.87	10.34	4.23	13265	14795	2.17	.03	2.03	72.91	5.29	1.31	.04	5.88
#2	5.91	10.34	4.20	13298	14832	2.16	.04	2.00	73.06	5.25	1.37	.04	5.74
Pittsburgh 8 - bag (Warner)	1.63	7.69	2.53	14062	15233	-	-	-	-	-	-	-	-
Pittsburgh 8 - bbl (Ames)	1.12	9.02	3.51	13660	15014	2.07	.01	1.43	76.62	5.21	1.43	-	4.21
(Warner)	2.64	10.68	3.12	12907	14450	1.67	.03	1.42	-	-	-	-	-
Kentucky 11 Repos (CTE) #1	9.52	10.72	3.30	12985	14544	1.53	.06	1.71	71.71	5.00	1.48	.00	7.79
#2	9.57	10.76	3.37	12996	14563	1.53	.07	1.77	71.92	4.94	1.45	.00	7.56
(Warner)	3.33	10.38	3.38	-	-	-	-	-	71.71	5.14	1.53	-	7.86
Kentucky 11 Lab (Warner)	8.28	6.68	3.43	-	-	-	-	-	73.21	5.46	1.50	-	9.72
(previous project)	-	7.26	3.51	13132	14314	1.59	.01	1.91	-	-	1.54	-	-
Kentucky 11 45x0 (Warner)	3.76	8.39	3.07	13159	14365	-	-	-	-	-	-	-	-
Illinois 6 Repos (CTE) #1	15.99	10.03	4.18	12790	14216	1.35	.04	2.79	70.36	5.19	1.35	.07	8.82
#2	15.63	9.91	4.23	12755	14158	1.36	.05	2.82	70.50	5.27	1.37	.08	8.64
(Warner)	12.62	10.15	4.08	-	-	-	-	-	67.74	4.95	1.54	-	9.54
Illinois 6 Lab (Warner) #1	3.42	9.23	4.03	12841	14147	-	-	-	70.41	5.24	1.34	-	9.76
(Warner) #2	4.22	9.82	4.05	12461	13817	-	-	-	-	-	-	-	-
Upper Freeport ROM (Ames)	.74	36.55	2.41	9534	15026	1.89	.03	.49	55.06	3.17	.96	-	1.85
(Warner) #1	1.52	37.48	2.48	9169	14667	1.98	.03	.47	-	-	-	-	-
(Warner) #2	.77	38.03	2.49	8908	14374	1.93	.02	.55	-	-	-	-	-
Upper Freeport - Clean (Warner) #1	1.20	9.90	1.25	13923	15453	.93	.02	.30	-	-	-	-	-
#2	1.13	9.53	1.23	14057	15537	.50	.01	.72	-	-	-	-	-

ANALYSES OF THE REPOSITORY COALS TAKEN FROM

Final Report

No. 1984-DYO-061A

TRW COAL REPOSITORY PROGRAM

May 13, 1981

Prepared by

Commercial Testing & Engineering Company

Cleveland, Ohio

TRW COAL REPOSITORY PROGRAM
No. 1984-DYO-061A

OHIO
Pittsburgh #8
North American Coal Company
Powhatan #6 Mine
Master Lab No: 9117512

14 Mesh DISTRIBUTION
Sample C
Lab No: 8110891

	Run#1	Run#2	Average
PROXIMATE ANALYSIS			
Moisture:	5.87	5.91	5.89
Dry Ash:	10.34	10.34	10.34
Dry Volatile:	41.47	41.76	41.62
Dry Fixed Carbon:	48.19	47.90	48.05
SULFUR ANALYSIS			
Dry Pyritic Sulfur:	2.17	2.16	2.17
Dry Sulfate Sulfur:	0.03	0.04	0.04
Dry Total Sulfur:	4.23	4.20	4.22
HEATING VALUE ANALYSIS			
Dry Btu Value:	13265	13298	13282
MAF Btu Value:	14795	14832	14813
ULTIMATE ANALYSIS			
Dry Carbon:	72.91	73.06	72.99
Dry Hydrogen:	5.29	5.25	5.27
Dry Nitrogen:	1.31	1.37	1.34
Dry Chlorine:	0.04	0.04	0.04
Dry Ash:	10.34	10.34	10.34
Dry Sulfur:	4.23	4.20	4.22
Dry Oxygen:	5.88	5.74	5.81
FREE SWELLING INDEX	7.5	7.5	7.5
ASH FUSION TEMPERATURES			
Initial:	2145	2135	2140
Softening (H=W):	2240	2255	2248
Hemispherical (H=W/2):	2335	2330	2333
Fluid:	2365	2375	2380

TRW COAL REPOSITORY PROGRAM
No. 1984-DYO-061A

OHIO
Pittsburgh #8
North American Coal Company
Powhatan #6 Mine
Master Lab No: 9117512

SET B
14 Mesh DISTRIBUTION
Sample C
Lab No: 8110891

	Run#1	Run#2	Average
MAJOR ELEMENTS ANALYSIS			
Silica:	40.86	39.84	40.35
Alumina:	20.25	19.85	20.05
Titania:	0.89	0.87	0.88
Ferric Oxide:	29.28	29.23	29.26
Calcium Oxide:	2.51	2.49	2.50
Magnesium Oxide:	0.86	0.77	0.82
Potassium Oxide:	1.39	1.43	1.41
Sodium Oxide:	0.73	0.72	0.73
Sulfur Trioxide:	2.11	2.16	2.14
Phos. Trioxide:	0.18	0.22	0.20
Strontium Oxide:	0.04	0.04	0.04
Barium Oxide	0.03	0.05	0.04
Manganese Oxide:	0.06	0.05	0.06
Undetermined:	0.81	2.28	1.52
TRACE ELEMENTAL ANALYSIS*			
Lead:	≤2	≤2	≤2
Mercury	0.15	0.15	0.15
Cadmium:	≤0.1	≤0.1	≤0.1
Arsenic:	5.4	5.5	5.5
Selenium:	2.2	1.7	2.0
Antimony:	≤0.3	≤0.3	≤0.3
Beryllium:	0.8	0.7	0.8
Chromium:	16	18	17
Copper:	8	7	8
Manganese:	24	24	24
Nickel:	8	9	9
Zinc:	22	22	22
Vanadium:	26	27	27
Boron:	100	100	100
Chlorine:	610	560	585

* Parts per million

TRW COAL REPOSITORY PROGRAM
No. 1984-DYO-061A

OHIO
Pittsburgh #8
North American Coal Company
Powhatan #6 Mine
Master Lab No: 9117512

SET B
14 Mesh DISTRIBUTION
Sample C
Lab No: 8110891

GEISELER PLASTOMETER

Maximum DDPM:	29954
Temperature °C at Maximum DDPM:	419

PARTICLE SIZE ANALYSIS

Passing	Retained On	% Weight
	14M	22.9
14	28	26.6
28	48	17.8
48	100	10.4
100	150	3.9
150	200	2.9
200	250	1.1
250	325	2.3
325M		12.1
		100.00%

TRW COAL REPOSITORY PROGRAM
No. 1984-DYO-061A

OHIO
Pittsburgh #8
North American Coal Company
Powhatan #6
Master Lab No: 9117512

SET B
14 Mesh DISTRIBUTION
Sample C
Lab No: 8110891

PETROGRAPHIC ANALYSIS
Reflectance Analysis
Maceral Group Analysis
Microlithotype Analysis

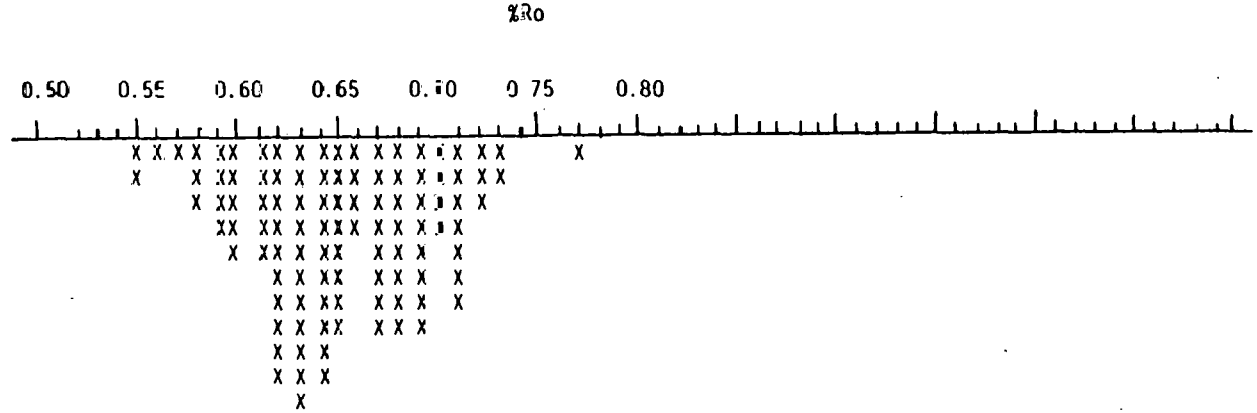
PYRITE SIZE DISTRIBUTION

Report of Analysis on Sample: 81-10891

REFLECTANCE ANALYSIS

Mean-Maximum Vitrinite Ro : 0.65

Distribution of Vitrinite Reflectance Readings:



V-Type Table for Vitrinites (=100%)

V- 5	V- 6	V- 7	V-
10.9	73.3	16.2	

V-Type Table for Vitrinites (= 73.2 %)

(Adjusted to = Maceral % of Reactive Vitrinites)

V- 5	V-6	V- 7	V-
7.6	53.7	11.9	

REPORT OF ANALYSIS ON SAMPLE: 81-10891

3/26/81

MACERAL ANALYSIS
(VOLUME PERCENT)
(MINERAL-MATTER CONTAINING BASIS)

MACERAL		MACERAL GROUP	
VITRINITE	73.2	VITRINITE	73.2
PSEUDOVITRINITE	0.0		
EXINITE	4.2	EXINITE (LIPTINITE)	4.2
RESINITE	0.0		
SEMI-FUSINITE*	6.5		
SEMI-MACRINITE*	0.0		
FUSINITE	3.0	INERTINITE	15.7
MACRINITE	0.1		
MICRINITE	6.1		
MINERAL MATTER**	6.9		6.9
TOTAL	100%		100%

TOTAL REACTIVES- 79.6
TOTAL INERTS- 20.4

++++++++++++

* CONSIDERED 1/3 REACTIVE, 2/3 INERT FOR PURPOSES OF COKE STABILITY PREDICTIONS.

** CALCULATED FROM 10.34 % DRYASH, 4.22 % DRY SULFUR

Report of Analysis on Sample: 81-10891

MICROLITHOTYPE ANALYSIS
(Volume Percent)

Vitrite	46.2
Liptite	
Cutite	0.0
Resite	0.0
Inertite	
Semifusite	2.5
Fusite	1.0
Clarite	5.5
Vitrinertite	14.8
Durite	0.0
Duroclarite	14.5
Vitrinertoliptite	0.0
Clarodurite	1.1
Carbominerite	14.4
TOTAL:	100.0

Based on 1000 point counts.

Sample Number: 81-10891

PYRITE ANALYSIS

	IRREGULAR	DISPERSED EUHEDRA	CLUSTERED EUHEDRA	FRACTURE FILLING	BLEBS	FRAMBOIDS	CELL FILLING	DENDRITIC
Area (Microns^2)	13508	12	1980	8760	7000	64	0	0
Total Area	31324							
% of Total Area	43.12	0.04	6.32	27.97	22.35	0.20	0.0	0.0
Occurrence	7	1	2	2	2	1	0	0
Total Occurrences	15							
% of Total Occurrences	46.7	6.7	13.3	13.3	13.3	6.7	0.0	0.0
Volume %	1.5							

Based on 1000 point counts.

TRW COAL REPOSITORY PROGRAM
No. 1984-DYO-061A

WEST KENTUCKY
Kentucky #11
Peabody Coal Company
River Queen Mine
Master Lab No: 9117484

14 Mesh DISTRIBUTION
Sample C
Lab No: 8110831

	Run#1	Run#2	Average
PROXIMATE ANALYSIS			
Moisture:	9.52	9.57	9.55
Dry Ash:	10.72	10.76	10.74
Dry Volatile:	40.20	40.86	40.53
Dry Fixed Carbon:	49.08	48.38	48.73
SULFUR ANALYSIS			
Dry Pyritic Sulfur:	1.53	1.53	1.53
Dry Sulfate Sulfur:	0.06	0.07	0.07
Dry Total Sulfur:	3.30	3.37	3.34
HEATING VALUE ANALYSIS			
Dry Btu Value:	12985	12996	12991
MAF Btu Value:	14544	14563	14554
ULTIMATE ANALYSIS			
Dry Carbon:	71.71	71.92	71.82
Dry Hydrogen:	5.00	4.94	4.97
Dry Nitrogen:	1.48	1.45	1.47
Dry Chlorine:	0.00	0.00	0.00
Dry Ash:	10.72	10.76	10.74
Dry Sulfur:	3.30	3.37	3.34
Dry Oxygen:	7.79	7.56	7.68
FREE SWELLING INDEX	5.5	5.5	5.5
ASH FUSION TEMPERATURES			
Initial:	2130	2130	2130
Softening (H=W):	2285	2275	2280
Hemispherical (H=W/2):	2375	2365	2370
Fluid:	2420	2430	2425

TRW COAL REPOSITORY PROGRAM
No. 1984-DYO-061A

WEST KENTUCKY
Kentucky #11
Peabody Coal Company
River Queen Mine
Master Lab No: 9117484

SET B
14 Mesh DISTRIBUTION
Sample C
Lab No: 8110831

	Run#1	Run#2	Average
MAJOR ELEMENTS ANALYSIS			
Silica:	47.41	47.05	47.23
Alumina:	20.56	20.34	20.45
Titania:	1.02	1.02	1.02
Ferric Oxide:	21.85	21.87	21.86
Calcium Oxide:	3.10	3.08	3.09
Magnesium Oxide:	0.93	0.96	0.95
Potassium Oxide:	2.49	2.44	2.45
Sodium Oxide:	0.60	0.61	0.61
Sulfur Trioxide:	1.75	1.70	1.73
Phos. Trioxide:	0.23	0.26	0.25
Strontium Oxide:	0.00	0.00	0.00
Barium Oxide	0.02	0.05	0.04
Manganese Oxide:	0.04	0.05	0.05
Undetermined:	0.00	0.57	0.27
TRACE ELEMENTAL ANALYSIS*			
Lead:	3	≤2	3
Mercury	0.10	0.08	0.09
Cadmium:	≤0.1	≤0.1	≤0.1
Arsenic:	4.1	3.1	4.1
Selenium:	1.3	1.0	1.2
Antimony:	≤0.3	≤0.3	≤0.3
Beryllium:	1.1	1.2	1.2
Chromium:	18	20	19
Copper:	9	9	9
Manganese:	33	33	33
Nickel:	12	14	13
Zinc:	38	44	41
Vanadium:	28	32	30
Boron:	140	130	135
Chlorine:	100	80	90

* Parts per million

TRW COAL REPOSITORY PROGRAM
No. 1984-DYO-061A

WEST KENTUCKY
Kentucky #11
Peabody Coal Company
River Queen Mine
Master Lab No: 9117484

SET B
14 Mesh DISTRIBUTION
Sample C
Lab No: 8110831

GEISELER PLASTOMETER

Maximum DDPM:	151
Temperature °C at Maximum DDPM:	426

PARTICLE SIZE ANALYSIS

Passing	Retained On	% Weight
	14M	24.4
14	28	27.5
28	48	18.4
48	100	10.0
100	150	3.5
150	200	2.5
200	250	1.1
250	325	1.9
325M		10.7
		100.00%

TRW COAL REPOSITORY PROGRAM
No. 1984-DYO-061A

WEST KENTUCKY
Kentucky #11
Peabody Coal Company
River Queen Mine
Master Lab No: 9117484

SET B
14 Mesh DISTRIBUTION
Sample C
Lab No: 8110831

PETROGRAPHIC ANALYSIS
Reflectance Analysis
Maceral Group Analysis
Microlithotype Analysis

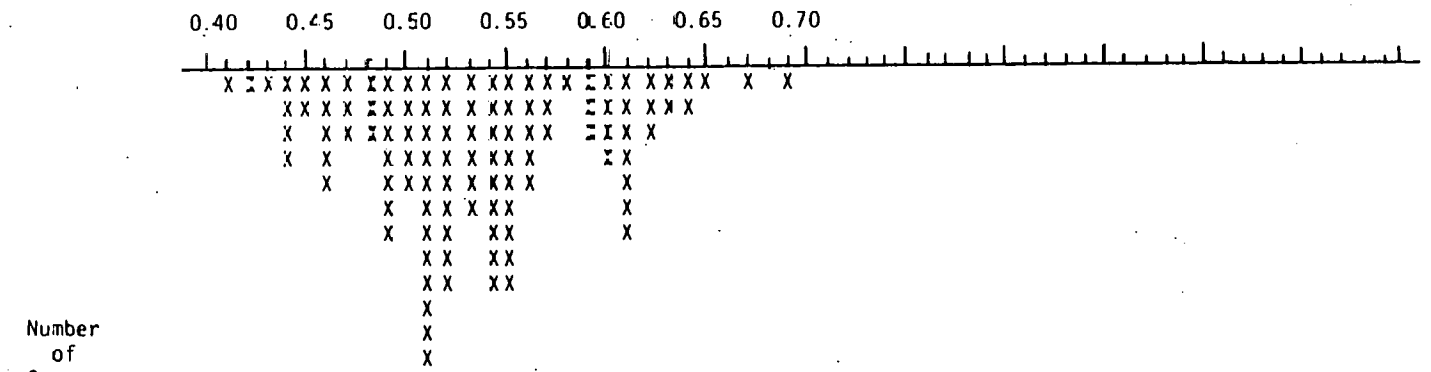
PYRITE SIZE DISTRIBUTION

Report of Analysis on Sample: 81-10831

REFLECTANCE ANALYSIS

Mean-Maximum Vitrinite Ro: 0.54

Distribution of Vitrinite Reflectance Readings:



V-Type Table for Vitrinites (=100%)

V-4	V-5	V-6	V-
24.6	56.4	19.0	

V-Type Table for Vitrinites (= 81.8%)

(Adjusted to = Macera % of Reactive Vitrinites)

V-4	V-5	V-6	V-
20.2	46.1	15.5	

REPORT OF ANALYSIS ON SAMPLE: 81-10831

3/26/81

MACERAL ANALYSIS
(VOLUME PERCENT)
(MINERAL-MATTER CONTAINING BASIS)

MACERAL		MACERAL GROUP	
VITRINITE	81.6	VITRINITE	81.8
PSEUDOVITRINITE	0.2		
EXINITE	2.8	EXINITE (LIPTINITE)	2.8
RESINITE	0.0		
SEMI-FUSINITE*	3.2		
SEMI-MACRINITE*	0.4		
FUSINITE	2.1	INERTINITE	8.6
MACRINITE	0.0		
MICRINITE	2.9		
MINERAL MATTER**	6.0		6.8
TOTAL	100%		100%

TOTAL REACTIVES- 85.8
TOTAL INERTS- 14.2

* CONSIDERED 1/3 REACTIVE, 2/3 INERT FOR PURPOSES OF COKE STABILITY PREDICTIONS.

** CALCULATED FROM 10.74 % DRYASH, 3.34 % DRY SULFUR

Report of Analysis on Sample: 81-10831

MICROLITHOTYPE ANALYSIS
(Volume Percent)

Vitrite	57.6
Liptite	
Cutite	0.1
Resite	0.0
Inertite	
Semifusite	2.0
Fusite	0.8
Clarite	11.1
Vitrinertite	5.6
Durite	0.7
Duroclarite	11.6
Vitrinertoliptite	0.0
Clarodurite	0.2
Carbominerite	10.3
TOTAL:	100.0

Based on 1000 point counts.

Sample Number: 81-10831

PYRITE ANALYSIS

	IRREGULAR	DISPERSED EUHEDRA	CLUSTERED EUHEDRA	FRACTURE FILLING	BLEBS	FRAMBOIDS	CELL FILLING	DENDRITIC
Area ($Microns^2$)	2280	68	16	0	144	0	800	0
Total Area	3308							
% of Total Area	68.92	2.06	0.48	0.0	4.35	0.0	24.18	0.0
Occurrence	3	6	1	0	1	0	1	0
Total Occurrences	12							
% of Total Occurrences	25.0	50.0	8.3	0.0	8.3	0.0	8.3	0.0
Volume %	1.2							

Based on 1000 point counts.

TRW COAL REPOSITORY PROGRAM
No. 1984-DYO-061A

ILLINOIS
Illinois #6
Freeman-United Coal Company
Crown #2 Mine
Master Lab No: 9117467

14 mesh DISTRIBUTION
Sample C
Lab No: 8110481

	Run#1	Run#2	Average
PROXIMATE ANALYSIS			
Moisture:	15.99	15.63	15.81
Dry Ash:	10.03	9.91	9.97
Dry Volatile:	43.37	43.38	43.38
Dry Fixed Carbon:	46.60	46.71	46.66
SULFUR ANALYSIS			
Dry Pyritic Sulfur:	1.35	1.36	1.36
Dry Sulfate Sulfur:	0.04	0.05	0.05
Dry Total Sulfur:	4.18	4.23	4.21
HEATING VALUE ANALYSIS			
Dry Btu Value:	12790	12755	12773
MAF Btu Value:	14216	14158	14187
ULTIMATE ANALYSIS			
Dry Carbon:	70.36	70.50	70.43
Dry Hydrogen:	5.19	5.27	5.23
Dry Nitrogen:	1.35	1.37	1.36
Dry Chlorine:	0.07	0.08	0.08
Dry Ash:	10.03	9.91	9.97
Dry Sulfur:	4.18	4.23	4.21
Dry Oxygen:	8.82	8.64	8.73
FREE SWELLING INDEX	3.0	3.0	3.0
ASH FUSION TEMPERATURES			
Initial:	2085	2130	2108
Softening (H=W):	2210	2260	2235
Hemispherical (H=W/2):	2340	2365	2353
Fluid:	2460	2475	2468

TRW COAL REPOSITORY PROGRAM
No. 1984-DYO-061A

ILLIONOIS
Illinois #6
Freeman-United Coal Company
Crown #2 Mine
Master Lab No: 9117467

SET B
14 Mesh DISTRIBUTION
Sample C
Lab No: 8110481

	Run#1	Run#2	Average
MAJOR ELEMENTS ANALYSIS			
Silica:	48.50	48.28	48.39
Alumina:	16.96	16.86	16.91
Titania:	0.82	0.82	0.82
Ferric Oxide:	19.00	19.29	19.14
Calcium Oxide:	3.85	3.87	3.86
Magnesium Oxide:	0.87	1.01	0.94
Potassium Oxide:	1.95	1.96	1.95
Sodium Oxide:	1.86	1.85	1.85
Sulfur Trioxide:	3.14	2.95	3.04
Phos. Trioxide:	0.15	0.13	0.14
Strontium Oxide:	0.03	0.01	0.02
Barium Oxide	0.02	0.04	0.03
Manganese Oxide:	0.08	0.05	0.06
Undetermined:	2.77	2.88	2.85
TRACE ELEMENTAL ANALYSIS*			
Lead:	≤2	≤2	≤2
Mercury	0.11	0.10	0.11
Cadmium:	≤0.1	≤0.1	≤0.1
Arsenic:	1.9	1.7	1.8
Selenium:	1.7	1.4	1.6
Antimony:	≤0.4	≤0.4	≤0.4
Beryllium:	1.1	1.2	1.2
Chromium:	18	19	19
Copper:	9	9	9
Manganese:	39	39	39
Nickel:	14	12	13
Zinc:	42	39	40
Vanadium:	23	22	23
Boron:	260	270	265
Chlorine:	880	880	880

* Parts per million

TRW COAL REPOSITORY PROGRAM
No. 1984-DYO-061A

ILLINOIS
Illinois #6
Freeman-United Coal Company
Crown #2 Mine
Master Lab No: 9117467

SET B
14 mesh DISTRIBUTION
Sample C
Lab No: 8110481

GEISELER PLASTOMETER

Maximum DDPM:	36
Temperature °C at Maximum DDPM:	415

PARTICLE SIZE ANALYSIS

Passing	Retained On	% Weight
	14M	33.3
14	28	30.6
28	48	11.8
48	100	8.8
100	150	2.7
150	200	1.9
200	250	0.8
250	325	1.5
325M		8.6
		100.00%

TRW COAL REPOSITORY PROGRAM
No. 1984-DYO-061A

ILLINOIS
Illinois #6
Freeman-United Coal Company
Crown #2 Mine
Master Lab No: 9117467

SET B
14 mesh DISTRIBUTION
Sample C
Lab No: 8110481

PETROGRAPHIC ANALYSIS
Reflectance Analysis
Maceral Group Analysis
Microlithotype Analysis

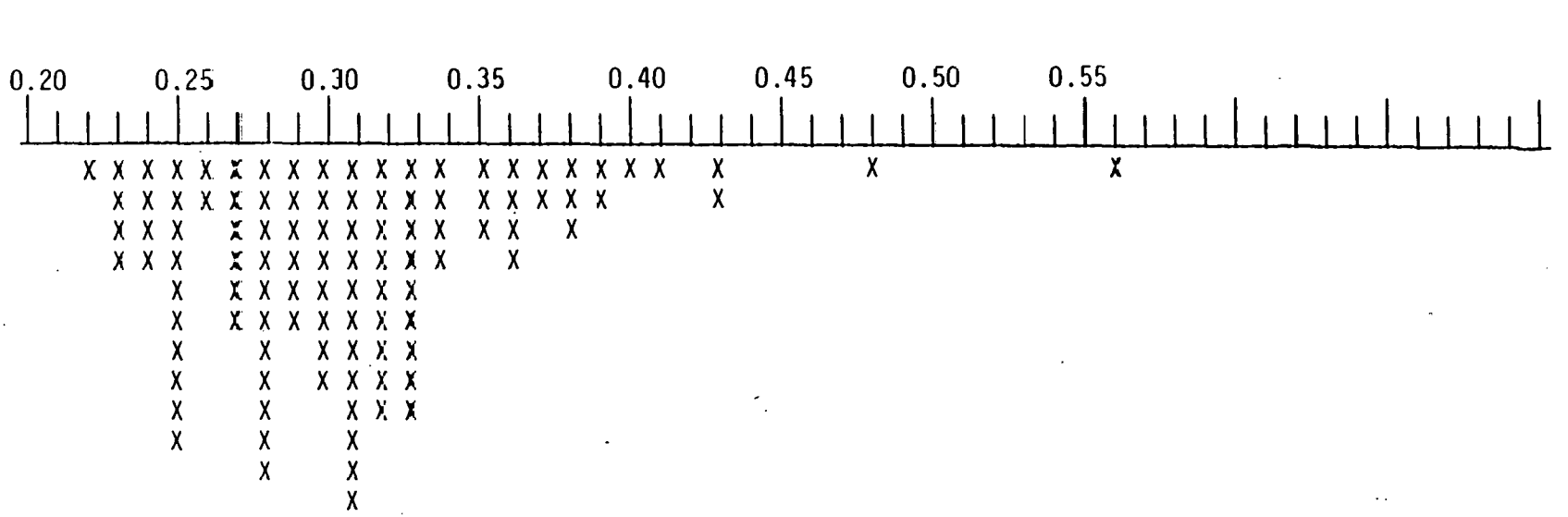
PYRITE SIZE DISTRIBUTION

Report of Analysis on Sample: 81-10481

REFLECTANCE ANALYSIS

Mean-Maximum Vitrinite Ro: 0.31

Distribution of Vitrinite Reflectance Readings:



Number of Counts (Total= 106)

V-Type Table for Vitrinites (=100%)

V-2	V-3	V-4	V-5

Report of Analysis on Sample: 81-0481

MACERAL ANALYSIS
(Volume Percent)

(Mineral-Matter Free Basis)

MACERAL:		MACERAL GROUP:	
Vitrinite	89.1		
		Vitrinite	89.1
Pseudovitrinite	0.0		
Exinite	2.4		
		Exinite (Liptinite)	2.7
Resinite	0.3		
Semi-Fusinite	4.1		
Semi-Macrinite	0.6		
Fusinite	2.9	Inertinite	8.2
Macrinite	0.0		
Micrinite	0.6		
Sclerotinite			
TOTAL:	100.0		100.0

Report of Analysis on Sample: 81-10481 - MICROLITHOTYPE ANALYSIS

Vitrite	70.7		
Liptite	0.0		
		Nonomaceral	72.2
Fusite	0.5		
Semi-Fusite	1.0		
Clarite	10.0		
Vitrinertite	6.7	Bimaceral	16.8
Durite	0.1		
Durociarite	3.6		
Vitrinertoliptite	0.0	Trimaceral	11.0
Clarodurite	0.0		
Carbominerite	7.4		
Total	100.0		100.0

Sample Number: 10481

PYRITE ANALYSIS

	IRREGULAR	DISPERSED EUHEDRA	CLUSTERED EUHEDRA	FRACTURE FILLING	BLEBS	FRAMBOIDS	CELL FILLING	DENDRITIC
Area ($Microns^2$)	20,288	34	725	0	0	0	0	100
Total Area	21,147							
% of Total Area	95.94	0.16	3.43	0.0	0.0	0.0	0.0	0.47
Occurrence	7	2	2	0	0	0	0	1
Total Occurrences	12							
% of Total Occurrences	58.3	16.7	16.7	0.0	0.0	0.0	0.0	8.3
Volume %	1.2							

Based on 1000 point counts.