Final Report for Phase I — Coal Desulfurization by Low Temperature Chlorinolysis

July 6, 1977 to November 6, 1977
(Contract No. J0177103)

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The Final Report for Phase I of the Coal Desulfurization by Low Temperature Chlorinolysis project conducted by the Jet Propulsion Laboratory under U. S. Bureau of Mines Contract No. J0177103 for the period of July 6, 1977, through November 6, 1977, is presented. The reported activity covers laboratory scale experiments on twelve bituminous, sub-bituminous and lignite coals, and preliminary design and specifications for bench-scale and mini-pilot plant equipment.

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Final Report for Phase I — Coal Desulfurization by Low Temperature Chlorinolysis

July 6, 1977 to November 6, 1977
(Contract No. J0177103)

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November 23, 1977

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Pasadena, California

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Prepared by the Jet Propulsion Laboratory,
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Department of Energy by agreement with the
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PREFACE

The work described in this report was performed by the Control and Energy Conversion Division of the Jet Propulsion Laboratory.

FOREWORD

The Final Report for Phase I of the Coal Desulfurization by Low Temperature Chlorinolysis project conducted by the Jet Propulsion Laboratory under U.S. Bureau of Mines Contract No. J0177103 for the period of July 6, 1977 through November 6, 1977 is presented here. The reported activity covers laboratory scale experiments on twelve bituminous, sub-bituminous and lignite coals, and preliminary design and specifications for bench-scale and mini-pilot plant equipment. A Phase II follow-on program will be carried out that includes bench-scale and mini-pilot plant construction and operation. The combined Phase I and Phase II programs are discussed in JPL Proposal 70-763 for "Coal Desulfurization by Low Temperature Chlorinolysis", dated December 30, 1976.

The work described in this final report involves the "Coal Desulfurization Process" invention that is the subject of a pending patent application made in the performance of Prime Contract NAS 7-100 between the National Aeronautics and Space Administration and California Institute of Technology.

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INTRODUCTION

A national need exists for low-cost processes to remove sulfur from coal to comply with both State and Environmental Protection Agency standards for SO_2 emissions. The emission standard of 1.2 lb/10^6 Btu in stack gases corresponds to approximately 0.7 weight percent sulfur in coal with a heating value of 12,000 Btu/lb. Only 12.3 percent (ref. 1) of the United States coal reserves are within this compliance level. The major recoverable fractions of eastern and midwestern coals contains more than 2 weight percent sulfur. The sulfur is normally equally distributed between organic and inorganic sulfur. Organic sulfur is intimately bound within the structure of the coal and requires the severance of C-S bonds to dislodge the organic sulfur. Inorganic sulfur is normally present as FeS_2 (iron pyrites) along with some sulfur present as sulfate. Removal of the pyritic sulfur can be accomplished by state-of-the-art physical separation methods such as float-sink methods. If pyrites are present as relatively coarse particles, pyritic removal by flotation is effective. However, with very fine particles of pyritic sulfur, only limited removal of pyritic sulfur is possible. On the average, 60 percent of pyritic sulfur removal is possible by float-sink methods. Other chemical treatment processes (ref. 2) are available for pyritic sulfur removal; however, the processing costs are high. Also, organic sulfur removal is obtained in conjunction with pyritic sulfur removal only to a limited extent. Claims for organic sulfur removal by various chemical treatment methods indicate a maximum of 40 percent organic sulfur removal with a lower organic sulfur removal being more likely, as indicated by inspection of published data for the cited chemical processes (ref. 3). Many of the chemical treatment methods claim no organic sulfur removal, which limits the application and effectiveness of these processes and points up the relatively high attendant cost for limited sulfur removal.

Other processes such as hydrogenation for coal liquefaction and coal gasification are beset by high costs and the problems of converting solid coal to a different state. Flue gas desulfurization requires the installation of scrubbers on new and existing plants. Although these costs for flue gas desulfurization are the most attractive of any of the desulfurization processes that are available today, the costs are still high at $12 to $15 per ton of coal. Flue gas desulfurization is still in a developmental stage and the equipment is beset with many maintenance problems because of corrosion, scaling, and plugging.

Solvent refined coal processing, although attractive in generating a 16,000 Btu/lb, low-ash coal, is expensive. Processing costs bring the price of coal to an estimated total of $60 per ton (ref. 4).

JPL started experimental work approximately two years ago on coal beneficiation and coal desulfurization under the Director's Discretionary fund. An outgrowth of this activity is the JPL Low Temperature Chlorinolysis process for coal desulfurization. The initial laboratory studies indicated that the process was capable of removing up to 70 percent organic sulfur, 76 percent pyritic sulfur and over 70 percent total sulfur from an Illinois No. 6 bituminous coal containing 4.77 weight percent total sulfur.

The literature indicates that this represented a higher organic sulfur removal than claimed by any existing chemical cleaning process for removal of sulfur from solid coal. A preliminary process cost estimate indicated that the attendant costs were a low $9-10 per ton of coal, because of the relatively mild conditions of temperature and pressure for processing and the relatively short retention times in the specific operations. The process and attendant costs were reviewed by Bechtel Corporation for the U.S. Bureau of Mines.

As a consequence of the favorable outlook for the process in terms of costs as well as sulfur removal, JPL has undertaken (under U.S. Bureau of Mines and Department of Energy sponsorship) a Phase I study of the process by investigating twelve high-sulfur coals, as well as providing a parametric investigation of operating conditions for the chlorination, hydrolysis and dechlorination steps that constitute the overall process. The Phase I study results are reported here. The Phase II program constitutes a follow-on to the present effort that completes the work outlined in JPL Proposal 70-763.

SUMMARY

The final report for Phase I of the Coal Desulfurization by Low Temperature Chlorinolysis Project carried out by the Jet Propulsion Laboratory under U.S. Bureau of Mines Contract No. J0177103 from July 6, 1977 to November 6, 1977, is presented here. The reported work was performed by the Jet Propulsion Laboratory at Pasadena, California. A Phase II follow-on program will be carried out by JPL under U.S. Department of Energy sponsorship to complete the program outlined in JPL Proposal 70-763, dated December 30, 1976.

The Phase I program consisted of:

(1) Laboratory testing of twelve coals including 9 bituminous, 2 sub-bituminous, 1 lignite, 1 high organic sulfur and 1 high pyritic sulfur coal. These were selected with consultation and approval of the United States Bureau of Mines.

(2) Preliminary design and equipment specifications for bench-scale (batch) and mini-pilot plant (continuous flow) coal desulfurization that included immersion testing of reactor construction materials. (Follow-on Phase II will include construction and operation of bench-scale and mini-pilot plant equipment.)

Laboratory testing was carried out on 100-gram samples of +200 mesh coal using laboratory glassware. Major process steps included: chlorination, hydrolysis and dechlorination. Parameters tested included time, temperature, solvent, water-to-coal ratios, chlorine rate and steam-to-coal ratios. The chlorination was carried out with chlorine gas bubbled through a moist powdered coal suspended in an organic solvent at temperatures of 50 to 100°C and atmospheric pressure for times of 10 to 120 minutes. Solvents included methyl chloroform, carbon tetrachloride and tetrachloroethylene at solvent/coal ratios of 2/1. Water/coal ratios were 0.3/1, 0.5/1 and 0.7/1. Chlorine feed rates were from 0.125 to 1.0 SCFH. Hydrolysis was conducted at water/coal ratios of 4/1 and 2/1 with a 2/1 and 1/1 water/coal displacement wash of the filter cake at water temperatures of 60 to 100°C and wash times of 20 to 120 minutes. Dechlorination tests were conducted at temperatures of 350 to 550°C using steam rates of 0.4 to 121 grams/hour with 2 to 10 gram samples of chlorinated and hydrolyzed coal contained in a 1-inch-diameter quartz tube rotated at 2 RPM.

PSOC-219 (HVA Bituminous, Kentucky No. 4, 2.56% total sulfur) was used for parametric screening of process operating conditions. Thirty screening runs were made with PSOC-219 and 17 runs were made on the eleven remaining coals. The twelve raw coal samples analyzed:

Organic sulfur - 0.46 - 2.24 wt. %

Pyritic sulfur - 0.20 - 5.01 wt. %

Total sulfur - 1.22 - 6.66 wt. %

Desulfurization results on the treated coal indicate:

Coal Description Rank, Seam, County, State, ERDA PSOC No.	Number of Runs	Raw Coal Total Sulfur (Wt. %)	Sulfur Removal Range (%)		
			Organic	Pyritic	Total
HVA Bit., Ky, No. 4, 219	30	2.56	3.7-87	11-100	15-75
Lignite, Zap, Mercer, N. Dak, 086	1	1.22	38-62	21-59	21-57
Sub-bit A, Seam 80, Carbon, Wyo, 097	1	1.23	0-17	18-87	1-34
Raw Head, 3A, Upper Freeport Seam, Sommerset, Pa, PHS-398	2	3.01	-	73-96	56-78
HVA Bit., Ill. No. 6, Knox, Ill, 190	2	3.05	15-34	87-98	37-58
HVB Bit., Pittsburgh, Wa, Pa, 108	2	3.13	16-83	36-96	26-78
Sub-bit. B, Big D, Lewis, Wa, 240 A1	1	3.36	72	58	64
HVB Bit., Ky, No. 9, 213	1	3.82	72	13	43
HVA Bit., Ohio No. 8, Harrison, Ohio, 276	3	5.15	48-74	64-99	49-83
HVA Bit., Clarion, Jefferson, Pa, 342	2	6.55	7-20	60-82	49-59
HVC Bit., Ill, No. 6, Saline, Ill, 026	1	6.66	37-42	79-89	67-75
Bit., Mine No. 513, Upper Clarion, Butler, Pa PHS-513	1	1.76	27-34	-	27-34

Sulfur removal data scatter is extensive. Average sulfur removals for PSOC-219 are: organic sulfur - 50-60 percent, pyritic sulfur - 60-70 percent, total sulfur - 60-70 percent. Of the remaining 11 coals, three had an organic sulfur reduction of less than 20 percent, four had an organic sulfur reduction of 71-83 percent, and the remaining coals had intermediate organic sulfur removals. The total sulfur reduction for 3 of the 11 coals is less than 34 percent. The remaining coals had total sulfur reduction of 37 to 78 percent with accompanying pyritic sulfur removals of 13 to 99 percent.

An evaluation of the accuracy and precision of the Galbraith Laboratory's sulfur analysis indicates that the mean deviation of five identical samples was ±0.13 (15%) on total sulfur and ±0.05 (7%) and ±0.07 (44%) on organic and pyritic sulfur, respectively, which could account for up to 5 percent variance in sulfur reduction values. These variances, although appreciable, cannot account for existing variances in the sulfur data. However, comparison of Galbraith Laboratory's analyses with those of the U.S. Department of Energy (analysis and Testing Laboratory, Pittsburgh, Pa) indicated that sulfur analyses performed by Galbraith gave substantially higher residual sulfur values than were reported by the U.S. Department of Energy. The U.S. Department of Energy indicated values for total sulfur of 0.28 weight percent (33 relative percent) less than Galbraith. Organic sulfur determinations were also 0.18 weight percent (27 relative percent) less and pyritic sulfur 0.09 weight percent (56 relative percent) less. If the Department of Energy results correctly identify a bias of 0.2 to 0.3 percent sulfur in the Galbraith Laboratory's data, the residual sulfur in a large fraction of the coals tested would fall in compliance with sulfur standards, i.e., less than 0.7 weight percent sulfur.

Multiple regression analyses of the laboratory data were conducted using an existing computer program at JPL for statistical analysis of data. Results of the analyses are included, and confirmed the large unaccountable variance in the majority of the data. The principal correlation (55% accounting of variance) was with high organic sulfur removal and high organic sulfur content in the raw coal. Other variables, including temperature, water, coal and retention time, showed very little correlation with the desulfurization results either in the form of residual sulfur values or with sulfur removal values. The variance in the data appears to be greater than that which can be explained by sulfur analysis deviations. Complexities in the coal desulfurization reactions are suggested, especially in view of the fact that increased retention times beyond 30 minutes appear to add very little to coal desulfurization. Parameters such as temperature, which should exhibit a significant effect on the reaction chemistry, lose any significance within the scatter of the data.

Hydrolysis of the chlorinated coal for 20 minutes at a water/coal ratio of 2/1 at temperatures of 80°C, combined with a 2/1 water/coal filter cake displacement wash, reduces residual sulfate to less than 0.1% in the treated coal.

Dechlorination of the treated coal samples has shown that substantial variations in residual chlorine are found under identical conditions of dechlorination. Residual chlorine levels range between 0.06 and ~1%. No satisfactory correlation with temperature, steam rates, retention time or coal has been noted for achieving consistent dechlorination to 0.1% residual chlorine. Additional research will be required to obtain low residual chlorine values (~0.1%) on a consistent basis.

Preliminary design and equipment specifications were completed for Phase II bench-scale equipment for testing of the coal desulfurization process at 2000 grams/batch and for a Phase II mini-pilot plant continuous flow operation at 2000 grams/hour. An overall mini-pilot plant equipment layout drawing is shown in Figures 6, 7 and 8, with accompanying detailed drawings of major equipment units presented in Figures 9 through 18.

Immersion tests were conducted for brick and mortar samples supplied by Pennwalt Corporation and Stebbins Engineering and Manufacturing Co. as candidate materials for chlorinator and hydrolyzer construction. Although the tests covered a shortened period of time, 18 to 30 days, the tests were effective in screening out materials that proved unsuitable after immersion in the chlorinator reaction conditions. Suitable construction materials for the chlorinator and hydrolyzer were recommended by Pennwalt and Stebbins on the basis of the materials immersion testing program.

Phase II activities will concentrate on bench-scale batch tests at 2000 grams per batch for parametric screening of the coal desulfurization operating conditions and construction and preliminary operation of a 2000 grams/hour continuous-flow mini-pilot plant. The mini-pilot plant will include equipment integration for continuous flow from a pulverized coal hopper through the chlorinator, hydrolyzer and dechlorinator to provide dried, desulfurized coal.

Laboratory data is shown in Tables 2 and 3, multiple regression analysis data in Tables 15 through 19, and mini-pilot plant and major equipment drawings are shown in Figures 9 through 18. A detailed discussion and representation of the coal desulfurization process is presented in the Technical Discussion (Tables 17, 18 and 19).

CONCLUSIONS

Phase I research and development of the JPL low temperature chlorinolysis process for coal desulfurization has demonstrated or indicated:

1) <u>Sulfur Removal</u>

   a) Generally high removal of organic sulfur with an average removal of better than 50 percent (27 runs out of 46 indicate over 50 percent organic sulfur removal).

   b) Highest removal of pyritic sulfur at optimum operating conditions was 100 percent. Average removals of 60 to 70 percent occurred in 41 runs out of 46.

   c) High removal of total sulfur with average removal of 60 to 70 percent (33 runs out of 45 indicated better than 60 percent total sulfur removal).

   d) Residual sulfur levels in the 12 coals treated averaged between 0.6 to 1.5 weight percent (44 out of 46 runs were below 1.5 weight percent total sulfur). Sulfur compliance levels for a 12,000 Btu/lb coal are 0.7 weight percent sulfur. Average heating values for eleven of the coals tested were 11,083 Btu/lb on an "as received basis" and 12,329 Btu/lb on a moisture-free basis.

   e) Since peak levels of organic sulfur removal and pyritic sulfur removal are 83 and 100 percent, respectively, the possibility exists of consistently achieving higher coal desulfurization levels than currently indicated by average sulfur removal data.

   f) A substantial amount of scatter exists in the coal desulfurization data for any given run as well as between runs. The data scatter can be explained in part by analytical errors and correlation with changes in operating parameters.

2) <u>Coals Tested</u>

   a) The majority of the 12 coals tested, including 9 bituminous, 2 sub-bituminous and 1 lignite coals, showed high organic and pyritic sulfur removal. Only 1 bituminous coal (PSOC-382, Clarion, Jefferson, Pa) and 1 sub-bituminous coal (PSOC-097, Seam 80 Carbon, Wyo) showed less than 20 percent organic sulfur removal and accompanying low total sulfur removal.

   b) Geographical origins of the coals tested included: Western - 2 sub-bituminous, 1 lignite; mid-Western - 5 bituminous; Eastern - 4 bituminous. No substantial differences were noted for sulfur removal based on geographical origin.

c) The coal desulfurization process should be applicable to a wide variety of bituminous, sub-bituminous and lignite coals that encompass eastern, mid-western and western coals.

3) Process Operation Conditions

a) Chlorination

1 Chlorination data suggests that reaction times of less than 1 hour may be optimum. Chlorinations for extended time periods may promote secondary reactions of sulfur compounds with the coal structure that may reintroduce sulfur into the coal. This complication may in part explain some of the scatter in coal desulfurization data.

2 No significant difference was observed among the use of methyl chloroform, carbon tetrachloride and tetrachlorethylene in the coal desulfurization results.

3 Chlorine injection rates at the maximum rate absorbed by the coal slurry without loss to the gas phase appear desirable to maximize the coal desulfurization reaction rates.

4 Water-to-coal ratios from 0.3 to 0.7 in the chlorination reaction provided no significant differences in the coal desulfurization results.

5 Multiple regression analyses of the laboratory data on coal desulfurization have confirmed the large unaccounted variance in the data. The major data correlation obtained was for organic sulfur removal correlating with the amount of organic sulfur present in the raw coal. Other parameters exhibited a very low influence on coal desulfurization data.

b) Hydrolysis

1 Combination of solvent distillation with the hydrolysis stage has simplified and improved solvent recovery.

2 Hydrolysis of chlorinated coal in a single stage wash with water/coal at 2/1 for 20 minutes and 80°C, followed by a water/coal at 2/1 for the filtration wash, is sufficient to consistently reduce sulfate sulfur to less than 0.1 percent. This provides a substantial improvement in time and water requirements over initial hydrolysis conditions.

c) Dechlorination

1 Dechlorination of heated coal with steam/coal ratios of 1/4, temperatures of 350-550°C, and times of 20 minutes to 1 hour provides residual chlorine levels of 0.06 to 1.0 weight percent.

2 Consistent dechlorination levels to less than 0.1 weight percent chlorine have not been achieved.

4) Analytical Chemistry Results

a) Galbraith Laboratory analyses of sulfur types in the heated coal samples have exhibited significant deviations between duplicate samples (average of ±0.13 percent total sulfur on 5 samples for total sulfur) and a possible bias towards reporting higher residual sulfur values (0.2 to 0.3 weight percent total sulfur). An improvement in analytical procedures might substantially increase the number of treated coal samples meeting compliance levels of less than 0.7 weight percent total residual sulfur.

b) Ultimate analyses of treated coal samples indicate a 1 to 3 percent reduction in hydrogen, a slight decrease in ash and a 1 to 3 percent carbon increase over the raw coal. A comparison of heating values between treated and raw coal samples is questionable since raw and treated coal samples were analyzed by different laboratories and relative values are questionable.

c) Trace metal analyses of treated coals indicate substantial reductions (48 to 91 percent) of titanium, phosphorus, arsenic, lead, vanadium, lithium and beryllium.

d) Product yields of coal have been demonstrated with coal losses of 3.81% to 23.67%. The 3.81% loss is representative of losses that have been accounted for, whereas the 23.67% loss includes unaccounted coal. Unaccounted coal is thought to be primarily solid particles of product coal lost in the dechlorination apparatus.

5) Materials Testing

Immersion tests of brick and mortar samples supplied by Pennwalt and Stebbins Engineering were successful in screening suitable materials recommended for construction of the reactors.

6) Equipment Specifications and Requirements

Preliminary design and major equipment specifications for a continuous flow mini-pilot plant for 2000 grams/hour have been completed. The pilot plant provides for an integrated flow operation from a ground coal hopper through a chlorinator, hydrolyzer, filter and dechlorinator to a clean coal product hopper.

7) Bench-Scale Screening Tests

Bench-scale screening tests of the coal desulfurization process at 2000 grams/batch should be extremely beneficial in complementing the laboratory data that has been obtained as well as providing equipment improvement for conduct of the chlorination reaction and thus achieving improved coal desulfurization results. The larger scale operation will also provide data more representative of engineering-scale operations.

RECOMMENDATIONS

1. Since Phase I, laboratory screening of 12 coals and extensive parametric investigations of PSOC-219 coal by the low temperature chlorinolysis process for coal desulfurization has been completed, Phase II activities that include bench-scale testing (2000 grams per batch) and construction of a mini-pilot plant for continuous flow operation at 2000 grams per hour should be initiated immediately.

2. Bituminous coals PSOC-219 (Ky #4, Hopkins, Ky HVA Bituminous Coal - 2.56 percent total sulfur) and PSOC-276 (Ohio #8, Harrison, Ohio, HVA Bituminous Coal - 5.15 percent total sulfur) are recommended for Phase II bench-scale and mini-pilot plant operations. PSOC-219 represents the extensively tested coal in Phase I and PSOC-276 represents a high sulfur coal with high organic and high pyritic sulfur content with a demonstrated potential for high (83 percent) total sulfur removal.

3. Phase II equipment designs and operations should reflect reduced reaction times of less than 1 hour for each of the chlorination, hydrolysis and dechlorination stages as reflected in the Phase I laboratory evaluation.

4. Provisions should be incorporated in the continuous flow mini-pilot plant for monitoring and recovery of HCl to demonstrate the viability and economics of HCl recovery for recycle to the Kel-chlor process.

5. Provisions should also be incorporated for monitoring and treatment of the waste water effluent from the hydrolyzer for recovery and/or disposal of the sulfuric acid and metal salts and providing an attendant economic analysis.

6. Fundamental investigations of the coal desulfurization reactions are recommended to obtain the necessary data to optimize the coal desulfurization process conditions and to achieve maximum levels of coal desulfurization in all cases. Since levels of organic sulfur removal of 83 percent and pyritic sulfur removal of 100 percent have been demonstrated, the possibility exists of consistently achieving higher coal desulfurization levels than currently indicated by average sulfur removal data.

TECHNICAL DISCUSSION

The Phase I program under U.S. Bureau of Mines Contract No. J0177103 consisted of the following tasks for investigation of the JPL Low Temperature Chlorinolysis process for Coal Desulfurization:

1.1 (Task I*) - Laboratory scale experimental testing of twelve bituminous, sub-bituminous, and lignite coals representing high sulfur coals, listed in Table 1, and parametric screening of coal desulfurization conditions using a selected bituminous coal, PSOC-219. The coals were selected with consultation and approval of the U.S. Bureau of Mines.

1.2 (Task IIIA*) - Design and equipment specifications for the bench-scale and continuous-flow mini-pilot plant for Phase II construction and operation. Bench-scale equipment is for 2000 grams of coal per batch. Mini-pilot plant is for 2000 grams of coal per hour. Process operations include chlorination, hydrolysis and dechlorination.

1.3 (Task IV*) - Analyses of raw coal and coal product samples for sulfur forms, caloric content, trace elements consisting of As, Se, Pb, Hg, Cd, Cl, ultimate analysis and attendant water and gas analyses.

1.4 (Task V*) - Experimental and analytical studies for elucidating coal desulfurization reactions.

1.5 (Task VI*) - Data analysis and report preparation.

Laboratory Scale Screening Studies (1.1)

The laboratory coal processing for desulfurization by the JPL Low Temperature Chlorinolysis process is depicted in Figure 1. A modification to the basic laboratory process illustrated in Figure 1 was made during the test program by integrating the solvent evaporation step with the hydrolysis by adding water to the coal slurry before solvent evaporation and then flashing the solvent from the coal-water slurry.

Apparatus

Laboratory apparatus for chlorination of the coal is depicted in Figure 2. Laboratory apparatus for hydrolysis of chlorinated coal is depicted in Figure 3. Dechlorination apparatus for the treated coal is depicted in Figure 4.

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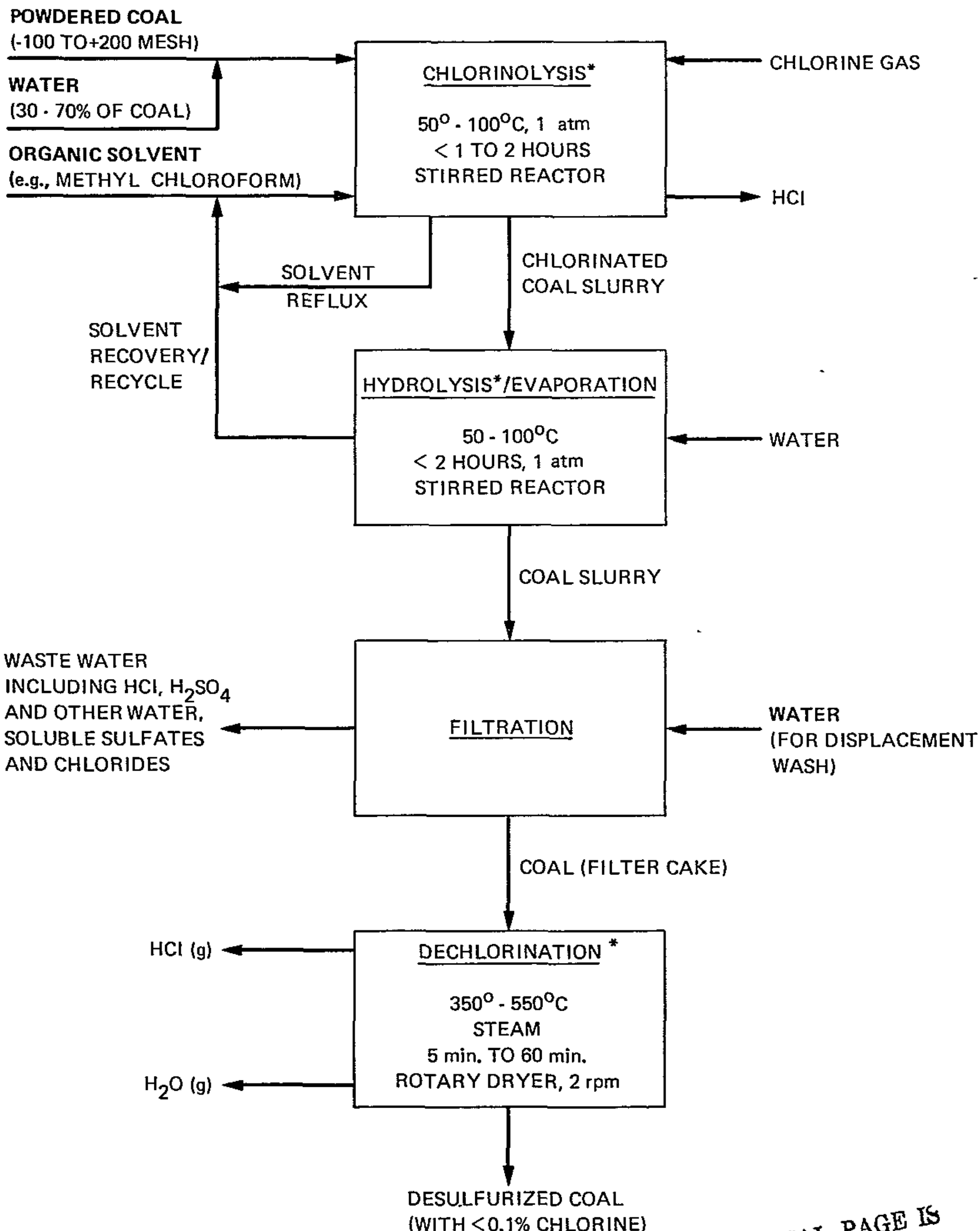
* Starred task numbers correspond to those in JPL Proposal 70-763.

Table 1. Selected Coals for Chlorinolysis Experiments Under Bureau of Mines-Sponsored Program

ERDA PSOC Number	Seam, County & State	Rank	Ash Content (Wt.%)	Sulphur Content, Wt. %		
				Organic	Pyritic	Total
108	Pittsburgh, Washington, Pennsylvania	HVA (Bit.)	9.50	1.07	2.06	3.13
219	Kentucky #4, Hopkins, Kentucky	HVA (Bit.)	8.06	1.08	1.40	2.56
190	Illinois, #6, Knox, Illinois	HVA (Bit.)	8.49	1.90	1.05	3.05
276	Ohio #8, Harrison, Ohio	HVA (Bit.)	11.19	2.24	2.07	5.15
026	Illinois #6, Saline, Illinois	HVC (Bit.)	10.84	2.08	4.23	6.66
342	Clarion, Jefferson, Pennsylvania	HVA (Bit.)	9.19	1.39	5.01	6.55
240A1	Big D, Lewis, Washington	Sub-bit B	29.40	1.75	1.60	3.36
097	Seam 80, Carbon, Wyoming	Sub-bit A	9.80	0.84	0.38	1.23
086	Zap, Mercer, N. Dakota	Lignite	11.49	0.63	0.56	1.22
213	Kentucky #9	HVB (Bit.)	9.36	1.86	1.89	3.32
PHS-398 (BOM)*	Raw Head, 3A, Upper Freeport Seam, Somerset, Pennsylvania	-	19.7	0.46	2.26	3.01
PHS-513 (BOM)*	Mine 513, Upper Clarion, Butler, Pennsylvania	-	-	1.76 (Physically cleaned, high organic coal	<0.2	1.76

* Samples received from Dr. Scott R. Taylor, Department of Energy, Pittsburgh, Pennsylvania.



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*LABORATORY GLASSWARE EQUIPMENT FOR THESE PROCESSES IS SHOWN IN FIGURES 2, 3 AND 4

Figure 1. Process flow diagram for laboratory scale coal desulfurization

Laboratory Data

Laboratory data on the coal desulfurization process is tabulated in Tables 2 and 3. Table 2 represents all 47 runs that have been made and lists operating parameters for chlorination, hydrolysis and dechlorination for each of the twelve coals that have been tested. It gives the analyses for organic, pyritic, sulfate and total sulfur forms for the raw and treated coal, and includes sulfur reduction in the treated coal for organic, pyritic and total sulfur. The data are grouped by coal with PSOC-219 (HVA Bituminous, Kentucky, No. 4 - 2.56 percent total sulfur) representing the coal selected for parametric analysis of operating conditions. Thirty runs were made with PSOC-219 and seventeen runs with the remaining 11 coals. The selected coals for testing are listed in Table 1 and represent organic sulfur from 0.63 to 2.24 weight percent, pyritic sulfur from 0.20 to 5.01 weight percent and total sulfur from 1.22 to 6.66 weight percent. Chlorine values are listed for the raw coals as well as the treated coals before and after dechlorination. Table 3 repeats the coal desulfurization data listed in Table 2, organized in terms of increasing total sulfur removal. The data for any given coal desulfurization run are separated according to the extent of total sulfur removal.

Chlorination. Coal chlorination was conducted by using 100 grams of +200 mesh coal moistened with water and suspended in 200 grams of organic solvent. The coal slurry was contained in a stirred 500 ml flask equipped with a reflux condenser, cold trap, water scrubber and gas holder. Chlorine injection was started at 0.125 SCFH and then increased to 0.25, 0.5 and 1.0 SCFH in ensuing runs. Chlorine injection rates of 1.0 SCFH were found to be excessive, with an immediate carry-over of chlorine into the cold trap. A 0.5 SCFH injection rate was found to be readily adsorbed by the coal slurry until a saturation level was reached after nearly 1 hour of chlorination. At that time, chlorine started leaving the coal slurry and was collected in the dry-ice cold trap. Reaction parameters that were investigated included:

Solvents - methyl chloroform, carbon tetrachloride, tetrachloroethylene

Temperatures - 50, 60, 74, 100°C

Reaction times - 10, 15, 20, 30, 60, 120 minutes

Water/coal - 0.3/1, 0.5/1, 0.7/1

Changing of the parameters under investigation did not produce significant effects on coal desulfurization. Correlation of the parameters with the coal desulfurization data is discussed in the Linear Multiple Regression Analysis Section.

Hydrolysis. Hydrolysis conditions were changed during the course of the test program by incorporating the coal slurry from the chlorinator directly into the hydrolyzer without first distilling off the organic solvent from the slurry. The solvent, insoluble in water, was flashed from the coal-water slurry by maintaining a temperature above the boiling point of the solvent. In the case of methyl chloroform, the boiling point is 74°C. Hydrolysis conditions included: water/coal at 2/1, 3/1 and 4/1 with one and two washes for 5, 10, 15, 20, 25, 30, 40, 45, 50,

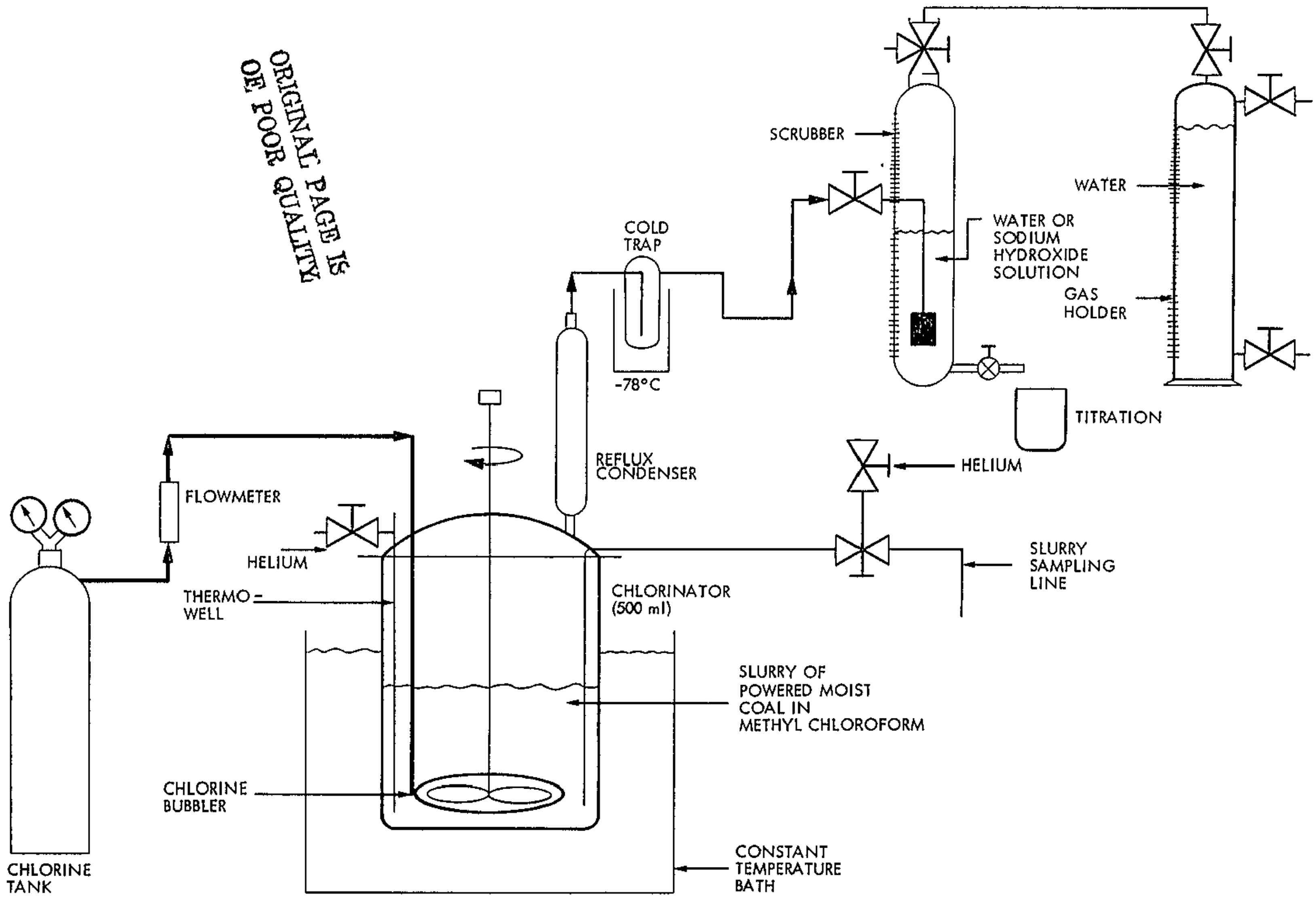

Figure 2. Laboratory glassware apparatus
for chlorination of coal

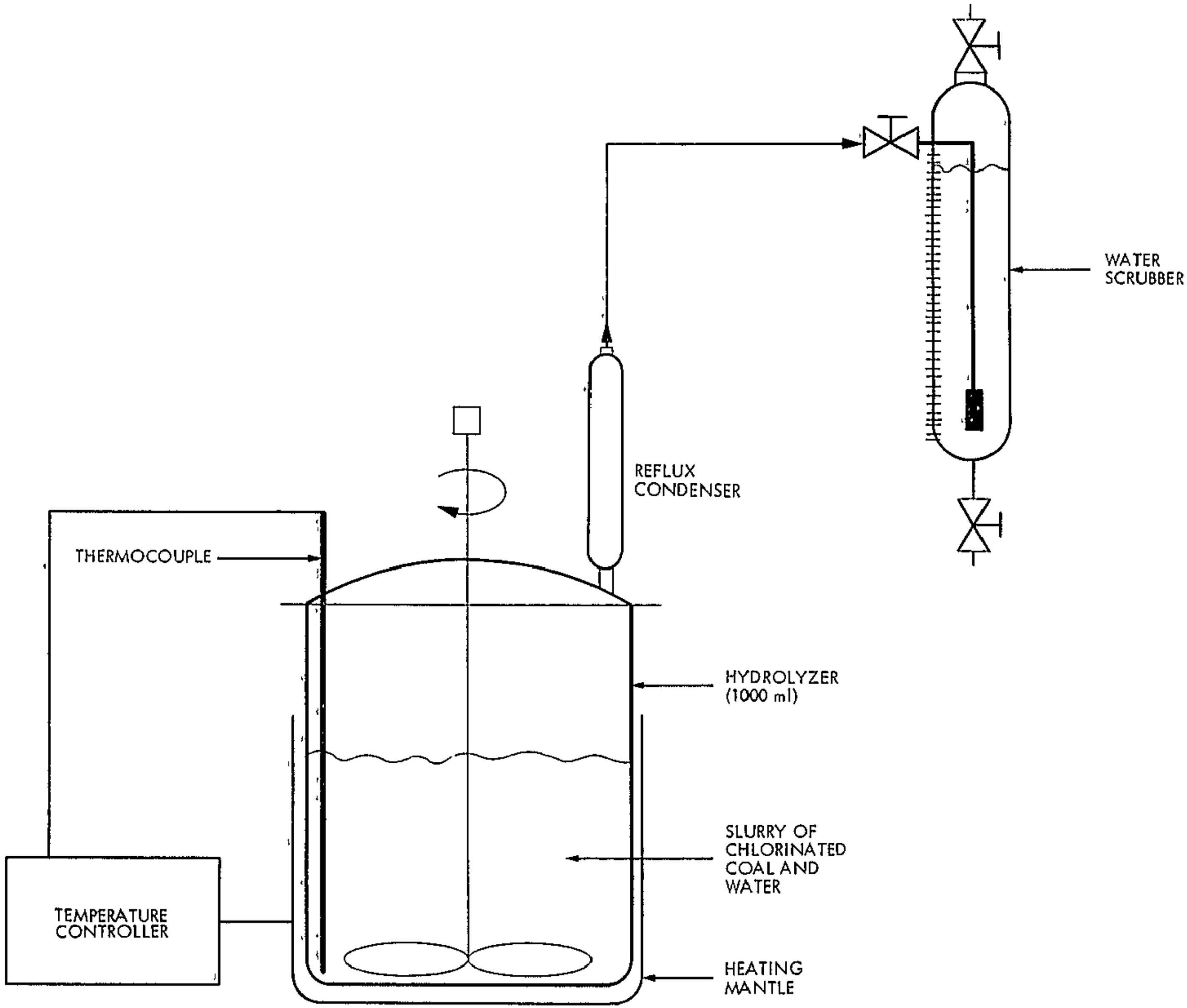

Figure 3. Laboratory glassware apparatus for hydrolysis of chlorinated coal

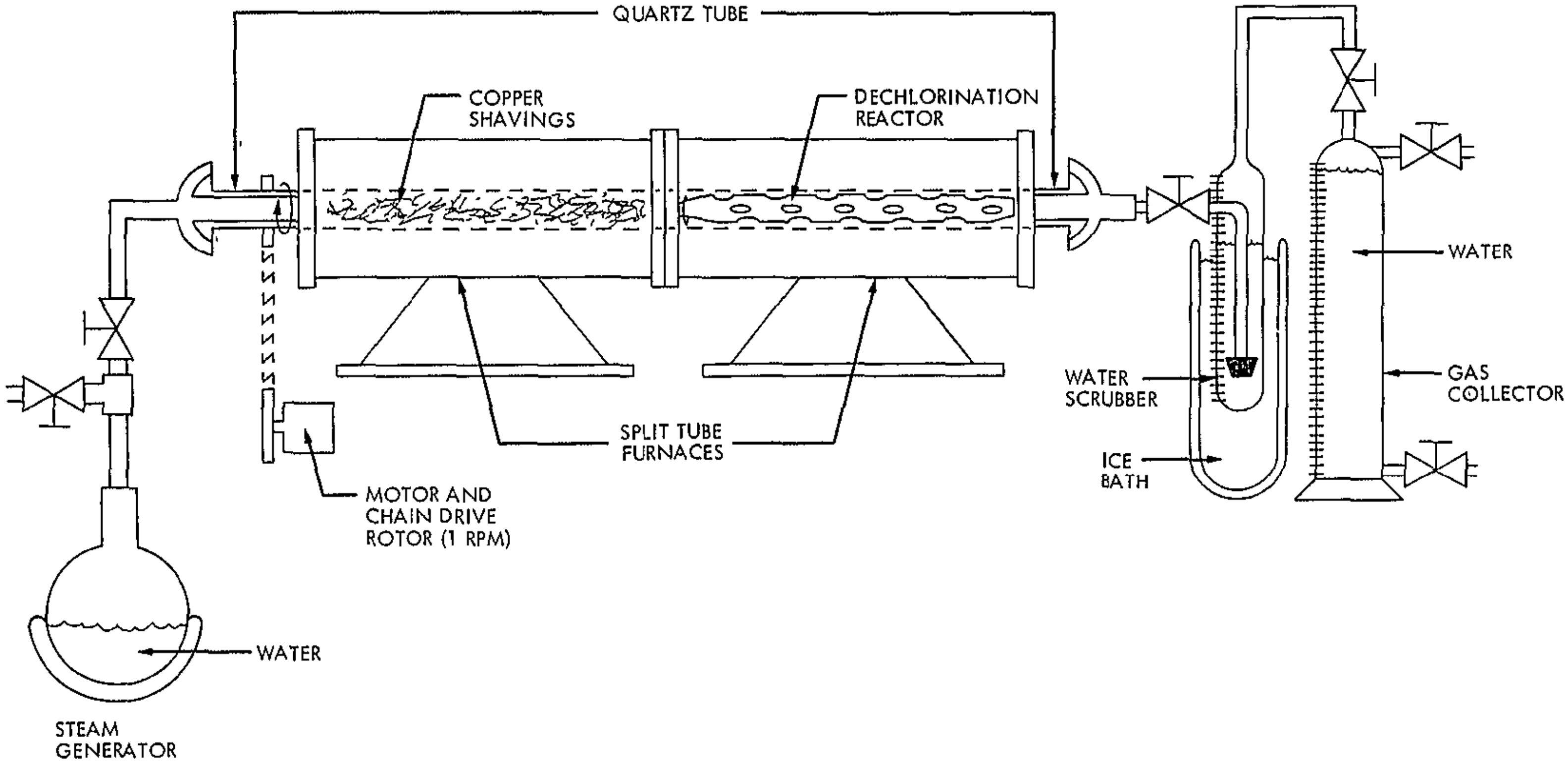

Figure 4. Laboratory equipment for dechlorination of coal

Table 2. Laboratory scale data - coal desulfurization by low temperature chlorination

RUN DATE	COAL (ERDA COAL CODE)	TREATMENT* CHLORINATION TIME (MIN)	TEMP (°C)	CHLORINE (SCFH)	WATER/COAL	SOLVENT	HYDROLYSIS TIME (MIN)	TEMP (°C)	WATER/COAL	DECHLORINATION TIME (MIN)	TEMP (°C)	STEAM (gm/hr)	CO_2 (gm/hr)	RESIDUAL SULFUR ANALYSIS (WT %) ORGANIC	PYRITIC	SULFATE	TOTAL	SULFUR REMOVAL (%) ORGANIC	PYRITIC	TOTAL	RESIDUAL CHLORINE (WT %) DECHLORINATION BEFORE	AFTER	REMARKS
102 6/27/77	PSOC 219 (HVA BIT KY NO 4)	RAW	COAL											1 08	1 40	0 08	2 56				0 03		HEATING VALUE OF RAW COAL = 13,400 BTU/LB (MF BASIS)
		120	74	0 25	0 5/1	METHYL CHLOROFORM	–	–	–	–	–	–	–	0 21	0 73	0 71	1 65	80 6	47 9	35 5			LOW CHLORINE RATE VERY LITTLE CHLORINE COLLECTED IN COLD TRAP
		120	74	0 25	0 5/1		–	–	–	–	–	–	–	0 17	0 87	0 77	1 81	84 3	37 9	29 3	12 41		
		120	74	0 25	0 5/1		120	60	4/1	–	–	–	–	0 46	0 50	0 03	0 99	57 4	64 3	61 3	10 17		
		120	74	0 25	0 5/1		120	60	4/1	60	450	≈70	–	0 38	0 41	<0 01	0 79	64 8	46 8	69 0		0 31	
105-7/19/77	PSOC 219	30	74	0 5	0 5/1		–	–	–	–	–	–	–	0 52	1 05	0 60	2 17	51 9	75 0	15 2	5 36		MODERATE CHLORINE RATE
		30	74	0 5	0 5/1		–	–	–	–	–	–	–	0 81	0 70	0 11	1 62	25 0	50 0	36 7	3 01		
		30	74	0 5	0 5/1		120	60	4/1	60	450	≈70	–	0 71	0 10	0 01	0 82	34 3	92 8	68 0		0 69	CHLORINE COLLECTS IN COLD TRAP AFTER 45 MINUTES OF RUN
		30	74	0 5	0 5/1		120	60	4/1	60	450	≈70	–	0 61	0 12	<0 01	0 73	43 5	91 4	71 5		0 84	
107 7/27/77	PSOC 219	30	74	0 5	0 5/1	"	–	–	–	–	–	–	–	0 87	0 79	0 42	2 08	19 4	43 6	18 8	5 41		
		30	74	0 5	0 5/1	"	120	60	4/1	60	450	≈70	–	0 72	0 11	<0 01	0 83	33 3	92 1	67 6		0 22	
		120	74	0 5	0 5/1	"	–	–	–	–	–	–	–	0 14	0 74	0 78	1 66	87 0	47 1	35 2	20 30		
		120	74	0 5	0 5/1	"	120	60	4/1	–	–	–	–	0 24	0 59	0 10	0 93	77 8	57 9	63 7	11 13		HEATING VALUE OF TREATED COAL = 12,782 BTU/LB (MF BASIS)
		120	74	0 5	0 5/1		120	60	4/1	60	450	≈70	–	0 63	0 22	0 02	0 87	41 7	84 3	66 0		0 48	
		120	74	0 5	0 5/1	"	120	60	4/1	60	450	≈70	–	0 35	0 37	<0 01	0 72	87 6	73 6	71 9		0 31	
108-8/1/77	PSOC 219	60	74	0 5	0 5/1		120	60	4/1	60	450	≈70	–	0 28	0 45	0 15	0 88	74 1	67 9	65 6		0 41	Na < 0 05%
		120	74	0 5	0 5/1	"	120	60	4/1	–	–	–	–	0 59	0 62	0 10	1 31	45 4	65 7	48 8	13 45		Na < 0 05%
		120	74	0 5	0 5/1	"	120	60	4/1	60	450	≈70	–	0 56	0 36	<0 01	0 92	48 1	74 3	64 1		0 33	
103 7/8/77	PSOC 219	30	74	1 0	0 5/1		–	–	–	–	–	–	–	0 42	0 85	0 49	1 76	61 1	39 3	31 3	6 27		HIGH CHLORINE RATE CHLORINE COLLECTS IN COLD TRAP FROM THE BEGINNING
		60	74	1 0	0 5/1	"	–	–	–	–	–	–	–	0 23	0 26	0 60	1 09	78 7	81 4	57 4	13 11		
		60	74	1 0	0 5/1		120	60	4/1	60	450	≈70	–	0 43	0 12	0 19	0 74	60 2	91 4	71 1		1 0	0 55% TOTAL RESIDUAL SULFUR (ASSUMING TOTAL WASTE REMOVAL)
		120	74	1 0	0 5/1		120	60	4/1	–	–	–	–	0 70	0 04	0 18	0 92	35 2	97 1	64 1	19 80		
115 8/26/77	PSOC 219	30	50	0 5	0 5/1		120	80	4/1	60	500	75 110	–	0 68	0 12	<0 01	0 80	37 0	91 4	68 8		< 0 01	LOWER CHLORINATION TEMP OF 50°C
		60	50	0 5	0 5/1		120	80	4/1	60	500	75 110	–	0 62	0 03	<0 01	0 65	42 6	97 9	74 6		0 45	
		120	50	0 5	0 5/1		120	80	4/1	–	–	–	–	0 66	0 26	0 01	0 93	38 9	81 4	63 7	18 25		
		120	50	0 5	0 5/1		120	80	4/1	60	500	75 110	–	0 52	0 06	0 11	0 69	51 8	95 7	73 0		0 50	
116 8/30/77	PSOC 219	30	60	0 5	0 5/1		120	80	4/1	60	500	75 110	-	0 71	0 16	<0 01	0 87	34 3	88 6	66 0			LOWER CHLORINATION TEMP OF 60°C
		60	60	0 5	0 5/1		120	80	4/1	–	–	–	–	0 69	0 31	0 13	1 13	36 1	77 9	55 8	8 64		
		60	60	0 5	0 5/1		120	80	4/1	60	500	75 110	–	0 74	0 06	<0 01	0 80	45 9	95 7	68 7		0 47	
		120	60	0 5	0 5/1		120	80	4/1	–	–	–	–	0 74	0 08	0 03	0 85	45 9	94 3	66 8	22 3		
		120	60	0 5	0 5/1		120	80	4/1	60	490	75 110	–	–	–	–	0 63	–	–	75 4		0 50	

*CHLORINATION CONDITIONS — 500 ml STIRRED FLASK 100 GRAM SAMPLES OF +200 MESH COAL SOLVENT/COAL = 2/1, ATM PRESSURE

HYDROLYSIS CONDITIONS — 1000 ml STIRRED FLASK 1 WASH FOR RETENTION TIMES <60 MINUTES AND 2 WASHES EACH AT THE STATED WATER/COAL AT 120 MINUTES, FILTRATION WITH 1/1 WATER/COAL WATERWASHES, WITH 1 WATERWASH FOR RUNS 101-122 AND 2 WATERWASHES FOR RUNS 123-147

DECHLORINATION CONDITIONS — 1-INCH DIAMETER QUARTZ ROTARY TUBE (RUNS 101 TO 114 AT 1 RPM RUNS 115 TO 148 AT 2 RPM) IN SPLIT TUBE FURNACES (2) STEAM ATMOSPHERE COAL CHARGED AT 2 TO 4 GRAMS/BATCH

(–) INDICATES NO TREATMENT FOR THAT PROCESSING STEP

Table 2. Laboratory scale data - coal desulfurization by low temperature chlorination (continued)

RUN DATE	COAL (ERDA COAL CODE)	TREATMENT* CHLORINATION TIME (MIN)	TEMP (°C)	CHLORINE (SCFH)	WATER/COAL	SOLVENT	HYDROLYSIS TIME (MIN)	TEMP (°C)	WATER/COAL	DECHLORINATION TIME (MIN)	TEMP (°C)	STEAM (gm/hr)	CO_2 (gm/hr)	RESIDUAL SULFUR ANALYSIS (WT %) ORGANIC	PYRITIC	SULFATE	TOTAL	SULFUR REMOVAL (%) ORGANIC	PYRITIC	TOTAL	RESIDUAL CHLORINE (WT %) DECHLORINATION BEFORE	AFTER	REMARKS
117 9/6/77	PSOC 219	30	50	0 5	0 5/1	METHYL CHLOROFORM	120	80	4/1	60	350	75 110	–	0 88	0 59	0 03	1 50	18 5	57 9	41 4		0 89	LOWER CHLORINATION TEMP OF 50°C
		60	50	0 5	0 5/1		120	80	4/1	60	450	75 110	–	0 77	0 23	0 01	1 01	28 7	83 6	60 5		0 60	
		120	50	0 5	0 5/1		120	80	4/1	–	–	–	–	0 49	0 39	0 13	1 01	54 6	82 6	60 5	19 08		
		120	50	0 5	0 5/1	"	120	80	4/1	60	450	75 110	–	0 33	0 56	<0 01	0 89	69 4	60 0	65 2		0 72	
		120	50	0 5	0 5/1		120	80	4/1	60	500	75 110	–	0 66	0 05	<0 01	0 71	38 9	96 4	72 3		0 30	
118 9/9/77	PSOC 219	60	74	0 5	0 5/1		60	80	4/1	–	–	–	–	0 33	0 45	0 18	0 96	69 4	67 9	62 5			
		60	74	0 5	0 5/1		60	80	4/1	20	500	1 0	5 9	0 63	0 28	<0 01	0 91	41 7	80,0	64 4	–	0 84	
		60	74	0 5	0 5/1		60	80	4/1	20	450	1 3	–	0 50	0 40	<0 01	0 90	53 7	71 4	64 8	–	1 11	
		60	74	0 5	0 5/1		60	80	4/1	20	400	100	–	0 51	0 39	<0 01	0 90	52 8	72 1	64 8	–	1 21	
		60	74	0 5	0 5/1		60	80	4/1	25	450	0 65	–	0 59	0 28	<0 01	0 87	45 4	80 0	66 0	–	2 01	
		60	74	0 5	0 5/1		60	80	4/1	25	500	7 0	5 9	0 68	0 33	<0 01	1 01	37 0	76 4	60 5	–	1 35	
		60	74	0 5	0 5/1		60	80	4/1	60	500	11 6	–	0 58	0 14	0 01	0 73	46 3	90 0	71 5	–	0 60	
		60	74	0 5	0 5/1		120	80	4/1	–	–	–	–	0 55	0 52	0 06	1 13	49 1	62 8	55 9	17 90		
		60	74	0 5	0 5/1		120	80	4/1	–	–	–	–	0 63	0 36	0 08	1 07	41 7	74 3	58 2	11 97		
		60	74	0 5	0 5/1		120	80	4/1	60	450	75 110	–	0 61	0 35	0 02	0 98	43 5	75 0	61 7	11 50	0 30	
119 9/13/77	PSOC 219	30	74	0 5	0 7/1		120	80	4/1	30	500	5	5 9	0 76	0 44	0 02	1 22	29 6	68 6	52 3		0 24	WATER/COAL INCREASED TO
		60	74	0 5	0 7/1		120	80	4/1	30	500	3 8	5 9	0 65	0 44	<0 01	1 09	39 8	68 6	57 4		0 21	0 7/1 IN PLACE OF
		120	74	0,5	0 7/1		120	80	4/1	–	–	–	–	0 69	0 39	0 08	1 16	36 1	72 1	54 7	11 4		0 5/1 FOR PREVIOUS RUNS
		120	74	0 5	0 7/1		120	80	4/1	–	–	–	–	0 85	0 42	0 04	1 31	21 3	70 0	48 8	11 5		LOW ORGANIC
		120	74	0 5	0 7/1		120	80	4/1	60	500	7 5,	–	0 65	0 03	<0 01	0 68	39 8	97 9	73 4		0 12	SULFUR REMOVAL
		120	74	0 5	0 7/1		120	80	4/1	60	350	75	–	0 75	0 68	0 02	1 45	30 5	51 4	43 4		1 03	COMPARED TO 0 5/1 WATER/COAL
120 9/16/77	PSOC 219	30	74	0 5	0 3/1		120	80	4/1	60	450	5	–	0 51	0 83	<0 01	1 34	52 8	40 7	47 6		0 22	
		60	74	0 5	0 3/1		60	80	4/1	60	450	121	–	0 66	0 28	<0 01	0 94	38 9	80 0	63 3		0 14	
		120	74	0 5	0 3/1		30	80	4/1	–	–	–	–	0 68	0 28	0 10	1 06	37 0	80 0	58 6	20 50		
		120	74	0 5	0 3/1		120	80	4/1	60	450	105	–	0 68	0 21	<0 01	0 89	37 0	85 0	65 2		0 26	
123 9/23/77	PSOC-219	30	74	0 5	0 7/1		60	80	2/1	–	–	–	–	–	–	–	–	–	–	–	5 1		
		30	74	0 5	0 7/1		60	80	2/1	60	500	1 26	5 9	0 79	0 47	0 01	1 27	26 9	66 4	50 4		0 11	
		30	74	0 5	0 7/1		120	80	2/1	–	–	–	–	–	–	–	–	–	–	–			
		120	74	0 5	0 7/1		60	80	4/1	–	–	–	–	0 70	0 60	0 05	1,35	35 2	57 1	47 3	11 12		
		60	74	0 5	0 7/1		120	80	3/1	–	–	–	–	0 57	0 48	0 06	1 23	47 2	65 7	52 0	9 18		
		60	74	0 5	0 7/1		120	80	3/1	30	500	4	–	0 67	0 52	<0 01	1 19	38 0	62 9	53 5		0 86	
		120	74	0 5	0 7/1		120	80	4/1	60	550	4	–	0 61	0 21	<0 01	0 82	43 5	85 0	68 0		0 17	
		120	74	0 5	0 7/1		120	80	4/1	25	500	1	–	0 62	0 56	<0 01	1 18	42 6	60 0	53 9		1 35	
		120	74	0 5	0 7/1		120	80	4/1	25	500	0 5	5 9	0 85	0 21	<0 01	1 06	21 3	85 0	58 6		0 45	
		120	74	0 5	0 7/1		120	80	4/1	25	550	2	–	0 86	0 32	0 01	1 19	20 4	77 1	53 5		0 74	
		120	74	0 5	0 7/1		120	80	4/1	25	550	1	5 9	0 53	0 67	<0 01	1 20	50 9	52 1	53,1		2 11	
		120	74	0 5	0 7/1		120	80	4/1	25	400	0 7	–	0 67	0 52	<0 01	1 19	38 0	62 9	53 5		2 17	
124 9/27/77	PSOC 219	30	74	0 5	0 3/1		60	80	2/1	–	–	–	–	0 54	0 83	0 13	1 50	50 0	40 7	41 4	4 74		
		30	74	0 5	0 3/1		120	80	2/1	60	450	0 4	–	0 73	0 53	0 04	1 30	32 4	62 1	49 2		1 69	
		60	74	0 5	0 3/1		30	80	3/1	–	–	–	–	0 44	0 61	0 11	1 16	59 3	56 4	54 7	9 07		
		60	74	0 5	0 3/1		60	80	3/1	–	–	–	–	0 42	0 69	0 10	1 22	61 1	50 7	52 3	8 66		
		60	74	0 5	0 3/1		120	80	3/1	60	500	20	–	0,54	0 27	0 01	0 82	50 0	80 7	68 0		0 28	
		120	74	0 5	0 3/1		5	100	4/1	–	–	–	–	–	–	–	–	–	–	–			
		120	74	0 5	0 3/1		10	100	4/1	–	–	–	–	0 48	0 64	0 18	1 30	55 6	54 3	49 2	18 30		
		120	74	0 5	0 3/1		15	100	4/1	–	–	–	–	–	–	–	–	–	–	–			
		120	74	0 5	0 3/1		20	100	4/1	–	–	–	–	0 73	0 39	0 11	1 23	32 4	72 1	52 0	16 36		
		120	74	0 5	0 3/1		25	100	4/1	–	–	–	–	–	–	–	–	–	–	–			

*CHLORINATION CONDITIONS 500 ml STIRRED FLASK 100 GRAM SAMPLES OF+200 MESH COAL SOLVENT/COAL = 2/1 ATM PRESSURE

HYDROLYSIS CONDITIONS 1000 ml STIRRED FLASK 1 WASH FOR RETENTION TIMES <60 MINUTES AND 2 WASHES EACH AT THE STATED WATER/COAL AT 120 MINUTES FILTRATION WITH 1/1 WATER/COAL WATERWASHES WITH 1 WATERWASH FOR RUNS 101–122 AND 2 WATERWASHES FOR RUNS 123–147

DECHLORINATION CONDITIONS 1–INCH DIAMETER QUARTZ ROTARY TUBE (RUNS 101 TO 114 AT 1 RPM, RUNS 115 TO 148 AT 2 RPM) IN SPLIT TUBE FURNACES (2) STEAM ATMOSPHERE COAL CHARGED AT 2 TO 4 GRAMS/BATCH

(–) INDICATES NO TREATMENT FOR THAT PROCESSING STEP

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Table 2. Laboratory scale data - coal desulfurization by low temperature chlorination (continued)

RUN DATE	COAL (ERDA COAL CODE)	TREATMENT* CHLORINATION TIME (MIN)	TEMP (°C)	CHLORINE (SCFH)	WATER/COAL	SOLVENT	HYDROLYSIS TIME (MIN)	TEMP (°C)	WATER/COAL	DECHLORINATION TIME (MIN)	TEMP (°C)	STEAM (gm/hr)	CO_2 (gm/hr)	RESIDUAL SULFUR ANALYSIS (WT %) ORGANIC	PYRITIC	SULFATE	TOTAL	SULFUR REMOVAL (%) ORGANIC	PYRITIC	TOTAL	RESIDUAL CHLORINE (WT %) DECHLORINATION BEFORE	AFTER	REMARKS
124 9/27/77	PSOC 219	120	74	0 5	0 3/1	METHYL CHLOROFORM	30	100	4/1	–	–	–	–	–	–	–	–	–	–	–			
		120	74	0 5	0 3/1	'	40	100	4/1	–	–	–	–	0 82	0 44	0 07	1 33	24 1	68 6	48 0	13 81		
		120	74	0 5	0 3/1	'	50	100	4/1	–	–	–	–	–	–	–	–	–	–	–			
		120	74	0 5	0 3/1		60	100	4/1	–	–	–	–	0 78	0 37	0 07	1 22	27 8	73 6	52 3			
		120	74	0 5	0 3/1		60	100	4/1	60	500	0 5	–	0 69	0 12	<0 01	0 81	36 1	91 4	68 4	1 63		
		120	74	0 5	0 3/1	'	60	100	4/1	40	500	0 4	–	0 80	<0 01	<0 01	0 80	25 9	100 0	68 8		1 34	
125 9/28/77	PSOC 219	10	74	0 5	0 5/1	"	30	60	2/1	–	–	–	–	0 69	0 94	0 07	1 70	36 1	32 9	33 2	4 78		
		10	74	0 5	0 5/1	"	60	60	2/1	15	500	0 4	–	0 88	0 64	<0 01	1 52	18 5	54 3	40 6			
		20	74	0 5	0 5/1	"	30	100	2/1	–	–	–	–	0 71	0 86	0 10	1 67	34 2	38 6	34 8	4 86		
		20	74	0 5	0 5/1	"	60	100	2/1	–	–	–	–	0 58	0 89	0 12	1 59	46 3	36 4	37 9	5 01		
		20	74	0 5	0 5/1	"	60	100	2/1	30	500	1 6	5 9	0 79	0 44	0 02	1 25	26 9	68 6	51 2		0 12	
		30	74	0 5	0 5/1	'	20	80	2/1	–	–	–	–	0 86	0 73	0 06	1 65	20 4	47 9	35 5	6 95	–	
		30	74	0 5	0 5/1	"	30	80	2/1	–	–	–	–	1 04	0 42	0 06	1 52	3 7	70 0	40 6	7 26		
		30	74	0 5	0 5/1	"	45	80	2/1	–	–	–	–	0 87	0 65	0 04	1 56	19 4	37 9	39 1	8 15		
		30	74	0 5	0 5/1		60	80	2/1	–	–	–	–	0 80	0 68	0 04	1 53	25 9	51 4	40 2	9 65		
		30	74	0 5	0 5/1	'	60	80	2/1	15	450	1	5 9	–	–	–	–	–	–	–			
		30	74	0 5	0 5/1	'	60	80	2/1	20	450	1	5 9	0 75	0 36	<0 01	1 12	30 5	74 3	56 2		0 77	
		30	74	0 5	0 5/1	'	60	80	2/1	15	500	2	–	0 71	0 37	<0 01	1 08	34 2	73 6	57 8		0 54	
		30	74	0 5	0 5/1	'	60	80	2/1	20	500	1 5	–	0 68	0 42	<0 01	1 10	37 0	70 0	57 0		1 16	
		30	74	0 5	0 5/1	'	60	80	2/1	15	500	0 4	–	0 73	0 39	<0 01	1 12	32 4	72 1	56 2		0 65	
		30	74	0 5	0 5/1	'	60	80	2/1	20	500	0 6	–	0 75	0 39	<0 01	1 14	30 5	72 1	55 5		1 10	
		30	74	0 5	0 5/1		60	80	2/1	15	450	0 76	5 9	0 73	0 46	0 01	1 20	32 4	67 1	53 1		1 22	
138 10/17/77	PSOC 219	60	74	0 5	0 5/1	'	60	80	4/1	–	–	–	–	0 69	0 46	0 08	1 23	36 1	67 1	52 0	9 86		
		60	74	0 5	0 5/1	'	60	80	4/1	60	500	9	–	0 58	0 23	<0 01	0 81	46 3	83 6	68 4		0 43	
		60	74	0 5	0 5/1	'	60	80	4/1	30	500	5 8	5 9	0 53	0 31	0 01	0 85	50 9	77 9	66 8		0 45	
		60	74	0 5	0 5/1	'	60	80	4/1	30	500	10	–	0 74	0 23	0 03	1 00	31 5	83 6	60 9		0 17	
136 10/13/77	PSOC 219	30	74	0 5	0 5/1	"	60	80	4/1	30	500	1	5 9	1 39	0 31	<0 01	1 70	–	77 9	33 6		0 08	
		60	74	0 5	0 5/1	"	60	80	4/1	–	–	–	–	0 49	1 24	0 03	1 76	54 6	11 4	31 2	4 59		
		60	74	0 5	0 5/1		60	80	4/1	30	500	3 5	–	0 33	1 21	0 01	1 55	69 4	13 6	39 5		0 01	
139 10/18/77	PSOC 219	30	74	0 5	0 5/1		60	80	4/1	30	500	5 5	5 9	0 89	0 38	<0 01	1 27	17 6	72 9	50 4		0 17	
		60	74	0 5	0 5/1		60	80	4/1	30	500	6 0	5 9	0 51	0 40	0 02	0 93	52 8	71 4	63 7		0 31	
		60	74	0 5	0 5/1		60	80	4/1	30	500	5 2	5 9	0 66	0 37	0 02	1 05	65 7	73 6	69 0		0 77	
141 10/21/77	PSOC 219	30	74	0 5	0 5/1	'	30	80	4/1	30	500	1	5 9	0 96	0 34	<0 01	1 30	11 1	75 7	49 2		0 10	COAL PARTICLE
		60	74	0 5	0 5/1	"	60	80	4/1	–	–	–	–	0 90	0 46	0 05	1 41	56 5	67 1	44 9	9 50		SIZE FOR RUN 141
		60	74	0 5	0 5/1		60	80	4/1	30	500	5 5	5 9	0 70	0 26	<0 01	0 96	35 2	81 4	62 5		0 06	WAS -70 TO +120
143 10/24/77	PSOC 219	30	74	1 0	0 5/1		60	80	4/1	30	500	5 5	5 9	0 54	0 28	0 02	0 84	50 0	80 0	67 2		0 57	CHLORINE ADDI
		60	74	1 0/0 25	0 5/1	'	60	80	4/1	30	500	5 5	5 9	0 44	0 31	<0 01	0 75	59 2	77 9	70 7		0 57	TION RATE 1 0
		60	74	1 0/0 25	0 5/1	"	60	80	4/1	–	–	–	–	0 32	0 68	0 25	1 25	70 4	51 4	51 2	11 59		SCFH FOR FIRST 30 MIN AND 0 25 FOR LAST 30 MIN

*CHLORINATION CONDITIONS — 500 ml STIRRED FLASK 100 GRAM SAMPLES OF +200 MESH COAL SOLVENT/COAL = 2/1 ATM PRESSURE

HYDROLYSIS CONDITIONS — 1000 ml STIRRED FLASK, 1 WASH FOR RETENTION TIMES <60 MINUTES AND 2 WASHES EACH AT THE STATED WATER/COAL AT 120 MINUTES FILTRATION WITH 1/1 WATER/COAL WATERWASHES WITH 1 WATERWASH FOR RUNS 101–122 AND 2 WATERWASHES FOR RUNS 123–147

DECHLORINATION CONDITIONS — 1 INCH DIAMETER QUARTZ ROTARY TUBE (RUNS 101 TO 114 AT 1 RPM RUNS 115 TO 148 AT 2 RPM) IN SPLIT TUBE FURNACES (2) STEAM ATMOSPHERE COAL CHARGED AT 2 TO 4 GRAMS/BATCH

(–) INDICATES NO TREATMENT FOR THAT PROCESSING STEP

Table 2. Laboratory scale data - coal desulfurization by low temperature chlorination (continued)

RUN DATE	COAL (ERDA COAL CODE)	TREATMENT* CHLORINATION TIME (MIN)	TEMP (°C)	CHLORINE (SCFH)	WATER/ COAL	SOLVENT	HYDROLYSIS TIME (MIN)	TEMP (°C)	WATER/ COAL	DECHLORINATION TIME (MIN)	TEMP (°C)	STEAM (gm/hr)	CO_2 (gm/hr)	RESIDUAL SULFUR ANALYSIS (WT %) ORGANIC	PYRITIC	SULFATE	TOTAL	SULFUR REMOVAL (%) ORGANIC	PYRITIC	TOTAL	RESIDUAL CHLORINE (WT %) DECHLORINATION BEFORE	AFTER	REMARKS
144 10/24/77	PSOC 219	60	85	0 5	0 5/1	METHYL CHLOROFORM	60	80	4/1	30	500	1 20	–	0 74	0 23	0 02	0 99	31 5	83 6	61 3		0 86	HIGHER CHLORI NATION TEMP
		60	85	0 5	0 5/1	'	60	80	4/1	–	–	–	–	0 56	0 46	0 22	1 24	48 1	67 1	51 6	11 35		
145 10/25/77	PSOC 219	60	74	0 5	0 5/1		60	80	4/1	30	500	0 6	5 9	0 68	0 04	<0 01	0 72	37 0	97 1	71 9		0 28	COAL SAMPLE FOR
		60	74	0 5	0 5/1		60	80	4/1	30	500	2 5	–	0 87	0 06	<0 01	0 93	19 4	95 7	63 7		0 89	RUN 145 PRE
		60	74	0 5	0 5/1	"	60	80	4/1	–	–	–	–	0 68	0 08	<0 01	0 76	37 0	94 3	70 3	8 72		VIOUSLY DECHLORINATED
146 10/28/77	PSOC 219	60	74	0 5	0 5/1	'	60	80	4/1	30	500	4 75	5 9	0 64	0 20	0 01	0 85	40 7	85 7	66 8		0 36	SOLVENT/COAL =
		60	74	0 5	0 5/1	'	60	80	4/1	–	–	–	–	0 60	0 29	0 06	0 95	44 4	79 3	62 9	15 97		4/1 FOR RUN 146
147 10/28/77	PSOC 219	60	74	0 5	0 5/1	'	60	80	4/1	–	–	–	–	0 72	0 78	0 11	1 61	33 3	44 3	37 1	7 34		
122 9/21/77	PSOC 219	15	100	0 5	0 5/1	TETRACHLO ROETHYLENE	60	80	4/1	60	350	2	–	0 66	0 77	0 01	1 44	38 9	45 0	43 7		0 44	TETRACHLORO ETHYLENE
		30	100	0 5	0 5/1	'	60	80	4/1	60	550	4	–	1 00	0 14	<0 01	1 14	7 4	90 0	55 5		0 31	SOLVENT
		60	100	0,5	0 5/1	'	60	80	4/1	–	–	–	–	0 64	0 77	0 08	1 49	40 7	45 0	41 8	23 06		
		60	100	0 5	0 5/1	'	60	80	4/1	60	550	4	–	0 82	0 08	0 02	0 92	24 1	94 3	64 1		0 39	
126 9/30/77	PSOC 219	15	74	0 5	0 5/1	"	60	60	2/1	60	500	0 3	–	0 99	0 34	0 02	1 35	8 3	75 7	47 3		1 29	
		30	74	0 5	0 5/1	'	30	60	2/1	–	–	–	–	0 65	0 60	0 13	1 38	39 8	57 1	46 1			
		30	74	0 5	0 5/1		60	60	2/1	–	–	–	–	–	–	–	–	–	–	–			
		30	74	0 5	0 5/1	'	60	60	2/1	60	500	7 5	–	0 58	0 19	<0 01	0 77	46 3	86 4	69 9		0 41	
		60	74	0 5	0 5/1		30	100	2/1	–	–	–	–	0 82	0 57	0 13	1 52	24 1	59 3	40 6			
		60	74	0 5	0 5/1	'	60	100	2/1	–	–	–	–	0 71	0 41	0 09	1 21	34 2	70 7	52 7	25 07		
		60	74	0 5	0 5/1		60	100	2/1	60	500	0 25	–	0 77	0 22	<0 01	0 98	28 7	84 3	61 7		1 22	
		20	74	0 5	0 5/1		30	80	2/1	–	–	–	–	0 72	0 53	0 25	1 50	33 3	62 1	41 4	24 41		
		20	74	0 5	0 5/1	'	60	80	2/1	60	500	1	–	0 82	0 07	<0 01	0 89	24 1	95 0	65 2		1 14	
132 10/7/77	PSOC 219	60	74	0 5	0 3/1		30	80	4/1	–	–	–	–	0 46	0 60	0 15	1 21	57 4	57 1	52 7	6 40		
		60	74	0 5	0 3/1		60	80	4/1	30	500	4	–	0 51	0 20	<0 01	0 71	52 8	85 7	72 3		0 80	
		120	74	0 5	0 3/1		60	80	4/1	30	500	5	5,9	0 73	0 35	<0 01	1 08	32 4	75 0	57 8		0 64	
		120	74	0 5	0 3/1		60	80	4/1	–	–	–	–	0 62	0 58	0 09	1 29	42 6	58 6	49 6	17 10		
134 10/10/77	PSOC 219	30	74	0 5	0 7/1	'	60	80	4/1	–	–	–	–	0 70	0 75	0 07	1 52	35 2	46 4	40 6	11 2		
		60	74	0 5	0 7/1	'	60	80	4/1	–	–	–	–	0 40	0 80	<0 01	1 20	63 0	42 9	53 1	14 34		
121 9/20/77	PSOC 219	30	74	0 5	0 7/1	CARBON TETRA- CHLORIDE	60	80	4/1	60	500	4	–	0 72	0 31	<0 01	1 03	33 3	77 8	59 8		0 21	CARBON TETRA- CHLORIDE SOLVENT
		60	74	0 5	0 7/1		60	80	4/1	–	–	–	–	0 56	0 50	0 09	1 15	48 1	64 3	55 1	11 91		
		60	74	0 5	0 7/1		60	80	4/1	60	550	3	–	0 65	0 08	0 02	0 75	39 8	94 3	70 7		0 15	
128 10/4/77	PSOC 219	60	74	0 5	0 5/1		30	80	2/1	–	–	–	–	0 76	0 57	0 12	1 45	29 6	59 3	43 4	5 78		
		120	74	0 5	0 5/1	'	60	60	2/1	–	–	–	–	0 66	0 79	0 05	1 50	38 9	43 6	41 4	5 38		
		120	74	0 5	0 5/1	'	60	60	2/1	60	500	8 5	-	0 86	0 35	<0 01	1 21	20 4	75 0	52 7		0 68	

*CHLORINATION CONDITIONS 500 ml STIRRED FLASK, 100 GRAM SAMPLES OF +200 MESH COAL SOLVENT/COAL = 2/1 ATM PRESSURE

HYDROLYSIS CONDITIONS 1000 ml STIRRED FLASK 1 WASH FOR RETENTION TIMES <60 MINUTES AND 2 WASHES EACH AT THE STATED WATER/COAL AT 120 MINUTES FILTRATION WITH 1/1 WATER/COAL WATERWASHES WITH 1 WATERWASH FOR RUNS 101–122 AND 2 WATERWASHES FOR RUNS 123–147

DECHLORINATION CONDITIONS 1-INCH DIAMETER QUARTZ ROTARY TUBE (RUNS 101 TO 114 AT 1 RPM RUNS 115 TO 148 AT 2 RPM) IN SPLIT TUBE FURNACES (2), STEAM ATMOSPHERE COAL CHARGED AT 2 TO 4 GRAMS/BATCH

(–) INDICATES NO TREATMENT FOR THAT PROCESSING STEP

Table 2. Laboratory scale data - coal desulfurization by low temperature chlorination (continued)

RUN DATE	COAL (ERDA COAL CODE)	TREATMENT* CHLORINATION TIME (MIN)	CHLORINATION TEMP (°C)	CHLORINATION CHLORINE (SCFH)	CHLORINATION WATER/COAL	SOLVENT	HYDROLYSIS TIME (MIN)	HYDROLYSIS TEMP (°C)	HYDROLYSIS WATER/COAL	DECHLORINATION TIME (MIN)	DECHLORINATION TEMP (°C)	DECHLORINATION STEAM (gm/hr)	DECHLORINATION CO_2 (gm/hr)	RESIDUAL SULFUR ANALYSIS (WT %) ORGANIC	PYRITIC	SULFATE	TOTAL	SULFUR REMOVAL (%) ORGANIC	PYRITIC	TOTAL	RESIDUAL CHLORINE (WT %) DECHLORINATION BEFORE	AFTER	REMARKS
130 10/5/77	PSOC 219	120	74	0 5	0 3/1	CARBON TETRA-CHLORIDE	60	80	4/1	–	–	–	–	0 58	0 56	0 12	1 26	46 3	60 0	50 5	17 63		
		120	74	0 5	0 3/1	"	30	80	4/1	30	500	2	5 9	0 47	0 30	0 03	0 80	56 5	78 6	68 7		1 25	
	PSOC 213 (HVB BIT	RAW	COAL											1 86	1 89	0 07	3 82				0 05		
101 6/22/77	KY NO 9)	120	74	0 125	0 5/1	METHYL CHLOROFORM	120	60	4/1	75	400	≈70	–	0 53	1 65	0 01	2 19	71 5	12 7	43 0	4 58	0 57	METHYL CHLORO FORM SOLVENT LOW CHLORINE RATE
	PSOC 108 (HVB BIT	RAW	COAL											1 07	2 06	0 00	3 13				0 06		1 5% MOISTURE
104 7/1/77	PITTS-BURGH	60	74	0 5	0 5/1	METHYL CHLOROFORM	120	60	4/1	–	–	–	–	0 18	0 96	0 13	1 27	83 2	53 4	59 4	8 00		
	WASH	60	74	0,5	0 5/1	"	120	60	4/1	60	450	≈70	–	0 45	0 26	0 01	0 71	58 0	87 4	77 3			
	PA)	120	74	0 5	0 5/1		120	60	4/1	–	–	–	–	0 90	0 28	< 0 01	1 18	15 9	86 4	62 3	13 57		
		120	74	0 5	0 5/1		120	60	4/1	60	450	≈70	–	0 78	0 28	< 0 01	1 01	27 1	88 8	67 7		0 39	
106 7/22/77		30	74	0 5	0 5/1		120	60	4/1	–	–	–	–	0 60	1 32	0 40	2 32	43 9	35 9	25 9	9 24		
		30	74	0 5	0 5/1		120	60	4/1	–	–	–	–	0 82	1 15	0 02	1 99	23 4	44 2	36 4	6 12		
		60	74	0 5	0 5/1		120	60	4/1	–	–	–	–	0 75	0 24	0 40	1 39	29 9	88 3	55 6	12 28		
		60	74	0 5	0 5/1		120	60	4/1	–	–	–	–	0 81	0 46	0 05	1 32	24 3	77 7	57 8	9 46		
		60	74	0 5	0 5/1	"	120	60	4/1	60	450	≈70	–	0 56	0 17	<0 01	0 73	47 7	91 7	76 6		0 88	
		60	74	0 5	0 5/1	"	120	60	4/1	60	450	≈70	–	0 61	0 09	<0 01	0 70	43 0	95 6	77 6		0 97	
		120	74	0 5	0 5/1	"	120	60	4/1	–	–	–	–	0 88	0 50	0 07	1 45	17 8	75 7	53 7	14 66		
	PSOC 190 (HVA BIT	RAW	COAL											1 90	1 05	0 10	3 05				0 04		
112 8/19/77	ILL NO 6 KNOX	60	74	0 5	0 5/1	METHYL CHLOROFORM	120	80	4/1	–	–	–	–	1 57	0 13	0 10	1 80	17 4	87 6	41 0	9 1		
	ILL)	60	74	0 5	0 5/1	"	120	80	4/1	60	500	75 110	–	1 34	0 03	0 03	1 40	29 5	97 1	54 1		0 08	
109 8/8/77		60	74	0 5	0 5/1	"	120	60	4/1	60	470	≈70	–	–	–	–	1 36	–	–	55 4		0 06	
		60	74	0 5	0 5/1	"	120	60	4/1	–	–	–	–	1 61	0 14	0 18	1 93	15 3	86 7	36 7	6 14		
		120	74	0 5	0 5/1	"	120	60	4/1	60	500	90	–	1 26	0 02	<0 01	1 28	33 7	98 1	58 0		0 13	
		120	74	0 5	0 5/1	"	–	–	–	–	–	–	–	1 27	0 09	0 48	1 84	33 2	91 4	39 7	17 64		
		120	74	0 5	0 5/1	"	120	60	4/1	–	–	–	–	1 50	0 08	0 02	1 60	21 1	92 4	47 5	12 57		
	PSOC 276 (HVA BIT	RAW	COAL											2 24	2 07	0 84	5 15				0 14		
114-8/23/77	OHIO NO, 8	60	74	0 5	0 5/1	METHYL CHLOROFORM	120	80	4/1	–	–	–	–	0 59	0 74	0 39	1 72	73 7	64 3	66 6	10 74		
	HARRISON	60	74	0 5	0 5/1		120	80	4/1	60	500	75-110	–	0 88	0 05	<0 01	0 93	60 7	97 6	82 0		0 54	
	OHIO)	120	74	0 5	0 5/1	"	120	80	4/1	60	500	75 110	–	1 03	0 09	<0 01	1 12	54 0	95 6	78 2			

*CHLORINATION CONDITIONS: 500 ml STIRRED FLASK, 100 GRAM SAMPLES OF +200 MESH COAL SOLVENT/COAL = 2/1 ATM PRESSURE

HYDROLYSIS CONDITIONS 1000 ml STIRRED FLASK 1 WASH FOR RETENTION TIMES <60 MINUTES AND 2 WASHES EACH AT THE STATED WATER/COAL AT 120 MINUTES, FILTRATION WITH 1/1 WATER/COAL WATERWASHES WITH 1 WATERWASH FOR RUNS 101-122 AND 2 WATERWASHES FOR RUNS 123-147

DECHLORINATION CONDITIONS 1-INCH DIAMETER QUARTZ ROTARY TUBE (RUNS 101 TO 114 AT 1 RPM, RUNS 115 TO 148 AT 2 RPM) IN SPLIT TUBE FURNACES (2) STEAM ATMOSPHERE COAL CHARGED AT 2 TO 4 GRAMS/BATCH

(–) INDICATES NO TREATMENT FOR THAT PROCESSING STEP

Table 2. Laboratory scale data - coal desulfurization by low temperature chlorination (continued)

RUN DATE	COAL (ERDA COAL CODE)	TREATMENT* CHLORINATION TIME (MIN)	CHLORINATION TEMP (C)	CHLORINATION CHLORINE (SCFH)	CHLORINATION WATER/COAL	SOLVENT	HYDROLYSIS TIME (MIN)	HYDROLYSIS TEMP (C)	HYDROLYSIS WATER/COAL	DECHLORINATION TIME (MIN)	DECHLORINATION TEMP (C)	DECHLORINATION STEAM (gm/hr)	DECHLORINATION CO_2 (gm/hr)	RESIDUAL SULFUR ANALYSIS (WT %) ORGANIC	PYRITIC	SULFATE	TOTAL	SULFUR REMOVAL (%) ORGANIC	PYRITIC	TOTAL	RESIDUAL CHLORINE (WT %) DECHLORINATION BEFORE	AFTER	REMARKS
111 8/16/77	PSOC 276	120	74	0 5	0 5/1	METHYL CHLOROFORM	120	80	4/1	60	500	75 110	–	0 87	0 02	<0 01	0 89	61 2	99 0	82 7		0 17	
		120	74	0 5	0 5/1		120	80	4/1	–	–	–	–	0 85	0 24	0 54	1 63	62 0	88 4	68 3	14 81		
		120	74	0 5	0 5/1		120	80	4/1	–	–	–	–	1 16	0 08	0 22	1 46	48 2	96 1	71 6	13 28		
110 8/10/77	PSOC 276	120	74	0 5	0 5/1		–	–	–	–	–	–	–	0 79	0 53	1 33	2 65	64 7	74 4	48 5	22 44		
		120	74	0 5	0 5/1		120	60	4/1	–	–	–	–	1 09	0 35	0 02	1 46	51 3	83 1	71 6	15 77		
		120	74	0 5	0 5/1	'	120	60	4/1	60	500	75 110	–	1 03	0 04	<0 01	1 07	54 0	98 1	79 2		0 28	
	PSOC 342 (HVA BIT	RAW	COAL											1 39	5 01	0 15	6 55						
142 10/21/77	CLARION	60	74	0 5	0 5/1	METHYL CHLOROFORM	60	80	4/1	30	500	1 0	5 9	1 60	1 69	0 04	3 33	–	66 3	49 2		0 93	
	JEFFERSON PA)	60	74	0 5	0 5/1		60	80	4/1	30	500	10	5 9	1 11	2 00	0 03	3 14	20 1	60 1	52 1			
127 10/3/77		120	74	0 5	0 5/1		120	80	4/1	–	–	–	–	1 29	2 02	0 06	3 37	7 2	59 7	48 5	12 83		
		120	74	0 5	0 5/1		120	80	4/1	60	500	4 8	–	1 82	0 88	<0 01	2 70	–	82 4	58 8		0 15	
	PSOC 097 (SUBBIT A	RAW	COAL											0 84	0 38	0 01	1 23						
129 10/5/77	SEAM 80 CARBON	30	74	0 5	0 5/1	METHYL CHLOROFORM	120	80	4/1	30	500	0 4	5 9	0 70	0 31	0 05	1 06	16 7	18 4	13 8		0 28	
	WYO)	60	74	0 5	0 5/1		120	80	4/1	60	500	1 0	5 9	0 74	0 05	0 02	0 81	11 9	86 8	34 1		0 13	
		120	74	0 5	0 5/1	'	120	80	4/1	–	–	–	–	0 84	0 29	0 09	1 22	0 0	23 7	1 0			
		120	74	0 5	0 5/1		120	80	4/1	60	550	2 4	5 9	0 75	0 09	0 04	0 88	10 7	76 3	28 5		0 22	
	PSOC 026 (HVC BIT	RAW	COAL											2 08	4 23	0 35	6 66						
131 10/7/77	ILL NO 6 SALINE	30	74	0 5	0 5/1	METHYL CHLOROFORM	120	80	4/1	30	500	3 3	–	1 30	0 89	0 02	2 21	37 5	79 0	66 8		0 20	
	ILLINOIS)	60	74	0 5	0 5/1		120	80	4/1	–	–	–	–	1 31	0 66	0 12	2 09	37 0	84 4	68 6	8 46		
		60	74	0 5	0 5/1		120	80	4/1	30	500	5 0	–	1 20	0 45	0 01	1 66	42 3	89 4	75 1		0 42	
	PSOC 086 (LIGNITE,	RAW	COAL											0 63	0 52	0 03	1 22				0 00		
135 10/12/77	ZAP MERCER N	30	74	0 5	0 5/1	METHYL CHLOROFORM	60	80	4/1	30	500	2 2	–	0 35	0 23	0 17	0 75	44 4	58 9	38 5		0 33	
	DAKOTA)	60	74	0 5	0 5/1		60	80	4/1	–	–	–	–	0.39	0 44	0 10	0 93	38 1	21 4	20 5	8 00		
		60	74	0 5	0 5/1	'	60	80	4/1	30	500	4 0	–	0 24	0 27	0 02	0 53	61 9	51 8	56 6			
	PSOC 240 A 1	RAW	COAL											1 75	1 60	0 01	3 36				0 02		
133 10/10/77	(SUBBIT B BIG D LEWIS WASH)	120	74	0 5	0 5/1	METHYL CHLOROFORM	60	80	4/1	30	500	3 5	–	0 49	0 68	0 05	1 22	72 0	57 5	63 7	0 26		

CHLORINATION CONDITIONS 500 ml STIRRED FLASK 100 GRAM SAMPLES OF +200 MESH COAL, SOLVENT/COAL = 2/1 ATM PRESSURE

HYDROLYSIS CONDITIONS 1000 ml STIRRED FLASK 1 WASH FOR RETENTION TIMES <60 MINUTES AND 2 WASHES EACH AT THE STATED WATER/COAL AT 120 MINUTES, FILTRATION WITH 1/1 WATER/COAL WATERWASHES WITH 1 WATERWASH FOR RUNS 101–122 AND 2 WATERWASHES FOR RUNS 123–147

DECHLORINATION CONDITIONS 1–INCH DIAMETER QUARTZ ROTARY TUBE (RUNS 101 TO 114 AT 1 RPM, RUNS 115 TO 148 AT 2 RPM) IN SPLIT TUBE FURNACES (2), STEAM ATMOSPHERE COAL CHARGED AT 2 TO 4 GRAMS/BATCH

(–) INDICATES NO TREATMENT FOR THAT PROCESSING STEP

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Table 2. Laboratory scale data - coal desulfurization by low temperature chlorination (continued)

RUN DATE	COAL (ERDA COAL CODE)	TREATMENT* CHLORINATION TIME (MIN)	TEMP (°C)	CHLORINE (SCFH)	WATER/ COAL	SOLVENT	HYDROLYSIS TIME (MIN)	TEMP (°C)	WATER/ COAL	DECHLORINATION TIME (MIN)	TEMP (°C)	STEAM (gm/hr)	CO_2 (gm/hr)	RESIDUAL SULFUR ANALYSIS (WT %) ORGANIC	PYRITIC	SULFATE	TOTAL	SULFUR REMOVAL (%) ORGANIC	PYRITIC	TOTAL	RESIDUAL CHLORINE (WT %) DECHLORINATION BEFORE	AFTER	REMARKS
	PHS 398	RAW	COAL											0 46	2 26	0 29	3 01					0 10	THE SULFUR FORM ANALYSIS OF RAW
137 10/14/77	RAW HEAD 3A UPPER FREEPORT SOMMERSET PA	30	74	0 5	0 5/1	METHYL CHLOROFORM	60	80	4/1	30	500	6 1	5 9	0 69	0 62	0 03	1 34	–	72 6	55 5		0 11	COAL SUPPLIED BY PITTSBURGH
		60	74	0 5	0 5/1		60	80	4/1	–	–	–	–	0 88	0 34	0 11	1 33	–	85 0	55 8	8 45		BUREAU OF MINES
		60	74	0 5	0 5/1		60	80	4/1	30	500	3 5	–	0 57	0 23	0 02	0 82	–	89 8	72 8		0 62	IS 0 31% ORGANIC
140 10/19/77		30	74	0 5	0 5/1		60	80	4/1	30	500	5 5	5 9	0 60	0 62	0 01	1 23	–	72 6	59 1		0 16	3 69% PYRITIC,
		60	74	0 5	0 5/1		60	80	4/1	–	–	–	–	0 55	0 11	0 05	0 71	–	95 1	76 4	8 24		0 01% SULFATE
		60	74	0 5	0 5/1	"	60	80	4/1	30	500	1 5	–	0 56	0 09	<0 01	0 65	–	96 0	78 4		0 77	4 01% TOTAL
	PHS 513	RAW	COAL											1 76	<0 20	<0 20	1 76				0 27		PHYSICALLY CLEANED HIGH ORGANIC COAL
48 11/21/77	BIT MINE NO 513 UPPER CLARION BUTLER PA	30	74	0 5	0 5/1	METHYL CHLOROFORM	60	80	4/1	30	500	10	–	1 27	<0 20	0 20	1 27	27 8	–	27 8		0 44	SULFUR AND RESIDUAL
		60	74	0 5	0 5/1		60	80	4/1	30	500	10	–	1 16	<0 20	<0 20	1 16	34 1	–	34 1		0 90	CHLORINE
		120	74	0 5	0 5/1	"	60	80	4/1	30	500	10	–	1 28	<0 20	<0 20	1 28	27 3	–	27 3		1 18	ANALYSIS PER FORMED BY JPL

*CHLORINATION CONDITIONS 500 ml STIRRED FLASK 100 GRAM SAMPLES OF +200 MESH COAL SOLVENT/COAL = 2/1 ATM PRESSURE

HYDROLYSIS CONDITIONS 1000 ml STIRRED FLASK, 1 WASH FOR RETENTION TIMES <60 MINUTES AND 2 WASHES EACH AT THE STATED WATER/COAL AT 120 MINUTES FILTRATION WITH 1/1 WATER/COAL WATERWASHES, WITH 1 WATERWASH FOR RUNS 101-122 AND 2 WATERWASHES FOR RUNS 123-147

DECHLORINATION CONDITIONS 1-INCH DIAMETER QUARTZ ROTARY TUBE (RUNS 101 TO 114 AT 1 RPM RUNS 115 TO 148 AT 2 RPM) IN SPLIT TUBE FURNACES (2) STEAM ATMOSPHERE COAL CHARGED AT 2 TO 4 GRAMS/BATCH

(–) INDICATES NO TREATMENT FOR THAT PROCESSING STEP

Table 3. Laboratory scale data on coal desulfurization organized according to increasing sulfur removal

RUN DATE	COAL (ERDA COAL CODE)	TREATMENT* CHLORINATION TIME (MIN)	CHLORINATION TEMP (°C)	CHLORINATION CHLORINE (SCFH)	CHLORINATION WATER/COAL	CHLORINATION SOLVENT	HYDROLYSIS TIME (MIN)	HYDROLYSIS TEMP (°C)	HYDROLYSIS WATER/COAL	DECHLORINATION TIME (MIN)	DECHLORINATION TEMP (°C)	DECHLORINATION STEAM (gm/hr)	DECHLORINATION CO_2 (gm/hr)	RESIDUAL SULFUR ANALYSIS (WT %) ORGANIC	PYRITIC	SULFATE	TOTAL	SULFUR REMOVAL (%) ORGANIC	PYRITIC	TOTAL	RESIDUAL CHLORINE (WT %) DECHLORINATION BEFORE	AFTER	REMARKS
	PSOC 213 (HVB BIT	RAW	COAL											1 86	1 89	0 07	3 82				0 05		
101 6/22/77	KY NO 9)	120	74	0 125	0 5/1	METHYL CHLOROFORM	120	60	4/1	75	400	≈70		0 53	1 65	0 01	2 19	71	12 7	43	4 58	0 57	LOW CHLORINE RATE
	PSOC-108 (HVB BIT	RAW	COAL											1 07	2 06	0 00	3 13				0 06		1 5% MOISTURE
106 7/22/77	PITTS-BURGH,	30	74	0 5	0 5/1	METHYL CHLOROFORM	120	60	4/1	–	–	–		0 60	1 32	0 40	2 32	43 9	35 9	25 9	9 24		
106 7/22/77	WASH PA)	30	74	0 5	0 5/1	METHYL CHLOROFORM	120	60	4/1	–	–	–		0 82	1 15	0 02	1 99	23 4	44 2	36 4	6 12		
106-7/22/77		120	74	0 5	0 5/1	METHYL CHLOROFORM	120	60	4/1	–	–	–		0 88	0 50	0 07	1 45	17 8	75 7	53 7	14 66		
106-7/22/77		60	74	0 5	0 5/1	METHYL CHLOROFORM	120	60	4/1	–	–	–		0 75	0 24	0 40	1 39	29 9	88 3	55 6	12 28		
106-7/22/77		60	74	0 5	0 5/1	METHYL CHLOROFORM	120	60	4/1	–	–	–		0 81	0 46	0 05	1 32	24 3	77 7	57 8	9 46		
104 7/1/77		60	74	0 5	0 5/1	METHYL CHLOROFORM	120	60	4/1	–	–	–		0 18	0 96	0 13	1 27	83 2	53 4	59 4	8 0		
104 7/1/77		120	74	0 5	0 5/1	METHYL CHLOROFORM	120	60	4/1	–	–	–		0 90	0 28	<0 01	1 18	15 9	86 4	62 3	13 57		
104-7/1/77		120	74	0 5	0 5/1	METHYL CHLOROFORM	120	60	4/1	60	450	≈70		0 78	0 28	<0 01	1 01	27 1	88 8	67 7		0 39	
106-7/22/77		60	74	0 5	0 5/1	METHYL CHLOROFORM	120	60	4/1	60	450	≈70		0 56	0 17	<0 01	0 73	47 7	91 7	77		0 88	
104-7/1/77		60	74	0 5	0 5/1	METHYL CHLOROFORM	120	60	4/1	60	450	≈70		0 45	0 26	0 01	0 71	58 0	87 4	77 3			
106-7/22/77		60	74	0 5	0 5/1	METHYL CHLOROFORM	120	60	4/1	60	450	90		0 61	0 09	<0 01	0 70	43 0	95 6	78		0 97	
	PSOC-190 (HVA BIT	RAW	COAL											1 90	1 05	0 10	3 05				0 04		
109 8/8/77	ILL NO 6 KNOW,	60	74	0 5	0 5/1	METHYL CHLOROFORM	120	60	4/1	–	–	–		1 61	0 14	0 18	1 93	15 3	86 7	36 7	6 14		
109 8/8/77	ILL)	120	74	0 5	0 5/1	METHYL CHLOROFORM	–	–	–	–	–	–		1 27	0 09	0 48	1 84	33 2	91 4	39 7	17 64		
112 8/19/77		60	74	0 5	0 5/1	METHYL CHLOROFORM	120	80	4/1	–	–	–		1 57	0 13	0 10	1 80	17 4	87 6	41 0	9 1		
109-8/8/77		120	74	0 5	0 5/1	METHYL CHLOROFORM	120	60	4/1	–	–	–		1 50	0 08	0 02	1 60	21 2	92 4	47 5	12 57		
112 8/19/77		60	74	0 5	0 5/1	METHYL CHLOROFORM	120	80	4/1	60	500	75 110		1 34	0 03	0 03	1 40	29 5	97 1	54 0		0 08	
109-8/8/77		60	74	0 5	0 5/1	METHYL CHLOROFORM	120	60	4/1	60	470	~70		–	–	–	1 36	–	–	55 0		0 06	
109-8/8/77		120	74	0 5	0 5/1	METHYL CHLOROFORM	120	60	4/1	60	500	~70		1 26	0 02	<0 01	1 28	33 7	98 1	58 0		0 13	
	PSOC-276	RAW	COAL											2 24	2 07	0 84	5 15				0 14		
110-8/10/77	(HVA BIT OHIO NO	120	74	0 5	0 5/1	METHYL CHLOROFORM	–	-	–	–	–	–		0 79	0 53	1 33	2 65	64 7	25 6	48 5	22 4		
114 8/16/77	8 HARRI SON,	60	74	0 5	0 5/1	METHYL CHLOROFORM	120	80	4/1	–	–	–		0 59	0 74	0 39	1 72	73 7	64 3	66 6	10 74		
111 8/16/77	OHIO)	120	74	0 5	0 5/1	METHYL CHLOROFORM	60	80	4/1	–	–	–	–	0 85	0 24	0 54	1 63	62 0	88 4	68 3	14 81		

*CHLORINATION CONDITIONS 500 ml STIRRED FLASK 100 GRAM SAMPLES OF +200 MESH COAL SOLVENT/COAL = 2/1, ATM PRESSURE

HYDROLYSIS CONDITIONS 1000 ml STIRRED FLASK 1 WASH FOR RETENTION TIMES X60 MINUTES AND 2 WASHES EACH AT THE STATED WATER/COAL AT 120 MINUTES FILTRATION WITH 1/1 WATER/COAL WATERWASHES, WITH 1 WATERWASH FOR RUNS 101

DECHLORINATION CONDITIONS 1 INCH DIAMETER QUARTZ ROTARY TUBE (RUNS 101 TO 114 AT 1 RPM RUNS 115 TO 148 AT 2 RPM) IN SPLIT TUBE FURNACES (2), STEAM ATMOSPHERE COAL CHARGED AT 2 TO 4 GRAMS/BATCH

(–) INDICATES NO TREATMENT FOR THAT PROCESSING STEP

Table 3. Laboratory scale data on coal desulfurization organized according to increasing sulfur removal (continued)

RUN DATE	COAL (ERDA COAL CODE)	TREATMENT: CHLORINATION TIME (MIN)	TEMP (C)	CHLORINE (SCFH)	WATER/COAL	SOLVENT	HYDROLYSIS TIME (MIN)	TEMP (C)	WATER/COAL	DECHLORINATION TIME (MIN)	TEMP (C)	STEAM (gm/hr)	CO_2 (gm/hr)	RESIDUAL SULFUR ANALYSIS (WT %) ORGANIC	PYRITIC	SULFATE	TOTAL	SULFUR REMOVAL (%) ORGANIC	PYRITIC	TOTAL	RESIDUAL CHLORINE (WT %) DECHLORINATION BEFORE	AFTER	REMARKS
111 8/16/77	PSOC 276	120	74	0 5	0 5/1	METHYL CHLOROFORM	120	80	4/1	-	–	-		1 16	0 08	0 22	1 46	48 2	96 1	71 6	13 28		
110 8/10/77		120	74	0 5	0 5/1	METHYL CHLOROFORM	120	60	4/1		–	–		1 09	0 35	0 02	1 46	51 3	83 1	71 7	15 77		
111 8/16/77		120	74	0 5	0 5/1	METHYL CHLOROFORM	120	80	4/1	60	500	75 110		1 03	0 09	<0 01	1 12	54 0	95 6	78 2		0 09	
110 8/10/77		120	74	0 5	0 5/1	METHYL CHLOROFORM	120	80	4/1	60	500	≈70		1 03	0 04	<0 01	1 07	54 0	98 1	79 2	14 81		
111 8/16/77		120	74	0 5	0 5/1	METHYL CHLOROFORM	120	60	4/1	60	500	≈70		1 03	0 04	<0 01	1 07	54 0	98 1	79 0		0 28	
111 8/16/77		60	74	0 5	0 5/1	METHYL CHLOROFORM	120	80	4/1	60	500	75 110		0 88	0 05	<0 01	0 93	60 7	97 6	82 0		0 54	
111 8/16/77		120	74	0 5	0 5/1	METHYL CHLOROFORM	120	80	4/1	60	500	75 110		0 87	0 02	<0 01	0 89	61 2	99 0	82 7		0 17	
	PSOC 342	RAW	COAL											1 39	5 01	0 15	6 55				0 10		
127 10/3/77	(HVA BIT CLARION JEFFER SON PA)	120	74	0 5	0 5/1	METHYL CHLOROFORM	120	80	4/1	–	–	–	–	1 29	2 02	0 06	3 37	7 2	59 7	48 5	12 83		
142 10/21/77		60	74	0 5	0 5/1	METHYL CHLOROFORM	60	80	4/1	30	500	1	5 9	1 60	1 69	0 04	3 33	–	66 3	49 2		0 93	
142 10/21/77		60	74	0 5	0 5/1	METHYL CHLOROFORM	60	80	4/1	30	500	10	5 9	1 11	2 00	0 03	3 14	20 1	60 1	52 1		0 42	
127 10/3/77		120	74	0 5	0 5/1	METHYL CHLOROFORM	120	80	4/1	60	500	4 8	–	1,82	0 88	0 01	2 70	–	82 4	58 8		0 15	
	PSOC 097	RAW	COAL											0 84	0 38	0 01	1 23				0 00		
129 10/5/77	(SUBBIT A SEAM 80 CARBON WYO)	120	74	0 5	0 5/1	METHYL CHLOROFORM	120	80	4/1	–	–	–	–	0 84	0 29	0 09	1 22	0 0	23 7	1 0			
129 10/5/77		30	74	0 5	0 5/1	METHYL CHLOROFORM	120	80	4/1	30	500	0 4	5 9	0 70	0 31	0 05	1 06	16 7	18 4	13 8		0 28	
129 10/5/77		120	74	0 5	0 5/1	METHYL CHLOROFORM	120	80	4/1	60	550	2 4	5 9	0 75	0 09	0 04	0 88	10 7	76 3	28 5		0 22	
129 10/5/77		60	74	0 5	0 5/1	METHYL CHLOROFORM	120	80	4/1	60	500	1 0	5 9	0 74	0 05	0 02	0 81	11 9	86 8	34 1		0 13	
	PSOC 026	RAW	COAL											2 08	4 23	0 35	6 66				0 00		
	(HVC BIT ILL NO 6 SALINE	30	74	0 5	0 5/1	METHYL CHLOROFORM	120	80	4/1	30	–	–	–	1 30	0 89	0 02	2 21	37 5	79 0	66 8		0 20	
131 10/17/77	ILLINOIS)	60	74	0 5	0 5/1	METHYL CHLOROFORM	120	80	4/1	–	–	–	–	1 31	0 66	0 12	2 09	37 0	84 4	68 6	8 46		
		60	74	0 5	0 5/1	METHYL CHLOROFORM	120	80	4/1	30	500	5 0	–	1 20	0 45	0 01	1 66	42 3	89 4	75 1		0 42	
	PSOC 086	RAW	COAL											0 63	0 56	0 03	1 22				0 00		
135 10/12/77	(LIGNITE ZAP MERCER N DAKOTA)	60	74	0 5	0 5/1	METHYL CHLOROFORM	60	80	4/1	–	–	–	–	0 39	0 44	0 10	0 93	38 1	21 4	20 5	8 00		
		30	74	0 5	0 5/1	METHYL CHLOROFORM	60	80	4/1	30	500	2 2	–	0 35	0 23	0 17	0 75	44 4	58 9	38 5		0 33	
		60	74	0 5	0 5/1	METHYL CHLOROFORM	60	80	4/1	30	500	4 0	–	0 24	0 27	0 02	0 53	61 9	51 8	56 6			

*CHLORINATION CONDITIONS — 500 ml STIRRED FLASK 100 GRAM SAMPLES OF +200 MESH COAL SOLVENT/COAL = 2/1 ATM PRESSURE

HYDROLYSIS CONDITIONS — 1000 ml STIRRED FLASK 1 WASH FOR RETENTION TIMES ≤60 MINUTES AND 2 WASHES EACH AT THE STATED WATER/COAL AT 120 MINUTES FILTRATION WITH 1/1 WATER/COAL WATERWASHES WITH 1 WATERWASH FOR RUNS 101–122 AND 2 WATERWASHES FOR RUNS 123–147

DECHLORINATION CONDITIONS — 1-INCH DIAMETER QUARTZ ROTARY TUBE (RUNS 101 TO 114 AT 1 RPM RUNS 115 TO 148 AT 2 RPM) IN SPLIT TUBE FURNACES (2) STEAM ATMOSPHERE COAL CHARGED AT 2 TO 4 GRAMS/BATCH

(–) INDICATES NO TREATMENT FOR THAT PROCESSING STEP

Table 3. Laboratory scale data on coal desulfurization organized according to increasing sulfur removal (continued)

RUN DATE	COAL (ERDA COAL CODE)	TREATMENT* CHLORINATION TIME (MIN)	CHLORINATION TEMP (°C)	CHLORINATION CHLORINE (SCFH)	CHLORINATION WATER/COAL	CHLORINATION SOLVENT	HYDROLYSIS TIME (MIN)	HYDROLYSIS TEMP (°C)	HYDROLYSIS WATER/COAL	DECHLORINATION TIME (MIN)	DECHLORINATION TEMP (°C)	DECHLORINATION STEAM (gm/hr)	DECHLORINATION CO_2 (gm/hr)	RESIDUAL SULFUR ANALYSIS (WT %) ORGANIC	PYRITIC	SULFATE	TOTAL	SULFUR REMOVAL (%) ORGANIC	PYRITIC	TOTAL	RESIDUAL CHLORINE (WT %) DECHLORINATION BEFORE	AFTER	REMARKS
	PSOC 240 A 1 (SUB BIT B BIG D, LEWIS WASH)	RAW	COAL											1.75	1.60	0.01	3.36				0.02		
133 10/10/77		120	74	0.5	0.5/1	METHYL CHLOROFORM	60	80	4/1	30	500	3.5	–	0.49	0.68	0.05	1.22	72.0	57.5	63.7	0.26		
	PHS 398 RAW HEAD 3A SOMER SET, PA	RAW	COAL											0.46	2.26	0.29	3.01					0.10	THE SULFUR FORM ANALYSIS OF RAW COAL SUPPLIED BY PITTSBURGH BUREAU OF MINES IS 0.31% ORGANIC
140-10/19/77		60	74	0.5	0.5/1	METHYL CHLOROFORM	60	80	4/1	–	–	–	–	0.90	0,46	0.05	1.41		87.1	44.9	9.50		
137 10/14/77		30	74	0.5	0.5/1	METHYL CHLOROFORM	60	80	4/1	30	500	6.1	5.9	0.69	0.62	0.03	1.34	–	72.6	55.6		0.11	
137 10/14/77		60	74	0.5	0.5/1	METHYL CHLOROFORM	60	80	4/1	–	–	–	–	0.88	0.34	0.11	1.33	–	85.0	55.8	8.45		
140 10/19/77		30	74	0.5	0.5/1	METHYL CHLOROFORM	60	80	4/1	30	500	5.5	5.9	0.60	0.62	0.01	1.23	–	72.6	59.1		0.16	
137 10/14/77		60	74	0.5	0.5/1	METHYL CHLOROFORM	60	80	4/1	30	500	3.5	–	0.57	0.23	0.02	0.82	–	89.8	72.8		0.82	3.69% PYRITIC 0.01% SULFATE 4.01% TOTAL
140-10/19/77		60	74	0.5	0.5/1	METHYL CHLOROFORM	60	80	4/1	–	–	–	–	0.55	0.11	0.05	0.71	–	95.1	76.4	8.24		
140-10/19/77		60	74	0.5	0.5/1	METHYL CHLOROFORM	60	80	4/1	30	500	1.5	–	0.56	0.09	<0.01	0.65	–	96.1	78.4		0.77	
148 11/21/77	PHS 513 BIT MINE NO 513 UPPER CLARION BUTLER PA	RAW	COAL											1.76	<0.2	<0.2	1.76				0.27		PHYSICALLY CLEANED HIGH ORGANIC SULFUR COAL
		120	74	0.5	0.5/1	METHYL CHLOROFORM	60	80	4/1	30	500	10	–	1.28	<0.2	<0.2	1.28	27.3	–	27.3		1.18	SULFUR AND RESIDUAL
		30	74	0.5	0.5/1	METHYL CHLOROFORM	60	80	4/1	30	500	10	–	1.27	<0.2	<0.2	1.27	27.8	–	27.8		0.44	CHLORINE ANALYSIS
		60	74	0.5	0.5/1	METHYL CHLOROFORM	60	80	4/1	30	500	10	–	1.16	<0.2	<0.2	1.16	34.1	–	34.1		0.90	PERFORMED BY JPL
	PSOC 219 (HVA BIT KY NO 4, HOPKINS KY)	RAW	COAL											1.08	1.40	0.08	2.56				0.03		HEATING VALUE OF RAW COAL 13 400 BTU/LB (MF BASIS)
136-10/13/77		30	74	0.5	0.5/1	METHYL CHLOROFORM	60	80	4/1	–	–	–	–	0.49	1.24	0.03	1.76	54.6	11.4	31.2	4.69		
125-9/28/77		10	74	0.5	0.5/1	METHYL CHLOROFORM	30	60	2/1	–	–	–	–	0.69	0.94	0.07	1.70	36.1	32.9	33.2	4.78		
136-10/13/77		30	74	0.5	0.5/1	METHYL CHLOROFORM	60	80	4/1	30	500	1	5.9	1.39	0.31	<0.01	1.70	–	77.9	33.6		0.08	
125-9/28/77		20	74	0.5	0.5/1	METHYL CHLOROFORM	30	100	2/1	–	–	–	–	0.71	0.86	0.10	1.67	34.2	38.6	34.8	4.86		
125-9/28/77		30	74	0.5	0.5/1	METHYL CHLOROFORM	20	80	2/1	–	–	–	–	0.86	0.73	0.06	1.65	20.4	47.9	35.5	6.95		
147 10/28/77		60	74	0.5	0.5/1	METHYL CHLOROFORM	60	80	4/1	–	–	–	–	0.72	0.78	0.11	1.61	33.3	44.3	37.1	7.34		
125-9/28/77		20	74	0.5	0.5/1	METHYL CHLOROFORM	60	100	2/1	–	–	–	–	0.58	0.89	0.12	1.59	46.3	36.4	37.9	5.01		
125-9/28/77		30	74	0.5	0.5/1	METHYL CHLOROFORM	45	80	2/1	–	–	–	–	0.87	0.65	0.04	1.56	19.4	37.9	39.1	8.15		
136-10/13/77		60	74	0.5	0.5/1	METHYL CHLOROFORM	60	80	4/1	30	500	3.5	–	0.33	1.21	0.01	1.55	69.4	13.6	39.5		0.01	
125-9/28/77		30	74	0.5	0.5/1	METHYL CHLOROFORM	60	80	2/1	–	–	–	–	0.80	0.68	0.04	1.53	25.9	51.4	40.2	9.65		
125-9/28/77		10	74	0.5	0.5/1	METHYL CHLOROFORM	60	60	2/1	15	500	0.4	–	0.88	0.64	<0.01	1.52	18.5	54.3	40.6			
125-9/28/77		30	74	0.5	0.5/1	METHYL CHLOROFORM	30	80	2/1	–	–	–	–	1.04	0.42	0.06	1.52	3.7	70.0	40.6	7.28		
124 9/27/77		30	74	0.5	0.3/1	METHYL CHLOROFORM	60	80	2/1	–	–	–	–	0.64	0.83	0.13	1.50	50.0	40.7	41.4	4.74		
117 9/6/77		30	74	0.5	0.5/1	METHYL CHLOROFORM	120	80	4/1	60	350	75 110	–	0.88	0.59	0.03	1.50	18.5	57.9	41.4		0.89	

*CHLORINATION CONDITIONS 500 ml STIRRED FLASK 100 GRAM SAMPLES OF+200 MESH COAL SOLVENT/COAL = 2/1 ATM PRESSURE

HYDROLYSIS CONDITIONS 1000 ml STIRRED FLASK 1 WASH FOR RETENTION TIMES <60 MINUTES AND 2 WASHES EACH AT THE STATED WATER/COAL AT 120 MINUTES FILTRATION WITH 1/1 WATER/COAL WATERWASHES WITH 1 WATERWASH FOR RUNS 101–122 AND 2 WATERWASHES FOR RUNS 123–147

DECHLORINATION CONDITIONS 1 INCH DIAMETER QUARTZ ROTARY TUBE (RUNS 101 TO 114 AT 1 RPM RUNS 115 TO 148 AT 2 RPM) IN SPLIT TUBE FURNACES (2) STEAM ATMOSPHERE COAL CHARGED AT 2 TO 4 GRAMS/BATCH

(–) INDICATES NO TREATMENT FOR THAT PROCESSING STEP

Table 3. Laboratory scale data on coal desulfurization organized according to increasing sulfur removal (continued)

	COAL (ERDA COAL CODE)	TREATMENT*												RESIDUAL SULFUR ANALYSIS (WT %)				SULFUR REMOVAL (%)			RESIDUAL CHLORINE (WT %)		
		CHLORINATION					HYDROLYSIS			DECHLORINATION											DECHLORINATION		
RUN DATE		TIME (MIN)	TEMP (°C)	CHLORINE (SCFH)	WATER/COAL	SOLVENT	TIME (MIN)	TEMP (°C)	WATER/COAL	TIME (MIN)	TEMP (°C)	STEAM (gm/hr)	CO_2 (gm/hr)	ORGANIC	PYRITIC	SULFATE	TOTAL	ORGANIC	PYRITIC	TOTAL	BEFORE	AFTER	REMARKS
119 9/13/77	PSOC 219	120	74	0 5	0 7/1	METHYL CHLOROFORM	60	80	4/1	60	350	75	–	0 75	0 68	0 02	1 45	30 5	51 4	43 4		1 03	
123 9/23/77		120	74	0 5	0 7/1	METHYL CHLOROFORM	60	80	4/1	–	–	–	–	0 70	0 60	0 05	1 35	35 2	57 1	47 3	11 12		
120 9/16/77		30	74	0 5	0 3/1	METHYL CHLOROFORM	120	80	4/1	60	450	5 0	–	0 51	0 83	<0 01	1 34	52 8	40 7	47 6		0 22	
124 9/27/77		120	74	0 5	0 3/1	METHYL CHLOROFORM	40	100	4/1	–	–	–	–	0 82	0 44	0 07	1 33	24 1	68 6	48 0	13 81		
119 9/13/77		120	74	0 5	0 7/1	METHYL CHLOROFORM	120	80	4/1	–	–	–	–	0 85	0 42	0 04	1 31	21 3	70 0	48 8	11 50		
108 8/1/77		120	74	0 5	0 5/1	METHYL CHLOROFORM	120	60	4/1	–	–	–	–	0 59	0 62	0 10	1 31	45 4	55 7	49 0	13 45		
124 9/27/77		30	74	0 5	0 5/1	METHYL CHLOROFORM	120	80	4/1	60	450	0 4	-	0 73	0 53	0 04	1 30	32 4	62 1	49 2		1 69	
141 10/21/77		30	74	0 5	0 5/1	METHYL CHLOROFORM	30	80	4/1	30	500	1 0	5 9	0 96	0 34	<0 01	1 30	11 1	75 7	49 2		0 10	COAL PARTICLE SIZE -70 TO +120 MESH
124 9/27/77		120	74	0 5	0 5/1	METHYL CHLOROFORM	10	100	4/1	–	–	–	–	0 48	0 64	0 18	1 30	55 6	54 3	49 2	18 30		
139 10/18/77		30	74	0 5	0 5/1	METHYL CHLOROFORM	60	80	4/1	30	500	5 5	5 9	0 89	0 38	<0 01	1 27	17 6	72 9	50 4			
123 9/23/77		30	74	0 5	0 7/1	METHYL CHLOROFORM	60	80	2/1	60	500	1 26	5 9	0 79	0 47	0 01	1 27	26 9	66 4	50 4		0 11	
125 9/28/77		20	74	0 5	0 5/1	METHYL CHLOROFORM	60	100	2/1	30	500	1 6	5 9	0 79	0 44	0 02	1 25	26 9	68 6	51 2		0 12	
143 10/24/77		60	74	0 5	0 5/1	METHYL CHLOROFORM	60	80	4/1	–	–	–	–	0 32	0 68	0 25	1 25	70 4	51 4	51 2	11 59		
144 10/25/77		60	85	0 5	0 5/1	METHYL CHLOROFORM	60	80	4/1	–	–	–	–	0 56	0 46	0 22	1 24	48 1	67 1	51 6	11 35		
123 9/23/77		60	74	0 5	0 7/1	METHYL CHLOROFORM	120	80	3/1	–	–	–	–	0 57	0 48	0 06	1 23	47 2	65 7	52 0	9 18		
124 9/27/77		120	74	0 5	0 3/1	METHYL CHLOROFORM	20	100	4/1	–	–	–	–	0 73	0 39	0 11	1 23	32 4	72 1	52 0	16 36		
138 10/17/77		60	74	0 5	0 5/1	METHYL CHLOROFORM	60	80	4/1	–	–	–	–	0 69	0 46	0 08	1 23	36 1	67 1	52 0	9 86		
119 9/13/77		30	74	0 5	0 7/1	METHYL CHLOROFORM	120	80	4/1	30	500	5	5 9	0 76	0 44	0 02	1 22	29 6	68 6	52 3		0 24	
124 9/27/77		120	74	0 5	0 3/1	METHYL CHLOROFORM	60	100	4/1	–	–	–	–	0 78	0 37	0 07	1 22	27 8	73 6	52 3			
124 9/27/77		60	74	0 5	0 3/1	METHYL CHLOROFORM	60	80	3/1	–	–	–	–	0 42	0 69	0 10	1 22	61 1	50 7	52 3	8 66		
123 9/23/77		120	74	0 5	0 7/1	METHYL CHLOROFORM	120	80	4/1	25	550	1 0	5 9	0 53	0 67	<0 01	1 20	50 9	52 1	53 1		2 11	
125 9/28/77		30	74	0 5	0 5/1	METHYL CHLOROFORM	60	80	2/1	15	450	0 76	5 9	0 73	0 46	0 01	1 20	32 4	67 1	53 1		1 22	
123 9/23/77		60	74	0 5	0 7/1	METHYL CHLOROFORM	120	80	3/1	30	500	4 0	–	0 67	0 52	<0 01	1'19	38 0	62 9	53 5		0 86	

*CHLORINATION CONDITIONS: 500 ml STIRRED FLASK 100 GRAM SAMPLES OF+200 MESH COAL SOLVENT/COAL = 2/1 ATM PRESSURE

HYDROLYSIS CONDITIONS: 1000 ml STIRRED FLASK 1 WASH FOR RETENTION TIMES <60 MINUTES AND 2 WASHES EACH AT THE STATED WATER/COAL AT 120 MINUTES FILTRATION WITH 1/1 WATER/COAL WATERWASHES WITH 1 WATERWASH FOR RUNS 101-122 AND 2 WATERWASHES FOR RUNS 123-147

DECHLORINATION CONDITIONS: 1 INCH DIAMETER QUARTZ ROTARY TUBE (RUNS 101 TO 114 AT 1 RPM RUNS 115 TO 148 AT 2 RPM) IN SPLIT TUBE FURNACES (2), STEAM ATMOSPHERE COAL CHARGED AT 2 TO 4 GRAMS/BATCH

(–) INDICATES NO TREATMENT FOR THAT PROCESSING STEP

Table 3. Laboratory scale data on coal desulfurization organized according to increasing sulfur removal (continued)

RUN DATE	COAL (ERDA COAL CODE)	TREATMENT: CHLORINATION TIME (MIN)	CHLORINATION TEMP (°C)	CHLORINATION CHLORINE (SCFH)	CHLORINATION WATER/COAL	CHLORINATION SOLVENT	HYDROLYSIS TIME (MIN)	HYDROLYSIS TEMP (°C)	HYDROLYSIS WATER/COAL	DECHLORINATION TIME (MIN)	DECHLORINATION TEMP (°C)	DECHLORINATION STEAM (gm/hr)	DECHLORINATION CO_2 (gm/hr)	RESIDUAL SULFUR ANALYSIS (WT %) ORGANIC	PYRITIC	SULFATE	TOTAL	SULFUR REMOVAL (%) ORGANIC	PYRITIC	TOTAL	RESIDUAL CHLORINE (WT %) DECHLORINATION BEFORE	AFTER	REMARKS
123 9/23/77	PSOC 219	120	74	0 5	0 7/1	METHYL CHLOROFORM	120	80	4/1	25	550	2 0	–	0 86	0 32	0 01	1 19	20 4	77 1	53 5		0 74	
123 9/23/77		120	74	0 5	0 7/1	METHYL CHLOROFORM	120	80	4/1	25	500	1 0	–	0 62	0 56	<0 01	1 18	42 6	60 0	53 9		1 35	
119 9/13/77		120	74	0 5	0 7/1	METHYL CHLOROFORM	120	80	4/1	–	–	–	–	0 69	0 39	0 08	1 16	36 1	72 1	54 7	12 00		
124 9/27/77		60	74	0 5	0 3/1	METHYL CHLOROFORM	30	80	3/1	–	–	–	–	0 44	0 61	0 11	1 16	59 3	56 4	54 7	9 07		
125 9/28/77		30	74	0 5	0 5/1	METHYL CHLOROFORM	60	80	2/1	20	500	0 6	–	0 75	0 39	<0 01	1 14	30 5	72 1	55 5		1 10	
116 8/30/77		60	60	0 5	0 5/1	METHYL CHLOROFORM	120	80	4/1	–	–	–	–	0 69	0 31	0 13	1 13	36 1	77 9	55 8	8 64		
118 9/9/77		60	74	0 5	0 5/1	METHYL CHLOROFORM	120	80	4/1	–	–	–	–	0 55	0 52	0 06	1 13	49 1	62 8	55 9	17 90		
125 9/28/77		30	74	0 5	0 5/1	METHYL CHLOROFORM	60	80	2/1	20	450	1 0	5 9	0 75	0 36	<0 01	1 12	30 5	74 3	56 2		0 77	
125 9/28/77		30	74	0 5	0 5/1	METHYL CHLOROFORM	60	80	2/1	15	500	0 4	–	0 73	0 39	<0 01	1 12	32 4	72 1	56 2		0 65	
125 9/28/77		30	74	0 5	0 5/1	METHYL CHLOROFORM	60	80	2/1	20	500	1 5	–	0 68	0 42	<0 01	1 10	37 0	70 0	57 0		1 16	
119 9/13/77		60	74	0 5	0 7/1	METHYL CHLOROFORM	120	80	4/1	30	500	3 8	5 9	0 65	0 44	<0 01	1 09	39 8	68 6	57 4		0 21	
125 9/28/77		30	74	0 5	0 5/1	METHYL CHLOROFORM	60	80	2/1	15	500	2 0	–	0 71	0 37	<0 01	1 08	34 2	73 6	57 8		0 54	
118 9/9/77		60	74	0 5	0 5/1	METHYL CHLOROFORM	120	80	4/1	–	–	–	–	0 63	0 36	0 08	1 07	41 7	74 3	58 2	11 97		
120 9/16/77		120	74	0 5	0 3/1	METHYL CHLOROFORM	30	80	4/1	–	–	–	–	0 68	0 28	0 10	1 06	37 0	80 0	58 6	20 50		
123 9/23/77		120	74	0 5	0 7/1	METHYL CHLOROFORM	120	80	4/1	25	500	0 5	5 9	0 85	0 21	<0 01	1 06	21 3	85 0	58 6		0 45	
139 10/18/77		60	74	0 5	0 5/1	METHYL CHLOROFORM	60	80	4/1	30	500	5 2	–	0 66	0 37	0 02	1 05	38 9	73 6	59 0		0 77	
118 9/9/77		60	74	0 5	0 5/1	METHYL CHLOROFORM	60	80	4/1	25	500	1 0	5 9	0 68	0 33	<0 01	1 01	37 0	76 4	60 5		1 35	
117 9/6/77		60	50	0 5	0 5/1	METHYL CHLOROFORM	120	80	4/1	60	450	75 110	–	0 77	0 23	0 01	1 01	28 7	83 6	60 5		0 60	
117 9/6/77		120	50	0 5	0 5/1	METHYL CHLOROFORM	120	80	4/1	–	–	–	–	0 49	0 39	0 13	1 01	54 6	82 6	60 5	19 08		
138 10/17/77		60	74	0 5	0 5/1	METHYL CHLOROFORM	60	80	4/1	30	500	10	–	0 74	0 23	0 03	1 00	31 5	83 6	60 9		0 45	
102 6/27/77		120	74	0 25	0 5/1	METHYL CHLOROFORM	120	60	4/1	–	–	–	–	0 46	0 50	0 03	0 99	57 4	64 3	61 3	10 17		
144 10/25/77		60	85	0 5	0 5/1	METHYL CHLOROFORM	60	80	4/1	30	500	1 20	–	0 74	0 23	0 02	0 99	31 5	83 6	61 3		0 86	
118 9/9/77		60	74	0 5	0 5/1	METHYL CHLOROFORM	120	80	4/1	60	450	75 110	–	0 61	0 35	0 02	0 98	43 5	75 0	61 7	11 50	0 30	
118 9/9/77		60	74	0 5	0 5/1	METHYL CHLOROFORM	60	80	4/1	–	–	–	–	0 33	0 45	0 18	0 96	69 4	67 9	62 5			
141 10/21/77		60	74	0 5	0 5/1	METHYL CHLOROFORM	60	80	4/1	30	500	5 5	5 9	0 70	0 26	<0 01	0 96	35 2	81 4	62 5		0 06	COAL PARTICLE SIZE –70 TO +120 MESH
146 10/28/77		60	74	0 5	0 5/1	METHYL CHLOROFORM	60	80	4/1	–	–	–	–	0 60	0 29	0 06	0 95	44 4	79 3	62 9	15 97		

*CHLORINATION CONDITIONS: 500 ml STIRRED FLASK 100 GRAM SAMPLES OF+200 MESH COAL SOLVENT/COAL = 2/1 ATM PRESSURE

HYDROLYSIS CONDITIONS: 1000 ml STIRRED FLASK 1 WASH FOR RETENTION TIMES ≤60 MINUTES AND 2 WASHES EACH AT THE STATED WATER/COAL AT 120 MINUTES FILTRATION WITH 1/1 WATER/COAL WATERWASHES WITH 1 WATERWASH FOR RUNS 101–122 AND 2 WATERWASHES FOR RUNS 123–147

DECHLORINATION CONDITIONS: 1 INCH DIAMETER QUARTZ ROTARY TUBE (RUNS 101 TO 114 AT 1 RPM RUNS 115 TO 148 AT 2 RPM) IN SPLIT TUBE FURNACES (2) STEAM ATMOSPHERE COAL CHARGED AT 2 TO 4 GRAMS/BATCH

(–) INDICATES NO TREATMENT FOR THAT PROCESSING STEP

Table 3. Laboratory scale data on coal desulfurization organized according to increasing sulfur removal (continued)

RUN DATE	COAL (ERDA COAL CODE)	TREATMENT* CHLORINATION TIME (MIN)	CHLORINATION TEMP (°C)	CHLORINATION CHLORINE (SCFH)	CHLORINATION WATER/COAL	CHLORINATION SOLVENT	HYDROLYSIS TIME (MIN)	HYDROLYSIS TEMP (°C)	HYDROLYSIS WATER/COAL	DECHLORINATION TIME (MIN)	DECHLORINATION TEMP (°C)	DECHLORINATION STEAM (gm/hr)	DECHLORINATION CO_2 (gm/hr)	RESIDUAL SULFUR ANALYSIS (WT %) ORGANIC	PYRITIC	SULFATE	TOTAL	SULFUR REMOVAL (%) ORGANIC	PYRITIC	TOTAL	RESIDUAL CHLORINE (WT %) DECHLORINATION BEFORE	AFTER	REMARKS
120 9/16/77	PSOC 219	60	74	0 5	0 3/1	METHYL CHLOROFORM	60	80	4/1	60	450	121	–	0 66	0 28	<0 01	0 94	38 9	80 0	63 3		0 14	
115 8/26/77		120	50	0 5	0 5/1	METHYL CHLOROFORM	120	80	4/1	–	–	–	–	0 66	0 26	0 01	0 93	38 9	81 4	63 7	18 25		
145 10/24/77		60	74	0 5	0 5/1	METHYL CHLOROFORM	60	80	4/1	30	500	2 5	–	0 87	0 06	<0 01	0 93	19 4	95 7	63 7		0 89	
103 7/8/77		120	74	1 0	0 5/1	METHYL CHLOROFORM	120	60	4/1	–	–	–	–	0 70	0 04	0 18	0 92	35 2	97 1	64 0	19 8		
107 7/27/77		120	74	0 5	0 5/1	METHYL CHLOROFORM	120	60	4/1	–	–	–	–	0 24	0 59	0 10	0 93	77 8	57 9	64 0	11 13		
108 8/1/77		120	74	0 5	0 5/1	METHYL CHLOROFORM	120	60	4/1	60	450	≈70	–	0 56	0 36	<0 01	0 92	48 1	74 3	64 0		0 33	
118 9/9/77		60	74	0 5	0 5/1	METHYL CHLOROFORM	60	80	4/1	20	500	1 0	5 9	0 63	0 28	<0 01	0 91	41 7	80 0	64 4		0 84	
118 9/9/77		60	74	0 f	0 5/1	METHYL CHLOROFORM	60	80	4/1	20	450	1 3	–	0 50	0 40	<0 01	0 90	53 7	71 4	64 8		1 11	
118 9/9/77		60	74	0 5	0 5/1	METHYL CHLOROFORM	60	80	4/1	20	400	100	–	0 51	0 39	<0 01	0 90	52 8	72 1	64 8		1 21	
117 9/6/77		120	50	0 5	0 5/1	METHYL CHLOROFORM	120	80	4/1	60	450	75 110	–	0 33	0 56	<0 01	0 89	69 4	60 0	65 2		0 72	
120 9/16/77		120	74	0 5	0 3/1	METHYL CHLOROFORM	120	80	4/1	60	450	105	–	0 68	0 21	<0 01	0 89	37 0	85 0	65 2		0 26	
107 7/27/77		120	74	0 5	0 5/1	METHYL CHLOROFORM	120	60	4/1	60	450	≈70	–	0 63	0 22	0 02	0 87	41 7	84 3	66 0		0 48	
108 8/1/77		60	60	0 5	0 5/1	METHYL CHLOROFORM	120	60	4/1	60	450	≈70	–	0 28	0 45	0 15	0 88	74 0	68 0	66 0		0 41	
116 8/30/77		30	60	0 5	0 5/1	METHYL CHLOROFORM	120	80	4/1	60	500	75 110	–	0 71	0 16	<0 01	0 87	34 3	88 6	66 0			
118 9/9/77		60	74	0 5	0 5/1	METHYL CHLOROFORM	60	80	4/1	25	450	0 65	–	0 59	0 28	<0 01	0 87	45 4	80 0	66 0		2 01	
116 8/30/77		120	60	0 5	0 5/1	METHYL CHLOROFORM	120	80	4/1	–	–	–	–	0 74	0 08	0 03	0 85	45 9	94 3	66 8	22 37		
146 10/28/77		60	74	0 5	0 5/1	METHYL CHLOROFORM	30	80	4/1	30	500	0 6	5 9	0 64	0 20	0 01	0 85	40 7	85 7	66 8		0 36	SOLVENT/COAL 4/1
143 10/24/77		30	74	1 0	0 5/1	METHYL CHLOROFORM	60	80	4/1	30	500	5 5	5 9	0 54	0 28	0 02	0 84	50 0	80 0	67 2		0 57	
105 7/19/77		30	74	0 5	0 5/1	METHYL CHLOROFORM	120	60	4/1	60	450	≈70	–	0 71	0 10	0 01	0 82	34 3	92 8	68 0		0 69	
107 7/27/77		30	74	0 5	0 5/1	METHYL CHLOROFORM	120	60	4/1	60	450	≈70	–	0 72	0 11	<0 01	0 82	33 3	92 1	68 0		0 22	
123 9/23/77		120	74	0 5	0 7/1	METHYL CHLOROFORM	120	80	4/1	60	550	4	–	0 61	0 21	<0 01	0 82	43 5	85 0	68 0		0 17	
124 9/27/77		60	74	0 5	0 3/1	METHYL CHLOROFORM	120	80	3/1	60	500	20	–	0 54	0 27	0 01	0 82	50 0	80 7	68 0		0 28	
124 9/27/77		120	74	0 5	0 3/1	METHYL CHLOROFORM	60	100	4/1	60	500	0 5	–	0 60	0 12	<0 01	0 81	36 1	91 4	68 4		1 63	
138 10/17/77		60	74	0 5	0 5/1	METHYL CHLOROFORM	60	80	4/1	60	500	9	–	0 58	0 23	<0 01	0 81	46 3	83 6	68 4		0 43	
116 8/30/77		60	60	0 5	0 5/1	METHYL CHLOROFORM	120	80	4/1	60	500	75 110	–	0 74	0 06	<0 01	0 80	45 9	95 7	68 7		0 47	
115 8/26/77		30	50	0 5	0 5/1	METHYL CHLOROFORM	120	80	4/1	60	500	75 110	–	0 68	0 12	<0 01	0 80	37 0	91 4	68 8		<0 01	LOWER CHLORINATION TEMP OF 50°C
124 9/27/77		120	74	0 5	0 3/1	METHYL CHLOROFORM	60	100	4/1	40	500	0 4	–	0 80	<0 01	<0 01	0 80	25 9	100 0	68 8		1 34	
102 6/27/77		120	74	0 25	0 5/1	METHYL CHLOROFORM	120	60	4/1	60	450	≈70	–	0 38	0 41	<0 01	0 79	64 8	46 8	69 0		0 31	
145 10/24/77		60	74	0 5	0 5/1	METHYL CHLOROFORM	30	80	4/1	–	–	–	–	0 68	0 08	<0 01	0 76	37 0	94 3	70 3	8 72		

*CHLORINATION CONDITIONS — 500 ml STIRRED FLASK 100 GRAM SAMPLES OF+200 MESH COAL SOLVENT/COAL = 2/1 ATM PRESSURE

HYDROLYSIS CONDITIONS — 1000 ml STIRRED FLASK 1 WASH FOR RETENTION TIMES <60 MINUTES AND 2 WASHES EACH AT THE STATED WATER/COAL AT 120 MINUTES FILTRATION WITH 1/1 WATER/COAL WATERWASHES, WITH 1 WATERWASH FOR RUNS 101–122 AND 2 WATERWASHES FOR RUNS 123–147

DECHLORINATION CONDITIONS — 1 INCH DIAMETER QUARTZ ROTARY TUBE (RUNS 101 TO 114 AT 1 RPM RUNS 115 TO 148 AT 2 RPM) IN SPLIT TUBE FURNACES (2) STEAM ATMOSPHERE COAL CHARGED AT 2 TO 4 GRAMS/BATCH

(–) INDICATES NO TREATMENT FOR THAT PROCESSING STEP

Table 3. Laboratory scale data on coal desulfurization organized according to increasing sulfur removal (continued)

RUN DATE	COAL (ERDA COAL CODE)	TREATMENT* CHLORINATION TIME (MIN)	TEMP (C)	CHLORINE (SCFH)	WATER/COAL	SOLVENT	HYDROLYSIS TIME (MIN)	TEMP (°C)	WATER/COAL	DECHLORINATION TIME (MIN)	TEMP (C)	STEAM (gm/hr)	CO_2 (gm/hr)	RESIDUAL SULFUR ANALYSIS (WT %) ORGANIC	PYRITIC	SULFATE	TOTAL	SULFUR REMOVAL (%) ORGANIC	PYRITIC	TOTAL	RESIDUAL CHLORINE (WT %) DECHLORINATION BEFORE	AFTER	REMARKS
143 10/24/77	PSOC 219	60	74	1 0/0 25	0 5/1	METHYL CHLOROFORM	60	80	4/1	30	500	5 5	5 9	0 44	0 31	<0 01	0 75	59 2	77 9	70 7		0 57	CHLORINE AT 1 0 SCFH FOR
103 7/8/77		60	74	1 0	0 5/1	METHYL CHLOROFORM	120	60	4/1	60	450	≈70	–	0 43	0 12	0 19	0 74	60 2	91 4	71 0			FIRST 30 MIN 0 25 SCFH FOR
118 9/9/77		60	74	0 5	0 5/1	METHYL CHLOROFORM	60	80	4/1	60	500	4 6	–	0 58	0 14	0 01	0 73	46 3	90 0	71 5		0 60	LAST 30 MIN
145-10/24/77		60	74	0 5	0 5/1	METHYL CHLOROFORM	60	80	4/1	30	500	0 6	5 9	0 68	0 04	<0 01	0 72	37 0	97 1	71 9		0 ?8	PREVIOUSLY DECHLORINATED
105-7/19/77		30	74	0 5	0 5/1	METHYL CHLOROFORM	120	60	4/1	60	450	≈70	–	0 61	0 12	<0 01	0 72	43 5	91 4	71 9		0 84	COAL SAMPLE
107 7/27/77		120	74	0 5	0 5/1	METHYL CHLOROFORM	120	60	4/1	60	450	≈70	–	0 35	0 37	<0 01	0 72	67 6	73 6	71 9		0 31	
117 9/6/77		120	50	0 5	0 5/1	METHYL CHLOROFORM	120	80	4/1	60	500	75 110	–	0 66	0 05	<0 01	0 71	38 9	96 4	72 3		0 30	
115 8/26/77		120	50	0 5	0 5/1	METHYL CHLOROFORM	120	80	4/1	60	500	75-110	–	0 52	0 06	0 11	0 69	51 8	95 7	73 0		0 50	
119 9/13/77		120	74	0 5	0 7/1	METHYL CHLOROFORM	120	80	4/1	60	500	7 5		0 65	0 03	<0 01	0 68	39 8	97 9	73 4		0 12	
115 8/26/77		60	50	0 5	0 5/1	METHYL CHLOROFORM	120	80	4/1	60	500	75 110	–	0 62	0 03	<0 01	0 65	42 6	97 9	74 6		0 45	
116 8/30/77		120	60	0 5	0 5/1	METHYL CHLOROFORM	120	80	4/1	60	490	75 110	–	–	–	–	0 63	–	–	76 4		0 50	
105 7/19/77		30	74	0 5	0 5/1	METHYL CHLOROFORM	–	–	–	–	–	–	–	0 52	1 05	0 60	2 17	51 9	25 0	15 2	5 36		SERIES OF RUNS FEATURING
107 7/27/77		30	74	0 5	0 5/1	METHYL CHLOROFORM	–	–	–	–	–	–	–	0 87	0 79	0 42	2 08	19 4	43 6	18 8	5 41		CHLORINATION ONLY NO
102 6/27/77		120	74	0 25	0 5/1	METHYL CHLOROFORM	–	–	–	–	–	–	–	0 17	0 87	0 77	1 81	84 3	37 9	29 3	12 41		HYDROLYSIS OR DECHLORINATION
103 7/8/77		30	74	1 0	0 5/1	METHYL CHLOROFORM	–	–	–	–	–	–	–	0 42	0 85	0 49	1 76	61 1	39 3	31 3	6 27		
107 7/27/77		120	74	0 5	0 5/1	METHYL CHLOROFORM	–	–	–	–	–	–	–	0 14	0 74	0 78	1 66	87 0	47 1	35 2	20 30		
102 6/27/77		120	74	0 25	0 5/1	METHYL CHLOROFORM	–	–	–	–	–	–	–	0 21	0 73	0 71	1 65	80 6	47 9	35 5			
105 7/19/77		30	74	0 5	0 5/1	METHYL CHLOROFORM	–	–	–	–	–	–	–	0 81	0 70	0 11	1 62	25 0	50 0	36 7	3 01		
103 7/8/77		60	74	1 0	0 5/1	METHYL CHLOROFORM	–	–	–	–	–	–	–	0 23	0 26	0 60	1 09	78 7	81 4	57 4	13 11		
126 9/30/77		60	74	0 5	0 5/1	TETRACHLOROETHYLENE	30	100	2/1	–	–	–	–	0 82	0 57	0 13	1 52	24 1	59 3	40 6		0 41	BEGIN TETRACHLOROETHYL
134 10/10/77		30	74	0 5	0 7/1	TETRACHLOROETHYLENE	60	80	4/1	–	–	–	–	0 70	0 75	0 07	1 52	35 2	46 4	40 6	11 2		ENE EVALUATION
126 9/30/77		120	74	0 5	0 5/1	TETRACHLOROETHYLENE	30	80	2/1	–	–	–	–	0 72	0 53	0 25	1 50	33 3	62 1	41 4	24 41		
122 9/21/77		60	100	0 5	0 5/1	TETRACHLOROETHYLENE	60	80	4/1	–	–	–	–	0 64	0 77	0 08	1 49	40 7	45 0	41 8	?3 06		
122 9/21/77		15	100	0 5	0 5/1	TETRACHLOROETHYLENE	60	80	4/1	60	350	2	–	0 66	0 77	0 01	1 44	38 9	45 0	43 7		0 44	
126-9/30/77		30	74	0 5	0 5/1	TETRACHLOROETHYLENE	30	60	2/1	–	–	–	–	0 65	0 60	0 13	1 38	39 8	57 1	46 1			
126-9/30/77		15	74	0 5	0 5/1	TETRACHLO	60	60	2/1	60	500	0 3	–	0 99	0 34	0 02	1 35	8 3	75 7	47 3		1 29	
132 10/7/77		120	74	0 5	0 3/1	ROETHYLENE	60	80	4/1	–	–	–	–	0 62	0 50	0 09	1 29	42 6	58 6	49 6	17 10		

*CHLORINATION CONDITIONS — 500 ml STIRRED FLASK 100 GRAM SAMPLES OF +200 MESH COAL SOLVENT/COAL = 2/1, ATM PRESSURE

HYDROLYSIS CONDITIONS — 1000 ml STIRRED FLASK 1 WASH FOR RETENTION TIMES <60 MINUTES AND 2 WASHES EACH AT THE STATED WATER/COAL AT 120 MINUTES FILTRATION WITH 1/1 WATER/COAL WATERWASHES WITH 1 WATERWASH FOR RUNS 101–122 AND 2 WATERWASHES FOR RUNS 123–147

DECHLORINATION CONDITIONS — 1 INCH DIAMETER QUARTZ ROTARY TUBE (RUNS 101 TO 114 AT 1 RPM, RUNS 115 TO 148 AT 2 RPM) IN SPLIT TUBE FURNACES (2) STEAM ATMOSPHERE COAL CHARGED AT 2 TO 4 GRAMS/BATCH

(–) INDICATES NO TREATMENT FOR THAT PROCESSING STEP

Table 3. Laboratory scale data on coal desulfurization organized according to increasing sulfur removal (continued)

RUN DATE	COAL (ERDA COAL CODE)	TREATMENT* CHLORINATION TIME (MIN)	CHLORINATION TEMP (°C)	CHLORINATION CHLORINE (SCFH)	CHLORINATION WATER/COAL	CHLORINATION SOLVENT	HYDROLYSIS TIME (MIN)	HYDROLYSIS TEMP (°C)	HYDROLYSIS WATER/COAL	DECHLORINATION TIME (MIN)	DECHLORINATION TEMP (°C)	DECHLORINATION STEAM (gm/hr)	DECHLORINATION CO_2 (gm/hr)	RESIDUAL SULFUR ANALYSIS (WT %) ORGANIC	PYRITIC	SULFATE	TOTAL	SULFUR REMOVAL (%) ORGANIC	PYRITIC	TOTAL	RESIDUAL CHLORINE (WT %) DECHLORINATION BEFORE	AFTER	REMARKS
126 9/30/77	PSOC 219	60	74	0 5	0 5/1	TETRACHLO ROETHYLENE	60	100	2/1	–	–	–	–	0 71	0 41	0 09	1 21	34 2	70 7	52 7	25 07		
134 10/10/77		60	74	0 5	0 7/1	TETRACHLO ROETHYLENE	60	80	4/1	–	–	–	–	0 40	0 80	<0 01	1 20	63 0	42 9	53 1	14 34		
122 9/21/77		30	100	0 5	0 5/1	TETRACHLO ROETHYLENE	60	80	4/1	60	550	4	–	1 00	0 14	<0 01	1 14	7 4	90 0	55 5		0 31	
132 10/7/77		120	74	0 5	0 3/1	TETRACHLO ROETHYLENE	60	80	4/1	30	500	5	5 9	0 73	0 35	<0 01	1 08	32 4	75 0	57 8		0 64	
126 9/30/77		60	74	0 5	0 5/1	TETRACHLO ROETHYLENE	60	100	2/1	60	500	0 25	–	0 77	0 22	<0 01	0 98	28 7	84 3	61 7		1 22	
122 9/21/77		60	100	0 5	0 5/1	TETRACHLO ROETHYLENE	60	80	4/1	60	550	4	–	0 82	0 08	0 02	0 92	24 1	94 3	64 1		0 39	
126 9/30/77		120	74	0 5	0 5/1	TETRACHLO ROETHYLENE	60	80	2/1	60	500	1	–	0 82	0 07	<0 01	0 89	24 1	95 0	65 2		1 14	
126 9/30/77		30	74	0 5	0 5/1	TETRACHLO ROETHYLENE	60	60	2/1	60	500	7 5	–	0 58	0 19	<0 01	0 77	46 3	86 4	69 9		0 41	
132 10/7/77		60	74	0 5	0 3/1	TETRACHLO ROETHYLENE	60	80	4/1	30	500	4	–	0 51	0 20	<0 01	0 71	52 8	85 7	72 3		0 80	
128 10/4/77		120	74	0 5	0 5/1	CARBON TETRA CHLORIDE	60	60	2/1	–	–	–	–	0 66	0 79	0 05	1 50	38 9	43 6	41 4	5 38	0 68	CARBON TETRA CHLORIDE EVALUATION
128 10/4/77		60	74	0 5	0 5/1	CARBON TETRA CHLORIDE	30	80	2/1	–	–	–	–	0 76	0 57	0 12	1 45	29 6	59 3	43 4	5 78		
130 10/5/77		120	74	0 5	0 3/1	CARBON TETRA CHLORIDE	60	80	4/1	–	–	–	–	0 58	0 56	0 12	1 26	46 3	60 0	50 5	17 63		
128 10/4/77		120	74	0 5	0 5/1	CARBON TETRA CHLORIDE	60	60	2/1	60	500	8 5	–	0 86	0 35	<0 01	1 21	20 4	75 0	52 7			
121 9/20/77		60	74	0 5	0 7/1	CARBON TETRA CHLORIDE	60	80	4/1	–	–	–	–	0 56	0 50	0 09	1 15	48 1	64 3	55 1	11 91		
121 9/20/77		30	74	0 5	0 7/1	CARBON TETRA CHLORIDE	60	80	4/1	60	500	4	–	0 72	0 31	<0 01	1 03	33 3	77 8	59 8		0 21	
130 10/5/77		120	74	0 5	0 3/1	CARBON TETRA CHLORIDE	30	80	4/1	30	500	2	5 9	0 47	0 30	0 03	0 80	56 5	78 6	68 7		1 25	
121 9/20/77		60	74	0 5	0 7/1	CARBON TETRA CHLORIDE	60	80	4/1	60	500	3	–	0 65	0 08	0 02	0 75	39 8	94 3	70 7		0 15	

*CHLORINATION CONDITIONS — 500 ml STIRRED FLASK 100 GRAM SAMPLES OF+200 MESH COAL SOLVENT/COAL = 2/1 ATM PRESSURE

HYDROLYSIS CONDITIONS — 1000 ml STIRRED FLASK 1 WASH FOR RETENTION TIMES <60 MINUTES AND 2 WASHES EACH AT THE STATED WATER/COAL AT 120 MINUTES, FILTRATION WITH 1/1 WATER/COAL WATERWASHES WITH 1 WATERWASH FOR RUNS 101–122 AND 2 WATERWASHES FOR RUNS 123–147

DECHLORINATION CONDITIONS — 1 INCH DIAMETER QUARTZ ROTARY TUBE (RUNS 101 TO 114 AT 1 RPM, RUNS 115 TO 148 AT 2 RPM) IN SPLIT TUBE FURNACES (2) STEAM ATMOSPHERE COAL CHARGED AT 2 TO 4 GRAMS/BATCH

(–) INDICATES NO TREATMENT FOR THAT PROCESSING STEP

60, and 120 minutes at temperatures of 60, 80 and 100°C with a 1/1 and 2/1 water/coal filter-cake wash. The majority of runs were with two washes at water/coal of 4/1 and 2 filter-cake washes at water/coal of 1/1 for a total water/coal ratio of 10. A 20 minute wash at water/coal of 2/1 with a filter-cake wash of 2/1 at 80°C reduced the sulfate-content to less than 0.1 weight percent. The total water requirement in this case is water/coal of 4/1. Inspection of the residual sulfate level indicates the adequacy of the hydrolysis.

Dechlorination. Dechlorination of the treated coal was carried out with 2 to 4 gram coal samples contained in a 1-inch-diameter quartz tube and then placed inside a split-tube muffle furnace. The quartz tube was rotated at 1 and 2 RPM. Steam flow rates from 0.25 to 121 grams per hour were employed at temperatures of 350 to 550°C with dechlorinations of 15, 20, 25, 30, 40, 60, and 75 minutes. Steam dechlorination of treated coal at 450°C, and a steam rate of 75 grams/hour over a 2 gram sample, indicated that HCl evolution from the treated coal stopped within 20 minutes (Figure 5). Carbon dioxide at 6 grams/hour was introduced in some runs along with steam to approximate the effect of combustion gases. Dechlorination results indicate that residual chlorine levels are reduced in some instances to less than 0.1 weight percent, but that in other cases under approximately the same dechlorination conditions residual chlorine levels are 1.0 weight percent. The existing dechlorination data do not appear to correlate with any given parameters for achieving chlorine levels of less than 0.1 weight percent, although these low levels of residual chlorine have been achieved in a significant number of cases. Reduction of steam rates from 100 to 1 gram/hour has no significant effect on dechlorination.

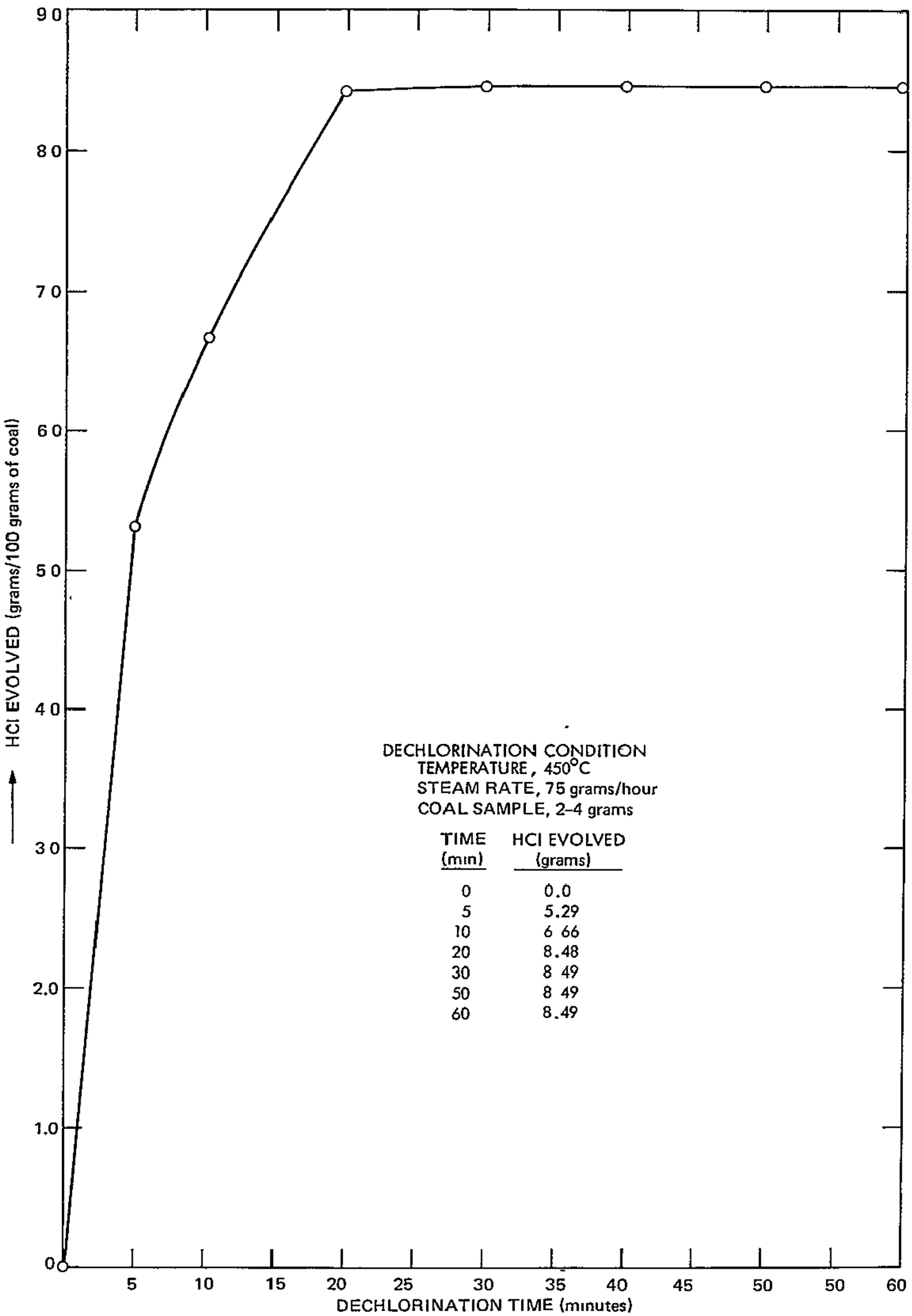

Figure 5. Steam dechlorination of treated coal with time

Analytical Chemistry (1.3)

Sulfur Analyses

Sample analyses were conducted primarily by Galbraith Laboratories, Knoxville, Tennessee. Sulfur analysis data for organic, pyritic, sulfate and total sulfur for raw and treated coal samples are included in Tables 2 and 3.

The scatter of coal desulfurization results for coal samples treated under duplicate conditions has raised questions about the precision and accuracy of sulfur analysis data. A sample of coal PSOC-219 treated in run 119-9/13/77 was divided into six samples. Five samples were submitted at different times to Galbraith Laboratories for analyses and the sixth sample was sent to the U.S. Bureau of Mines laboratory in Pittsburgh, Pennsylvania. The results are reported in Table 4. The Galbraith Laboratory analyses showed an average deviation of total sulfur of ±0.13 percent (±15 percent), organic sulfur ±0.05 percent (±7 percent) and pyritic sulfur ± 0.07 percent (±44 percent). The scatter of the total sulfur data was somewhat greater than expected by ASTM standards but not great enough in itself to explain the large variation in coal desulfurization results. However, comparison of the Galbraith Laboratory results to those of the U.S. Bureau of Mines indicated a substantial bias of the sulfur data to higher values than those reported by the U.S. Bureau of Mines. The Bureau of Mines data were lower in total sulfur by +0.28 weight percent (+33 relative percent) organic sulfur by +0.18 weight percent (+27 relative percent) and pyritic sulfur by +0.09 weight percent (+56 relative percent). The comparison of the data between two laboratories should, according to ASTM procedures, be substantially closer than recorded in Table 4. However, even considering the differences in sulfur data between the two laboratories, the scatter in coal desulfurization data was greater than can be explained by analytical differences. If the Galbraith analyses of residual sulfur quantities were biased in the high direction as indicated, the coal desulfurization results after correction for the bias become substantially more attractive, with a significant number of samples meeting sulfur compliance requirements.

Ultimate Analyses

Ultimate analyses of raw and treated coals PSOC-219 and PSOC-190 are given in Table 5. Coal PSOC-219 exhibits a significant reduction in hydrogen, approximately 2 weight percent, whereas PSOC-190 exhibits less than 1 weight percent reduction in hydrogen. The nitrogen content in the PSOC-219 raw coal appears in error at 0.1 weight percent. The carbon content of PSOC-190 rises sharply after treatment, apparently as a result of the sharp decrease in oxygen (by 2.8 percent) after treatment.

Table 4. Comparison of sulfur and chlorine analyses in duplicate* samples of treated coal

(Coal sample PSOC-219, treatment run 119 - 9/13/77)

Sample Submitted	Sulfur Analyses (Wt. %) Organic	Pyritic	Sulfate	Total	Chlorine (Wt. %)
Galbraith Labs					
#1 – 10/21/77	0.69	0.23	0.06	0.98	0.77
#2 – 10/21/77	0.63	0.10	<0.01	0.73	0.81
#3 – 10/21/77	0.72	0.12	<0.01	0.84	1.02
#4 – 10/17/77	0.61	0.08	<0.01	0.69	0.46
#5 – 10/27/77	0.71	0.26	0.09	1.06	0.74
Average	0.67	0.16	0.036	0.86	0.76
Ave. Dev	±0.05 (7%)	±0.07 (44%)	±0.031 (86%)	±0.13 (15%)	±0.13 (17%)
U.S Bureau of Mines Laboratory					
#6 – 11/2/77	0.49	0.07	0.02	0.58	0.47
Dev (Ave. #1-5) - (#6)	+0.18 (+27%)	+0.09 (+56%)	+0.016 (+44%)	+0.28 (+33%)	±0.29 (38%)

*Multiple samples were obtained of -100 to +200 mesh coal treated in run 119-9/13/77 and 5 samples submitted to Galbraith Laboratories, Knoxville, Tenn and 1 sample to the U S. Bureau of Mines, Coal Preparation and Analysis Laboratory, Pittsburgh, Pa

Trace Metals

Trace metals analysis in raw/treated PSOC 219 and PHS 398 coals indicate sharp reductions for titanium, phosphorous, arsenic, lead, vanadium, lithium and beryllium in that order of reduction (Table 6). Reductions are from 50 to 91 percent in treated coal.

Water Solution Analyses

The chlorinator water scrubber solutions from runs 112, 118, 138, and 142 were analyzed for sulfate, sulfite, chloride and total organic carbon (Table 7). Negligible sulfate and sulfite were found. A substantial carryover of HCl from the chlorinator was indicated, representing 5 to 10 percent of the total chlorine feed. Total organic carbon carryover to the water scrubber was negligible.

Hydrolysis water solutions were analyzed for sulfate, chloride, sulfite, total organic carbon, iron, calcium and trace metals (Table 8). Approximately 17 to 25 grams of the 45 grams of chlorine feed were present in the water along with 1 to 3 grams of sulfur present as sulfate. Total organic carbon varied from 0.1 to 2.0 grams. Iron represented 0.8 to 1.0 gram in solution for runs 118 and 138. Other quantities of trace metals were substantially less, although calcium, aluminum and sodium were in the range of 50 to 160 mg/liter. Distilled water was used for the hydrolyses; thus, the total contribution of trace materials identified was from coal.

Dechlorinator water scrubber solution was analyzed for sulfate, chloride and total organic carbon (Table 9). Sulfate and total organic carbon represented 0.5 to 2.7 percent of the coal feed. Sulfate sulfur represented 6 to 17 percent of the total sulfur in the coal. Chloride represented 18 to 33 percent of the total chlorine feed.

Gas Analyses

Mass spectrometer analyses were performed of the gases contained in the gas holder connected to the chlorinator, hydrolyzer, dechlorinator and solvent evaporator for runs 118, 132, 134, 138 and 142 (Table 10). Air contaminating the gas holders was eliminated by correction of the gas sample for nitrogen and oxygen. Only trace quantities of methyl chloroform, CO, CO_2, CH_4, acetone and chlorohydrocarbons were found in the gas holders, and no sulfur was detected.

Material Balances

Material balances were obtained for coal, methyl chloroform, chlorine, and sulfur for runs 112, 118, 138, and 142 (Tables 11, 12, 13 and 14, respectively). Total accounting was made

Table 5. Ultimate analyses of treated coals PSOC-219 and PSOC-190

Component	PSOC-219 (HVA Bit. KY No. 4)				PSOC-190 (HVA Bit. ILL. No. 6, Knox Ill.)	
		Treated Coal				Treated Coal
	Raw Coal (Wt. %)	Run 138–10/17/77 (Wt. %)	Run 138–10/17/77 (Wt. %)	Run 120–9/16/77 (Wt. %)	Raw Coal (Wt. %)	Run 109–8/8/77 (Wt. %)
C	74 16	75.53	74.83	77 30	69.15	74.15
H	5 30	3.46	2.38	3 16	4.89	3.99
N	0 10	1.84	1.65	1 26	1 00	1.36
S	2 56	0 88	1.02	1 00	3.05	1.36
Cl	0.03	0 45	0 75	1.40	0 06	0.06
Ash	8 06	7.78	7.40	6 23	8 49	8.29
0 (by difference)	9 79	10.06	11.97	9 65	13 42	10 80
Moisture	0 00	1.40	2 30	0 00	0.00	–
Heating Value (Btu/lb)	13,398	12,412	12,780	–	–	–

Table 6. Trace metals analyses of raw/treated PSOC-219 and PHS-398

	PSOC-219[a] Raw Coal	PSOC-219 Treated Coal				PHS-398[b] Raw Coal	PHS-398 Treated Coal	
		Run 107 - 7/27/77		Run 120 - 9/16/77			Run 140 - 10/20/77	
Analyses	PPM	PPM	Percent Reduction (Wt. %)	PPM	Percent Reduction (Wt. %)	PPM	PPM	Percent Reduction (Wt. %)
Titanium	1086	510	53.0	680	37.4	1400	700	50.0
Phosphorous	131	68/130	48 1/0.8	68	48.1	1040	700	32.7
Arsenic	73	25	65.8	49	32.3	85	9	89.4
Lead	46	4	91.3	5	89 1	0.5	3	–
Vanadium	46	12	81 0	48	0.0	<25	<25	~0.0
Lithium	<10	5	~ 50 0	–	–	20	21	0 0
Barium	5	5	0 0	–	–	<10	92	
Beryllium	8	4	50 0	13	0.0	5	4	20.0
Cadmium	1	<1		–	–	–	–	–
Mercury	<1	<1		–	–	<0.5	<0.5	~0.0
Selenium	<1	<1		–	–	<1	<1	~0 0

[a] HVA Bit. Ky No. 4.

[b] Raw Head, 3A, Freidens (Somerset), Pa. Received from Dr. Scott R. Taylor, Bureau of Mines, Pittsburgh, Pa.

Table 7. Analyses of chlorinator water scrubber solutions from processing of three different coals

Analyses	Run 112[a] (mg/ℓ)	Run 118[b] (mg/ℓ)	Run 138[c] (mg/ℓ)	Run 142[d] (mg/ℓ)
Sulfate (as S)	0.1	0.1	0.1	0.1
Sulfite (as SO_3)	0.1	0.1	–	–
Chloride	12,500	11,000	7510	4010
Total Organic Carbon	10	10	75	50

[a]Basis: 100 grams of PSOC-190, 200 cc Scrubber Solution

[b]Basis: 100 grams of PSOC-219, 200 cc Scrubber Solution

[c]Basis: 100 grams of PSOC-219, 600 cc Scrubber Solution

[d]Basis: 100 grams of PSOC-342, 600 cc Scrubber Solution

Table 8. Analysis of hydrolysis water solution from treatment of three different coals

Analyses	Run 112[a] (mg/ℓ)	Run 118[b] (mg/ℓ)	Run 138[c] (mg/ℓ)	Run 142[d] (mg/ℓ)
Sulfate (as S)	1,050	1,410	2,170	5,010
Chloride	17,500	17,900	30,170	42,670
Sulfite (as SO_3)	1.0	0.1		
Total Organic Carbon	75.0	480	1,830	3,330
Fe		800	1,600	
Ca		140	55.0	
Al		50.0	125	
Na		160	–	
Pb		5 0	12 5	
As			3 8	
Mg			41 0	
K			22 5	
Ti			8.5	
P			6 6	

[a]100 grams of PSOC-190 coal, 1000 cc Hydrolysis Water Solution

[b]100 grams of PSOC-219 coal, 1000 cc Hydrolysis Water Solution

[c]100 grams of PSOC-219 coal, 600 cc Hydrolysis Water Solution

[d]100 grams of PSOC-342 coal, 600 cc Hydrolysis Water Solution

on three of the four runs for coal, chlorine and sulfur. Coal accounting was 83 to 88 percent, chlorine accounting 94 to 99 percent and sulfur accounting 90 to 96 percent. Methyl chloroform losses were appreciable at 11.3 and 15.0 percent for runs 118 and 112, respectively. However, substantial improvement in solvent recovery was noted in runs 138 and 142, with only 1.4 and 3.6 percent unaccounted losses. Since the solvent was in contact with a substantial amount of hydrolysis wash water, the loss may be explained by limited solubility and entrainment of small quantities of solvent with water-coal slurry. Careful processing of wash water should allow recovery of even these small losses of methyl chloroform. Product coal recovered represented 76 to 80 percent of the coal fed for runs 118, 138 and 142. If unaccounted losses, which are assumed to be primarily solid particle losses of coal in the dechlorination apparatus are assumed to be recovered as product, coal product recovery is 91 to 96 percent. The high sulfur content of 6.55 percent for coal PSOC-342 reduced product coal yield to 92.5 percent by virtue of the high sulfur removal. The majority of the methyl chloroform (82-98 percent) was recovered in the solvent evaporation stage, with only 1 to 3 percent recovered in the chlorinator cold trap. Chlorine was recovered as HCl from the hydrolyzer as 40 to 60 percent of the feed chlorine; from 6 to 32 percent of the HCl was recovered in the chlorinator cold trap. The remaining HCl (21 to 32 percent) was recovered in the dechlorinator gas scrubber. Sulfur was recovered primarily with the hydrolysis wash water. A small amount of the sulfur (6 to 17 percent) was recovered in the dechlorinator gas scrubber.

Experimental and Analytical Studies for Coal Desulfurization Reactions (1.4)

Experimental Data

Forty seven runs are included in Table 2, representing the total operating data for chlorination, hydrolysis and dechlorination of twelve coals. Thirty of these runs were conducted with PSOC-219 for parametric screening of operating conditions. The data were grouped in terms of increasing total sulfur removal in Table 3 to provide visibility for coordination of operating conditions with sulfur removal.

The data show a substantial scatter for organic, pyritic and total sulfur in terms of residual sulfur levels and sulfur reduction values for duplicate sets of operating conditions. Analytical error cannot explain the large disparity in the data. Analysis of the data in terms of an important parameter such as chlorination time indicates that extending time beyond 30 minutes does not improve desulfurization. In fact, some data suggest that sulfur may be reintroduced into the organic structure of the coal by reaction of intermediate sulfur compounds in extending the chlorination conditions beyond the optimum, or by failing to remove sulfur compounds from the coal slurry during the chlorination. If, in fact, competing sulfur reactions are present for introduction of the sulfur into the coal structure, then it becomes evident that the reaction mechanisms controlling coal desulfurization become much more complex

Table 9. Analysis of dechlorinator water scrubber solution from treatment of three different coals

Analyses	Run 112[a] (mg/ℓ)	Run 118[b] (mg/ℓ)	Run 138[c] (mg/ℓ)	Run 142[d] (mg/ℓ)
Sulfate (as S)	58	20	59	115
Chloride	1370	1490	1387	3410
Total Organic Carbon	67	60	360	391

[a]Basis 2 grams of PSOC-190, 155 cc Scrubber Solution
[b]Basis 2 grams of PSOC-219, 150 cc Scrubber Solution
[c]Basis 2 grams of PSOC-219, 150 cc Scrubber Solution
[d]Basis 4 grams of PSOC-342, 150 cc Scrubber Solution

Table 10. Mass spectrometer analyses of process off-gases

Analyses	Run 138					Run 134		Run 118		Run 132		Run 142	
	Chlorinator[a]		Hydrolyzer	Dechlorinator[b]		Solvent Evaporator[c]		Chlorinator[d]		Chlorinator[e]		Chlorinator[f]	
	Vol. %	Grams		Vol. %	Grams	Vol. %	Grams	Vol. %	Grams	Vol. %	Grams	Vol. %	Grams
He			NO GAS EVOLVED	0.0	0 0	70 4	0 01	74.9	0 07	97 9	0.32	1.3	0 0009
Ar	62 5	0.02		0 0	0 0	7 0	0 001	8 6	0.008	1 5	0.005	41 7	0.03
CO	25 0	0 008		9 31	0 004	0 0	0 0	0.0	0.0	0.0	0 0	41 7	0 03
Acetone	0 0	0 00		67 24	0 064	1 4	0 0002	16.1	0 015	0 6	0.002	13.9	0 01
Methyl Chloroform	12 5	0 004		0 0	0.0	21 2	0.003	0 4	0.0004	0.0	0.0	1 4	0.001
S	0 0	0 00		0.0	0 0	0 0	0.0	0 0	0.0	0.0	0 0	0.0	0.0
CO_2	0 0	0 00		12.00	0.009	0 0	0 0	0 0	0 0	0.0	0 0	0 0	0.0
CH_4	0 0	0 00		11.28	0 003	0 0	0.0	0 0	0 0	0 0	0.0	0.0	0 0
Chlorohydrocarbons	0.0	0 0		0 17	0 037	0.0	0 0	0.0	0 0	0 0	0 0	0 0	0 0
	100.0	0.032		100 00	0 117	100.0	0.0142	100 0	0.0934	100 0	0 327	100 0	0 0719

[a]1 hr, 1200 cc gas evolved.

[b]Basis 500°C, 1 hr, 4 gms dry coal, 37 cc gas evolved

[c]~800 cc gas evolved, no hydrolysis.

[d]Total gas evolved 870 cc, chlorinator purged with helium initially.

[e]2 hr run, 2020 cc gas evolved, system purged with helium initially

[f]Total gas evolved 1215 cc

Table 11. Material balance for run 112-8/19/77, coal PSOC-190

Process Unit	Process Stream	Coal (Incl. Sulfur)		Methyl Chloroform		Chlorine		Sulfur	
		Grams	Wt. %	Grams	Wt. %	Grams	Wt. %	Grams	Wt. %
Chlorinator (Feed)	Coal, Cℓ, Solvent, S	90		200		45		3 0	
Chlorinator Cold Trap	$CH_3CC\ell_3$, Cℓ			6	3	14.16	31 5		
Chlorinator Gas Scrubber	Cℓ, SO_4, TOC	0 002[c]	0 002[c]			2 5[b]	5.6[b]	$<10^{-5}$	0
Chlorinator Gas Collector		–	–						
Solvent Evaporator	$CH_3CC\ell_3$			164	82				
Hydrolyzer	Cℓ, SO_4, TOC,	0 075[c]	0 083[c]			17 5[b]	38 9[b]	1 05[a]	34 4[a]
	Trace Metals	–[e]	–[e]						
Dechlorinator Gas Scrubber	Cℓ, SO_4, TOC	0 47[c]	0 52[c]			9 55[b]	21 2[b]	0 40[a]	13 1[a]
Dechlorinator Gas Collector		–	–						
Product Coal Storage	Product Coal, Cℓ, S	–[d]	–[d]			–	–	–	–
Total Accounting		–	–	170	85	–	–	–	–
Unaccounted		–	–	30	15	–	–	–	–

[a] $SO_4^{=}$ as Sulfur

[b] Chloride

[c] Carbon

[d] Product Storage Including Unaccounted Coal

[e] Trace Metals

Table 12. Material balance for run 118-9/9/77, coal PSOC-219

Process Unit	Process Stream	Coal (Incl. Sulfur)		Methyl Chloroform		Chlorine		Sulfur	
		Grams	Wt. %	Grams	Wt. %	Grams	Wt. %	Grams	Wt. %
Chlorinator (Feed)	Coal, $C\ell$, Solvent	96 67	2	200		45		2 56	
Chlorinator Cold Trap	$CH_3 CC\ell_3$, $C\ell$			1 4	0 7	11 5	25 5		
Chlorinator Gas Scrubber	$C\ell$, SO_4, TOC	0 002[c]	0 002[c]			2.2[b]	4 9[b]	<0 01[a]	–
Chlorinator Gas Collector		0 011	0 011						
Solvent Evaporator	$CH_3 CC\ell_3$			176 0	88 0				
Hydrolyzer	$C\ell$, SO_4, TOC,	0 48[c]	0 50[c]			17 9[b]	39 8[b]	1.41[a]	55 1[a]
	Trace Metals	1.155[e]	1 195[e]						
Dechlorinator Gas Scrubber	$C\ell$, SO_4, TOC	0.47[c]	0 49[c]			11 2[b]	24 9[b]	0 16[a]	6 2[a]
Dechlorinator Gas Collector		–	–						
Product Coal Storage	Product Coal, $C\ell$, S	76 55 (92 98)[d]	79 19 (96 19)[d]			0 23	0 5	0 75	29 3
Total Accounting		30 24	83.00	117.4	88 7	43 03	95 6	2 32	90 6
Unaccounted		16 43	17 00	22 6	11 3	1.97	4 4	0 24	9 4

[a] $SO_4^=$ as Sulfur
[b] Chloride
[c] Carbon
[d] Product Storage Including Unaccounted Coal
[e] Trace Metals

Table 13. Material balance for run 138-10/7/77, coal PSOC-219

Process Unit	Process Stream	Coal (Incl Sulfur) Grams	Coal (Incl Sulfur) Wt %	Methyl Chloroform Grams	Methyl Chloroform Wt. %	Chlorine Grams	Chlorine Wt. %	Sulfur Grams	Sulfur Wt. %
Chlorinator (Feed)	Coal, $C\ell$, Solvent, S	97 07		200		45		2 56	
Chlorinator Cold Trap	$CH_3\,CC\ell_3$, $C\ell$			1 3	0.7	12.69	28 2		
Chlorinator Gas Scrubber	$C\ell$, SO_4, TOC	0 045[c]	0 046[c]						
Chlorinator Gas Collector		0 0029	0 003						
Solvent Evaporator	$CH_3\,CC\ell_3$			195 8	97 9	1 5	3 3	<0 01[a]	
Hydrolyzer	$C\ell$, SO_4, TOC,	1 1[c]	1 1[c]			18.1[b]	40 2[b]	1.30[a]	50 8[a]
	Trace Metals	1 125[e]	1 16[e]						
Dechlorinator Gas Scrubber	$C\ell$, SO_4, TOC	2 88[c]	2.97[c]			9.72[b]	21 6[b]	0 44[a]	17 2[a]
Dechlorinator Gas Collector		2 31	2 38						
Product Coal Storage	Product Coal, $C\ell$, S	74 09 (87 86)[d]	76 33 (90.52[d])			0 34	0 8	0 71	27 7
Total Accounting		83 30	85 81	197 1	98 6	42 34	94 1	2 45	95 7
Unaccounted		13 77	14 19	2.9	1 4	2 66	5 9	0.11	4 3

[a] $SO_4^=$ as Sulfur
[b] Chloride
[c] Carbon
[d] Product Storage Including Unaccounted Coal
[e] Trace Metals

Table 14. Material balance for run 142-10/21/77, coal PSOC-342

Process Unit	Process Stream	Coal (Incl Sulfur)		Methyl Chloroform		Chlorine		Sulfur	
		Grams	Wt %	Grams	Wt %	Grams	Wt. %	Grams	Wt. %
Chlorinator Feed		95 33		200		44.8		6.55	
Chlorinator Cold Trap	CH_3CCl_3, Cl, S			4 0	2 0	2 8	6 3		
Chlorinator Gas Scrubber	Cl, SO_4, TOC	0 032[c]	0 034[c]			0 8[b]	1 8[b]	<0 01	
Chlorinator Gas Collector		0 0205	0 0215						
Solvent Evaporator	CH_3CCl_3			188 8	94 4				
Hydrolyzer	Cl, SO_4, TOC,	2 0[c]	2 0[c]			25 6[b]	56 9[b]	3 0[a]	45.8[a]
	Trace Metals	–[e]	–[e]						
Declorinator Scrubber	Cl, SO_4, TOC	1 64[c]	1 72[c]			14 2[b]	31 7[b]	0 48[a]	7 3[a]
Declorinator Gas Collector		–	–						
Product Coal Storage	Product Coal, Cl, S	76 39 (88 16)[d]	80 13 (92 48)[d]			0 72	1 6	2 54	38 8
Total Accounting		83 56	87 65	192 8	96 4	44 12	98 5	6 02	91 9
Unaccounted		11 77	12 35	7 2	3 6	0 68	1 5	0 53	8 1

[a]$SO_4^{=}$ as Sulfur

[b]Chloride

[c]Carbon

[d]Product Storage Including Unaccounted Coal

[e]Trace Metals

than previously supposed. The complexity of coal desulfurization reactions may also obscure the effects of the parameters of time, temperature, water/coal ratios and solvent type. Early data on the chlorination reaction were obtained at relatively low chlorine injection rates. Thus, these data indicated that desulfurization increased with increasing reaction time. This was a situation in which chlorine injection was controlling because of the low chlorine feed rate. In later chlorinations at chlorine feed rates of 0.5 SCFH, the requisite chlorine for saturation of the coal slurry was obtained in 45 minutes. Thus, addition of chlorine beyond 45 minutes does not produce further benefits. It is possible that acceptable coal desulfurization can be achieved with a retention time less than that required to saturate the coal slurry with chlorine. It is also possible that the effects of water/coal ratio, solvent type and temperature may have pronounced effects if the chlorination reaction is restricted to times of less than 45 minutes.

Coal desulfurization data for PSOC-219 coal indicate that organic sulfur removal was from a few percent to 87 percent, pyritic sulfur removal was from a few percent to 100 percent, and total sulfur removal was 15 percent to 75 percent. The average organic sulfur removal was 42 percent. Average pyritic sulfur removal was 60 to 70 percent, and average total sulfur removal was 50 to 60 percent. Generally, the average level of residual total sulfur was in the range of 0.7 to 1.5 percent, which is above sulfur compliance levels with emission standards. The other ten coals tested provided similar coal desulfurization results, with only two of the coals showing organic sulfur removals of less than 20 percent.

Because of the relatively large amount of coal desulfurization data and the large scatter in the sulfur analyses of the processed coal, a statistical interpretation of the data was considered appropriate. The statistical analysis of the coal desulfurization data follows.

Linear Multiple Regression Analysis

Statistical multiple regression analysis provides an analysis of the ability of a large number of experimental data to correlate with a set of independent variables. The analysis is especially useful when there is a large variation in the data that does not seem to correlate with any given set of variables. By assuming an equation which relates the dependent variable to a set of selected independent variables, multiple regression analysis will fit the data with the equation and yield the best set of coefficients for the equation. In addition, valuable statistical information may be obtained such as the mean, standard deviation, variance, partial variance contributed by the variation of each independent variable, the percentage of variance unexplained by the selected equation form, and how good the data fitting is in terms of statistical testings such as confidence level. There are many existing computer programs for multiple regression analysis. The second edition of Statistical Package for the Social Sciences (SPSS) prepared by the University of Chicago (Authors: N.H. Nie et al.) and published by McGraw-Hill Book Company in 1975 was chosen by JPL because it is a complete and well-recognized statistical analysis package that has been used successfully in many engineering applications.

Without detailed understanding of the functional dependence of each independent parameter upon the dependent variable, a linear correlation is always the first logical approximation to be used. Therefore, linear multiple regression analysis (e.g., $Y' = C_0 + C_1X_1 + C_2X_2 + C_3 X_3 + \ldots$) was selected for fitting data from coal desulfurization by chlorinolysis experiments and conducting corresponding statistical analyses. Table 15 lists the regression notation used in this section of the report. Three cases were analyzed. Table 16 contains the regression analysis input data for PSOC-219 coal and Table 17 contains the data from nine other coals. Analysis of the coal data was done in three cases: Case I - Total Coal Input Data; Case II - PSOC-219 Coal Only and Case III Nine Coals Not Including PSOC-219. In Case I, 57 percent of the input data are representative of PSOC-219 coal; therefore, the analysis was broken into three cases to eliminate total domination by PSOC-219 coal. Representative data were selected for each run to avoid weighting each run with several sets of analyses.

Regression analysis input data for nine other coals is given in Table 17. It includes the high-pyritic-sulfur coal PHS-398 provided by BOM, but not the PSOC-240A1 coal, since these analytical data were not ready in time.

Results of correlation analyses for the three cases are summarized in Table 18 for six equations correlating sulfur removal. The data omitted in Cases 2 and 3 of Table 18 are constants, and thus not suitable for inclusion in a multiple regression analysis.

Case 1 consists of the combined analyses of PSOC-219 and the nine other coals. Case 2 presents the data fitting for PSOC-219 only. Case 3 consists of data fitting for the other nine coals. The reason for separating PSOC-219 from the other coals in Cases 2 and 3 is that PSOC-219 was used in extensive parametric analyses and provided a broad base of data from a single coal. Results from correlation of data for all three cases are summarized in the following paragraphs:

Table 15. List of Notations for Linear Multiple Regression Analysis

I. Dependent Variables (Input)

y_t = Total residual sulfur weight percent in treated coal

y_o = Residual organic sulfur weight percent in treated coal

y_p = Residual pyritic sulfur weight percent in treated coal

Y_t = Percentage of total sulfur reduction

Y_o = Percentage of organic sulfur reduction

Y_p = Percentage of pyritic sulfur reduction

Z = Residual chlorine weight percent in treated coal

II. Independent Variables (Input)

X_1 = Total sulfur in raw coal (weight percent)

X_2 = Organic sulfur in raw coal (weight percent)

X_3 = Pyritic sulfur in raw coal (weight percent)

X_4 = Time of chlorination (minutes)

X_5 = Chlorine flow rate (SCFH)

X_6 = Water-to-coal ratio by weight in the chlorination step

X_7 = Temperature of chlorination (°C)

X_8 = Steam rate in dechlorination (grams/hour)

X_9 = Temperature of dechlorination (°C)

X_{10} = Chlorine in coal before dechlorination (weight percent)

X_{11} = Time of dechlorination (minutes)

III. Regression Analysis (Output)

$C_1,\ldots,C_{11}$ -- Coefficient obtained from linear multiple regression analysis corresponding to each independent parameter $X_1,\ldots,X_{11}$

C_o --The constant coefficient for the linear fitting

Table 15. List of Notations for Linear Multiple Regression Analysis (continued)

Superscript prime (') -- predicted dependent variable for the linear fit

N -- Number of sets of data to be fitted for the specific equation

k -- Number of independent variables for that equation

$\overline{Y}$ -- Mean of a dependent variable Y

σ_{st} -- A statistical way of expressing standard deviation, called standard error =

$$\sqrt{(Y + Y')^2/N}$$

R^2 -- The regression sum of squares divided by the total sum of squares; i.e., the ratio of regression variance to total variance =

$$\Sigma\ (Y' - \overline{Y})^2 \ /\ \Sigma(Y - Y')^2$$

$R_i^2 \times 100\%$ -- The percentage of contribution explainable from variations in X_i with respect to the specific regression data fitting

$R_t^2 \times 100\%$ -- The total percentage of variance ratio R^2 explainable by the specific linear regression analysis on the selected set of parameters, =

$$(\Sigma\ R_i^2 \times 100\%)$$

$R_u^2 \times 100\%$ -- The unexplainable percentage of variance ratio based on the selected independent variables, the data, and the linear fitting.

F -- A standard statistical test leading to the confidence level or the quality of correlation =

$$\frac{R^2/k}{(1 - R^2)(N - k - 1)}$$

α -- Probability of percent data which will not fit the correlation; e.g., F = 2.84, then σ= 0.10, which means 90 percent probability the data will fit the specific correlation or a 90 percent confidence level in the engineering sense. This indicates a satisfactory correlation.

Table 16. Input data from coal PSOC-219 for the linear multiple regression analysis

Run No	PSOC Coal Type Code	y_t (Wt%)	y_o (Wt%)	y_p (Wt%)	Y_t (%)	Y_o (%)	Y_p (%)	Z (Wt%)	X_1 (Wt%)	X_2 (Wt%)	X_3 (Wt%)	X_4 (Min)	X_5 (SCFH)	X_6	X_7 (°C)	X_8 (gm/hr)	X_9 (°C)	X_{10} (Wt%)	X_{11} (Min)
107	219	0 83	0 72	0 11	68	33 3	92 1	0 22	2 56	1 08	1 40	30	0 5	0 5	74	70	450	—	60
107	219	0 87	0 63	0 22	66	41 7	84 3	0 48	2 56	1 08	1 40	120	0 5	0 5	74	70	450	11 13	60
107	219	0 72	0 35	0 37	72	67 6	73 6	0 31	2 56	1 08	1 40	120	0 5	0 5	74	70	450	11 13	60
108	219	0 73	0 28	0 45	71 5	74,	68	0 41	2 56	1 08	1 40	60	0 5	0 5	74	70	450	—	60
		(0 88)			(65 6)														
102	219	0 79	0 38	0 41	69	64 8	46 8	0 31	2 56	1 08	1 40	120	0 25	0 5	74	70	450	10 17	60
103	219	0 55	0 43	0 12	78 5	60 2	91 4	1 0	2 56	1 08	1 40	60	1 0	0 5	74	70	450	—	60
115	219	0 80	0 68	0 12	68 8	37 0	91 4	0 01	2 56	1 08	1 40	30	0 5	0 5	50	95†	500	—	60
115	219	0 65	0 62	0 03	74 6	42 6	97 9	0 45	2 56	1 08	1 40	60	0 5	0 5	50	91†	500	—	60
116	219	0 80	0 74	0 06	68 7	45 9	95 7	0 47	2 56	1 08	1 40	60	0 5	0 5	60	99†	500	8 64	60
119	219	0 68	0 65	0 03	73 4	39 8	97 9	0 12	2 56	1 08	1 40	120	0 5	0 7	74	7	500	11 5	60
																(7 5)			
120	219	0 94	0 66	0 28	63 3	38 9	80	0 14	2 56	1 08	1 40	60	0 5	0 3	74	121	450	11 33	60
																		(—)	
120	219	1 34	0 51	0 83	47 7	52 8	40 7	0 22	2 56	1 08	1 40	30	0 5	0 3	74	5	450	—	60
119	219	1 09	0 65	0 44	57 4	39 8	68 6	0 21	2 56	1 08	1 40	30	0 5	0 7	74	3 8	500	—	30
												(60)		'					
118	219	0 98	0 61	0 35	61 7	43 5	75 0	0 30	2 56	1 08	1 40	60	0 5	0 5	74	90†	450	11 97	60
118	219	0 73	0 58	0 14	71 5	46 2	90 0	0 60	2 56	1 08	1 40	60	0 5	0 5	74	11 6	500	11 97+	60
123	219	1 27	0 79	0 47	50 4	26 8	66 4	0 11	2 56	1 08	1 40	30	0 5	0 7	74	4	450	5 1	60
																(1 26)	(500)		
123	219	1 19	0 67	0 52	53 5	38 0	62 9	0 86	2 56	1,08	1 40	60	0 5	0 7	74	4	500	—	30
123	219	0 82	0 61	0 21	68 0	43 5	85 0	0 17	2 56	1 08	1 40	120	0 5	0 7	74	4	550	9 18	60
																		(11 12)+	
123	219	1 06	0 85	0 21	58 6	21 3	85 0	0 45	2 56	1 08	1 40	120	0 5	0 7	74	1	550	13 8	25
																(0 5)	(500)	(—)	
124	219	1 30	0 73	0 53	49 2	32 4	62 1	1 69	2 56	1 08	1 40	30	0 5	0 3	74	4	450	4 74+	60
																(0 4)			
124	219	0 82	0 54	0 27	68 0	50 0	80 7	0 28	2 56	1 08	1 40	60	0 5	0 3	74	4	500	8 66+	60
																(20)			
124	219	0 81	0 69	0 12	68 4	36 1	91 4	1 63	2 56	1 08	1 40	120	0 5	0 3	74	0 5	500	13 8+	60
125	219	1 08	0 71	0 37	57 8	34,2	73 6	0 54	2 56	1 08	1 40	30	0 5	0 5	74	2	500	9 65	15
125	219	1 10	0 68	0 42	57 0	37 0	70 0	1 16	2 56	1 08	1 40	30	0 5	0 5	74	1 5	500	9 65	20
138	219	0 81	0 58	0 23	68 4	46 3	83 6	0 44	2 56	1 08	1 40	60	0 5	0 5	74	9 73	500	9 86	60
								(0 43)								(9 0)			
143	219	0 84	0 54	0 28	67 2	50 0	80 0	0 5	2 56	1 08	1 40	30	1 0	0 5	74	5 35	500	8 3	30
								(0 57)											
144	219	0 99	0 74	0 23	61 3	31 5	83 6	0 86	2 56	1 08	1 40	60	0 5	0 5	85	1 20	500	11 35	30
146	219	0 85	0 64	0 20	66 8	40 7	85 7	0 36	2 56	1 08	1 40	60	0 5	0 5	74	4 75	500	15 92	30

( ) Correct values inserted directly below the incorrect data used in the analysis
† Best-guess values, listed in Tables 2 and 3 as 75 110 gm/hr
+ Data extrapolated from samples identical except for hydrolysis time

Table 17. Input data from nine coals other than PSOC-219 for the linear multiple regression analysis

Run No	PSOC Coal Type Code	y_t (Wt%)	y_o (Wt%)	y_p (Wt%)	Y_t (%)	Y_o (%)	Y_p (%)	Z (Wt%)	X_1 (Wt%)	X_2 (Wt%)	X_3 (Wt%)	X_4 (Min)	X_5 (SCFH)	X_6	X_7 (°C)	X_8 (gm/hr)	X_9 (°C)	X_{10} (Wt%)	X_{11} (Min)
111	276	1 07	1 03	0 04	79	54	98 1	0.28	5 15	2 24	2 07	120	0 5	0 5	74	94†	500	15 77 (14 81)+	60
111	276	0 89	0 87	0 02	82 7	61.2	99.0	0 17	5 15	2 24	2.07	120	0 5	0.5	74	88†	500	14 81	60
114	276	1 12	1.03	0 09	78.2	54	95 6	0.09	5.15	2 24	2 07	120	0 5	0 5	74	75†	500	——	60
114	276	0 93	0 88	0.05	82 0	60.7	97.6	0 54	5 15	2.24	2 07	60	0 5	0 5	74	104†	500	10 74	60
101	213	2 19	0 53	1 65	43	71 (71.5)	12 7	0.57	3.82	1 86	1 89	120	0 125	0 5	74	75**	400	4 58	60
106	108	0 70	0 61	0 09	78	43	96.6 (95 6)	0 97	3 13	1.07	2.06	60	0 5	0 5	74	75**	450	9 46	60
106	108	0 73	0 56	0 17	77	47 7	91.7	0 88	3 13	1 07	2 06	60	0 5	0 5	74	75**	450	9 46	60
104	108	0 71	0 45	0.26	77	58 0	87 4	0.39 (——)	3.13	1 07	2 06	60	0 5	0 5	74	75**	450	8 00	60
112	190	1 40	1 34	0 03	54 0	29 5	97 1	0 08	3.05	1 90	1 05	60	0 5	0 5	74	90†	500	9.1	60
109	190	1 28	1 26	0 02	58	33 7	98 1	0 13	3 05	1 90	1.05	120	0 5	0 5	74	90	500	12 57	60
142	342	3 33	1 60	1 69	49 1	——	66.3	0.93	6.55	1 39	5 01	60	0 5	0 5	74	1	500	——	30
127	342	2 70	1 82	0 88	58 8	——	82 4	0 99 (0 15)	6.55	1 39	5 01	120	0 5	0 5	74	4 8	500	12.83	60
129	097	0 81	0 74	0 05	34 2	11.9	86 8	0 13	1 23	0 84	0 38	60	0 5	0 5	74	1	500	——	30
129	097	1 06	0 70	0 31	13 8	16 7	18.4	0 28	1 23	0 84	0 38	30	0 5	0 5	74	0 5 (0 4)	500	——	30
131	026	1 69 (1 66)	1 22 (1 20)	0 46 (0 45)	74.6 (75 1)	41 3 (42 3)	89 1 (89.4)	0 42	6 66	2 08	4 23	60	0 5	0 5	74	5	500	8 40 (8 46)	30
131	026	2 21	1 30	0 89	66.8	37 5	79	0 20	6 66	2 08	4 23	30	0 5	0 5	74	3 3	500	——	30
135	086	0 75	0 35	0 23	38 5	44 4	58 9	0 19 (0 33)	1 22	0 63	0 56	30	0 5	0 5	74	4 (2 2)	500	——	30
135	086	0 53	0 24	0 17 (0 27)	64 7 (56 6)	61 9	69 6 (51 8)	0 33 (0 19)	1 22	0 63	0 56	60	0 5	0 5	74	2 (4)	500	8 0	30
137	(PHS 398)	0 82	0 57	0 23	72 8	——	89 8	0 82	3 01	0 46	2 26	60	0 5	0 5	74	3 5	300	8 45	30
140	High pyritic	0 65	0 56	0 09	88 4 (78 4)	——	96	0 77	3 01	0 46	2 26	60	0 5	0 5	74	1 5	500	8 24	30
140	sulfur coal pro vided by BOM	1 23	0 60	0 62	59 1	——	72 6	0 16	3 01	0 46	2 26	30	0 5	0 5	74	4 0	500	5 3	30

( ) Correct values are inserted directly below the incorrect data used in the analysis
† Best guess values, listed in Tables 2 and 3 as 75–110 gm/hr.
+ Data extrapolated from samples identical except for hydrolysis time

Case 1. The results tabulated for Case 1 represent the combined regression analyses of PSOC-219 and nine other coals. The results of this analysis provide a comparison between residual sulfur and percentage of sulfur removed for pyritic, organic and total sulfur forms. The degree of data correlation for the varying sulfur forms is represented by the total percentage-of-variance ratios (R_t^2 x 100 percent). For the sulfur forms considered, residual sulfur provides better correlation than is shown by the (R_t^2 x 100 percent for y_t' = 76.28 percent, and for Y_t' = 17.45 percent) percentage of sulfur removed in all three cases. The unexplained percentage-of-variance ratio (R_u^2 x 100 percent) for residual total sulfur (23.72 percent) indicates relatively good correlation with fitting the data to the equation and with parameter selection. The confidence level for percentage reduction of organic sulfur, Y_0' (percent), indicates that the probability of the data fitting the equation within the standard deviation is low. It appears that for the given data and equation in Case 1, the most sensitive parameter for residual organic sulfur is the organic sulfur content of the raw coal.

Case 2. The data fit for PSOC-219 coal is presented in Case 2. Poor correlation of the data with the equation is indicated by the unexplained variance ratio (R_u^2 x 100 percent) that ranges from 52 to 92 percent. The confidence levels and standard deviations for Case 2 are generally low. This is especially true of residual organic sulfur (y_0') and percentage organic sulfur reduction (Y_0') where confidence levels are <50 percent. The low confidence levels indicate poor correlation of organic sulfur within the specific equation. Sulfur reduction is not affected by the independent variables chlorine flow rate (X_5), water/coal ratio (X_6) and chlorination temperature (X_7). The only parameter showing an effect on sulfur reduction is time of chlorination (X_4), as illustrated in the specific equation column (R_4^2 x 100 percent).

Case 3. Data fitting of nine coals, excluding PSOC-219, is presented as Case 3. Values for residual sulfur forms in the unexplained percentage-of-variance ratio (R_u^2 x 100 percent) are low, showing a good linear fit of the data. The most sensitive parameters for residual organic sulfur are the organic sulfur content of the raw coal (R_2^2 x 100 percent = 63.43 percent) and chlorine flow rate (R_5^2 x 100 percent = 72.67 percent). In the case of residual pyritic sulfur (y_p), the standard deviation (0.14 percent) is large compared to the mean of the experimental data (0.28 percent), showing significant scattering in the data.

Table 18. Coefficients and statistical information obtained from linear multiple regression analysis on sulfur removal data

Dependent Variable	C_0	C_1	C_2	C_3	C_4	C_5	C_6	C_7	$\bar{Y}$	σ_{st}	F	α	Confidence Level	$R_1^2 \times 100\%$	$R_2^2 \times 100\%$	$R_3^2 \times 100\%$	$R_4^2 \times 100\%$	$R_5^2 \times 100\%$	$R_6^2 \times 100\%$	$R_7^2 \times 100\%$	$R_t^2 \times 100\%$	$R_u^2 \times 100\%$
Case 1 Data fitting of the following equations with the input X_i from all the tested coals (see Tables 16 and 17) ($Y' = C_o + C_1X_1 + C_2X_2 + C_3X_3 + C_4X_4 + C_5X_5 + C_6X_6 + C_7X_7$)																						
Y'_t	- 0 158	- 1 133	2 012	1 217	- 0 001	- 0 774	- 0 549	0 011	0 99	0 19	16 53	0 5	> 95%	15 97%	26 33%	6 25%	8 79%	12 42%	4 71%	1 80%	76 28%	23 72%
Y'_o	0 017	- 0 476	1 174	0 443	- 0 001	0 129	- 0 021	- 0 001	0 69	0 17	8 99	0 005	> 95%	5 96%	54 74%	0 01%	2 54%	0 24%	0 01%	\0 13%	63 64%	37 36%
Y'_p	- 0 193	- 0 694	0 897	0 822	0 0004	- 0 870	- 0 515	0 010	0 28	0 23	4 75	0 005	> 95%	8 76%	0 06%	10 11%	3 99%	15 51%	6 32%	3 28%	48 03%	51 97%
Y_o(%)	43 52	21 91	-31 87	-17 44	0 096	- 0 04	5 36	- 0 08	44%	13 6%	1 09	0 50	50%	4 49%	4 75%	0 72%	6 86%	0 32%	0 31%	0 0%	17 45%	82 55%
Y_1(%)	68 24	28 04	-43 09	-16 73	0 095	31 85	20 25	- 0 52	64%	9 4%	8 01	0 005	> 95%	7 15%	14 66%	11 30%	16 33%	4 26%	4 38%	2 83%	61 0%	39 0%
Y_p(%)	40 48	19 11	-18 60	-15 35	- 0 007	68 01	40 48	- 0 87	79%	16 0%	4 27	0 01	> 95%	1 42%	10 28%	0 31%	6 81%	13 27%	8 41%	4 90%	45 4%	54 6%
Case 2 Data fitting of the following equation with the input X_i from only one coal – PSOC-219, which is the coal used for parametric studies (see Table 16) ($Y' = C_o + C_4X_4 + C_5X_5 + C_6X_6 + C_7X_7$)																						
Y_t	0 650	--	--	--	- 0 0024	- 0 562	- 0 214	0 011	0 91	0 16	5 34	0 005	> 95%	--	--	--	31 19%	5 57%	2 51%	8 90%	48 16%	51 84%
Y'_o	0 819	--	--	--	- 0 00084	- 0 187	- 0 011	- 0 0006	0 62	0 14	0 47	>0 50	< 50%	--	--	--	4 99%	2 05%	0 02%	0 50%	7 56%	92 44%
Y_p	- 0 146	--	--	--	- 0 0016	- 0 381	- 0 197	0 0112	0 29	0 15	3 79	0 025	> 95%	--	--	--	20 46%	2 84%	2 67%	13 77%	39 74%	60 26%
Y'_t(%)	74 62	--	--	--	0 095	21 96	8 23	- 0 429	65%	6 2%	5 26	0 005	> 95%	--	--	--	30 90%	5 59%	2 43%	8 87%	47 78%	52 22%
Y'_o(%)	33 71	--	--	--	- 0 064	16 69	4 23	- 0 067	44%	12 5%	0 47	>0 5	< 50%	--	--	--	5 34%	1 90%	0 28%	0 001%	7 52%	92 48%
Y_p(%)	107 08	--	--	--	0 062	37 23	18 45	- 0 817	79%	12 0%	3 23	0 05	95%	--	--	--	11 89%	7 48%	12 86%	11 89%	36 0%	64 0%
Case 3 Data fitting of the following equation with the input X_i from nine coals other than PSOC-219 coal (see Table 17) ($Y' = C_o + C_1X_1 + C_2X_2 + C_3X_3 + C_4X_4 + C_5X_5$)																						
Y_t	1 60	- 0 774	1 65	0 82	- 0 0062	- 2 63	--	--	1 13	0 25	10 87	0 005	> 95%	11 93%	24 61%	10 18%	7 25%	30 51%	--	--	84 5%	15 5%
Y_o	- 1 0	- 0 786	1 71	0 72	- 0 0019	1 80	--	--	0 82	0 13	19 58	0 005	> 95%	21 0%	63 43%	0 002%	1 49%	4 80%	--	--	90 7%	9 3%
Y_p	2 40	- 0 046	0 036	0 175	- 0 0036	- 4 33	--	--	0 28	0 14	25 09	0 005	> 95%	0 32%	0 006%	16 10%	3 53%	72 67%	--	--	92 6%	7 4%
Y_t(%)	- 8 9	16 6	-31 9	3 15	0 36	84 39	--	--	63%	14 0%	4 29	0 025	> 95%	5 3%	29 7%	11 7%	15 06%	6 44%	--	--	68 2%	31 8%
Y_o(%)	87 5	38 0	-61 6	-31 1	0 19	-88 3	--	--	45%	13 0%	2 54	0 10	90%	24 3%	6 6%	1 85%	6 35%	16 86%	--	--	56 0%	44 0%
Y_p(%)	-96 3	-19 5	29 37	25 07	0 40	265 6	--	--	80%	17 0%	5 45	0 025	> 95%	0 6%	19 7%	0 073%	10 54%	42 28%	--	--	73 2%	26 8%

As described before, based on the column on unexplained variance ratio by the multiple regression analysis (R_u^2 x 100 percent), it appears that the statistical correlation fits the best for Case 3 (nine coals other than PSOC-219), second for Case 1 (combination of the above nine coals with PSOC-219), and least well with Case 2 (PSOC-219 coal only). However, it should be recognized that since PSOC-219 coal was used for extensive parametric analysis in this program, much of the input data for Case 2 are based on unoptimized conditions. Thus the statistical linear multiple regression analyses for the three cases are all meaningful in giving an overall scientific analysis of the substantial amount of data provided.

From Table 19, on the basis of the column for explained variance by the specific linear multiple regression analysis (R_t^2 x 100 percent), the only satisfactory correlation is Case 2, which involves data fitting with only PSOC-219 coal (i.e., R_t^2 = 65.4 percent). The level of confidence is also greater than 95 percent in this case, where the most sensitive parameter is chlorine in coal before dechlorination (R_{10}^2 x 100 percent = 28.30 percent). As to Case 3, Table 19 implies that temperature of dechlorination (X_9) could be a sensitive parameter (as R_9^2 x 100 percent = 34.49 percent). For Case 1 and Case 3, it can be said that there are significant factors or errors other than those involved in X_8 (steam rate in dechlorination), X_9 (temperature of dechlorination), X_{10} (chlorine in coal before dechlorination), and X_{11} (time of dechlorination) contributing to the data fitting of equation Z' (residual chlorine = $C_o + C_8X_8 + C_9X_9 + C_{10}X_{10} + C_{11}X_{11}$).

Table 19. Coefficients and statistical information obtained from linear multiple regression analysis on residual chlorine data

Dependent Variable	C_0	C_8	C_9	C_{10}	C_{11}	$\bar{Y}$	σ_{st}	F	α	Confidence Level	$R_8^2 \times 100\%$	$R_9^2 \times 100\%$	$R_{10}^2 \times 100\%$	$R_{11}^2 \times 100\%$	$R_t^2 \times 100\%$	$R_u^2 \times 100\%$
Case 1 Data fitting of the above equation with the input X_i from all the tested coals (see Tables 16 and 17) ($Y' = C_0 + C_8X_8 + C_9X_9 + C_{10}X_{10} + C_{11}X_{11}$)																
Z'	3 24	-0 005	-0 051	-0 011	0 0014	0 51	0 32	2 84	0 10	90%	12 64%	11 54%	6 03%	0 21%	30 4%	69 6%
Case 2 Data fitting of the above equation with the input X_i from only one coal – PSOC-219, which is used for parametric studies (see Table 16)																
Z	5 434	-0 0054	-0 0072	-0 09	-0 0056	0 53	0 28	5 20	0 025	> 95%	16 64%	16 67%	28 30%	3 83%	65 4%	34 6%
Case 3 Data fitting of the above equation with the input X_i from nine coals other than PSOC-219 coal (see Table 17)																
Z'	3 325	-0 0076	-0 00676	0 04	0 0088	0 50	0 28	2 07	0 25	75%	8 29%	34.49%	0 007%	2 53%	45 3%	54 7%

Design and Equipment Specifications for Bench-Scale Equipment and Mini-Pilot Plant (1.2)

Bench-scale testing of the coal desulfurization process will be conducted in Phase II on a scale of 2000 grams of coal per batch, using chlorinator, hydrolyzer and dechlorinator equipment representative of equipment suitable for engineering scale-up.

Parallel with the bench-scale equipment test program, a continuous flow mini-pilot plant will be constructed for an integrated equipment operation. Coal will be fed at a nominal rate of 2000 grams per hour from the pulverized coal feed hopper through the chlorination, hydrolysis and dechlorination stages. The coal desulfurization mini-pilot plant is represented as an integrated equipment unit in Figures 6, 7, and 8. Major equipment units are portrayed in Figure 9 (ground coal hopper and blender), Figure 11 (chlorinator), Figure 13 (hydrolyzer), Figure 15 (rotary vacuum filter), Figure 16 (flash dryer), Figure 17 (dechlorinator) and Figure 18 (clean coal storage hopper).

Design Considerations

The layout of the mini-pilot plant takes advantage of gravity flow wherever possible to reduce the number of mechanical transporters of coal and slurries. Except for the dechlorinator, the progress of coal is vertically up or down through the system, resulting in a tall narrow structure that can be serviced easily by one overhead hoist and a three-level catwalk on either side.

Design of individual units is discussed in the following paragraphs.

A rotating screw feeds coal from the storage hopper to the chlorinator. This method was chosen over a simple gravity feed for two reasons. The screw gives close control over the feed rate, and it acts as a one-way valve to prevent back-flow of gases to the hopper.

The chlorinator and hydrolyzer are lined with acid-resistant brick instead of, for example, tantalum cladding. The brick lining results in a heavy, bulky vessel, but the cost of brick is about one-tenth the cost of the cheapest cladding process.

Rotary air locks were chosen as the means of isolating major units at four places. The required rotary air locks are smaller than any now available, and will have to be specially fabricated of teflon. They are, however, the best method of preventing contamination of one part of the process by the effluents of another.

The flash drier that removes the moisture remaining in the coal cake after filtering is included for three reasons. It provides to the dechlorinator coal that is dry, so that chlorine is more easily recovered from the process off-gases. The coal is fluffy and gives up its chlorine more readily. The energy required to heat the dechlorinator tube is reduced, since the coal is dried by flue gases drawn from the dechlorinator burners.

The dechlorinator was first planned as a direct-fired unit, heated by combustion products from an external burner. Such a unit, however, would have exposed the dry, finely-ground coal to a large volume of gases,

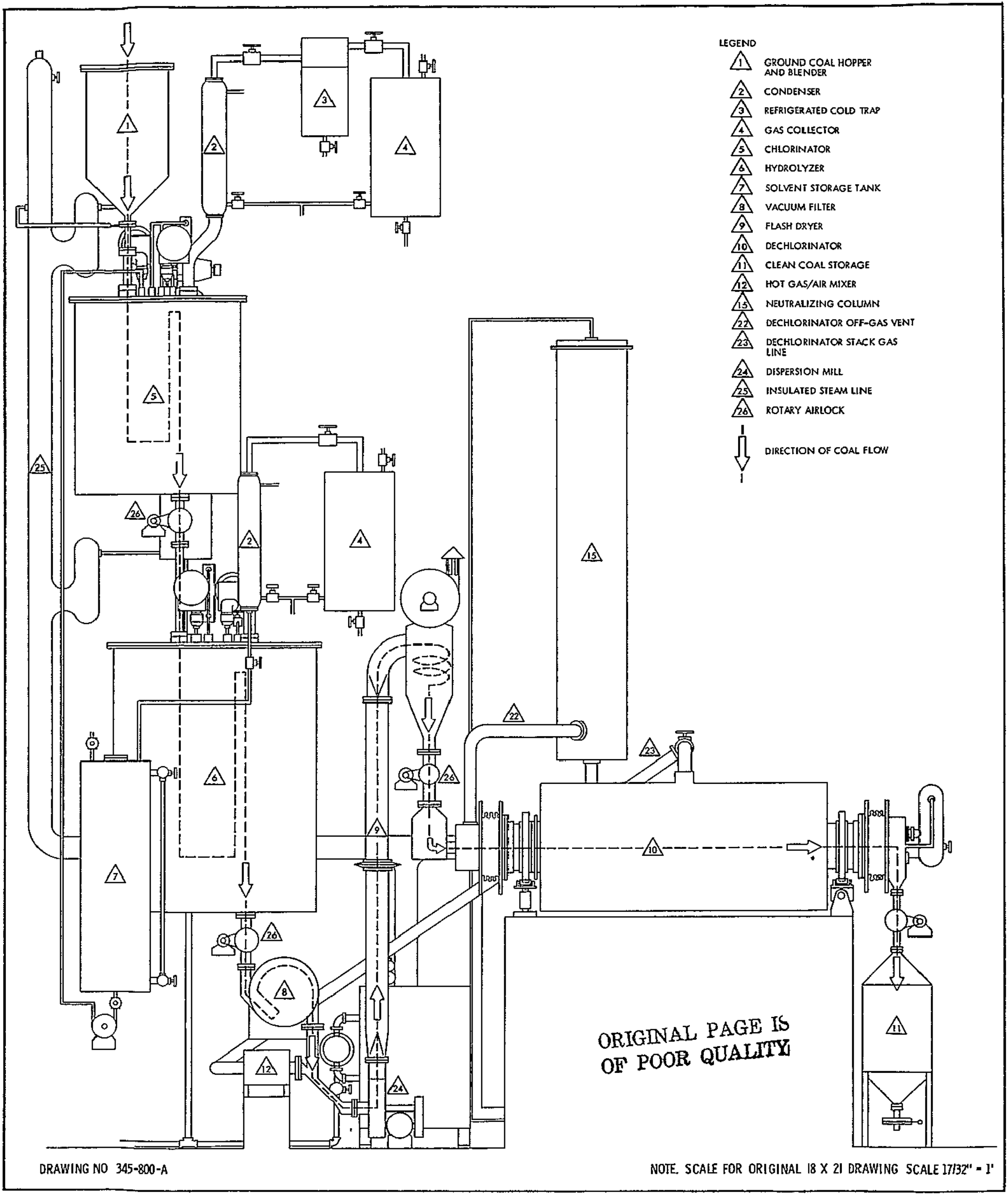

Figure 6. JPL coal desulfurization mini-pilot plant -- side elevation

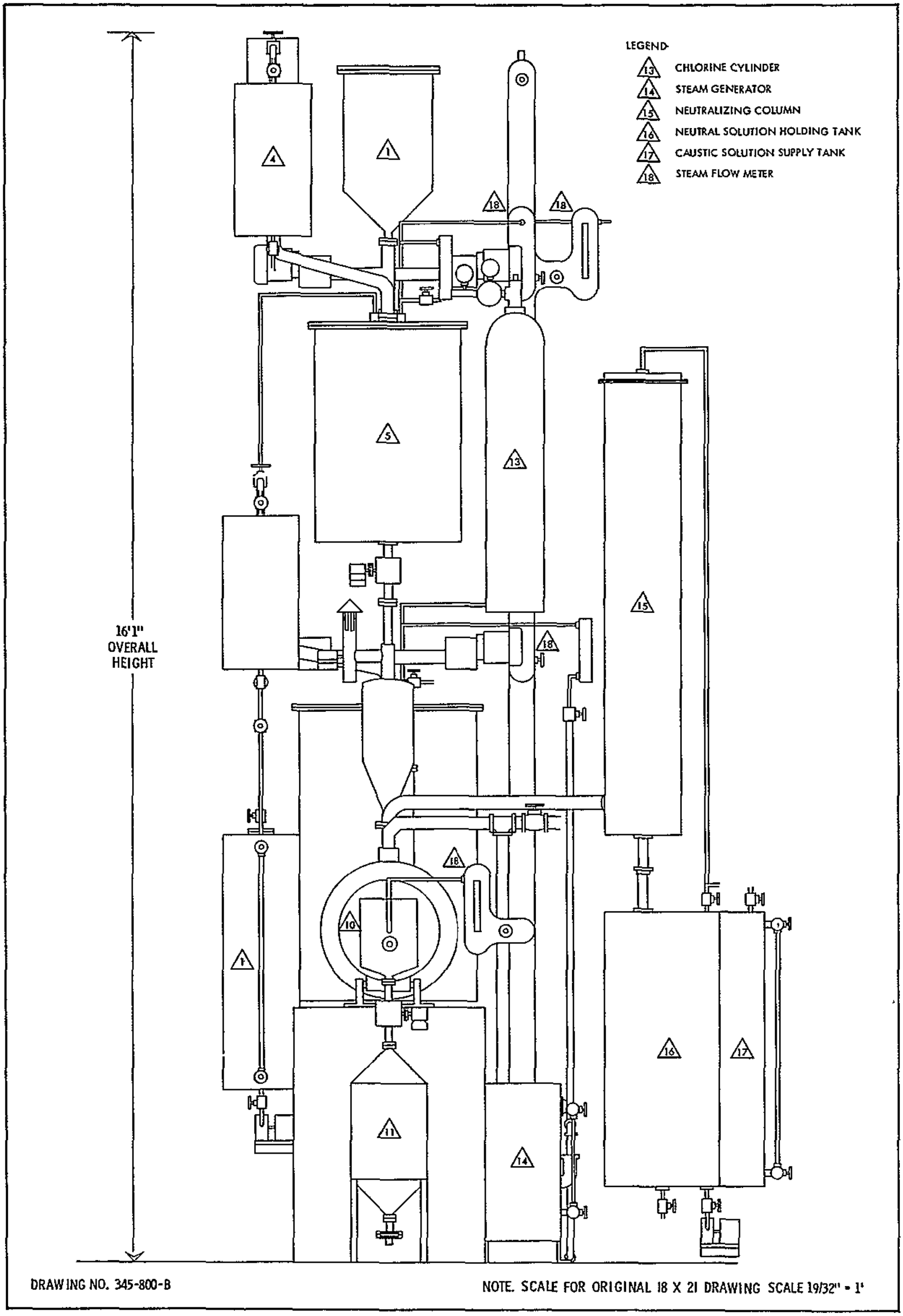

Figure 7. JPL coal desulfurization mini-pilot plant -- frontal view

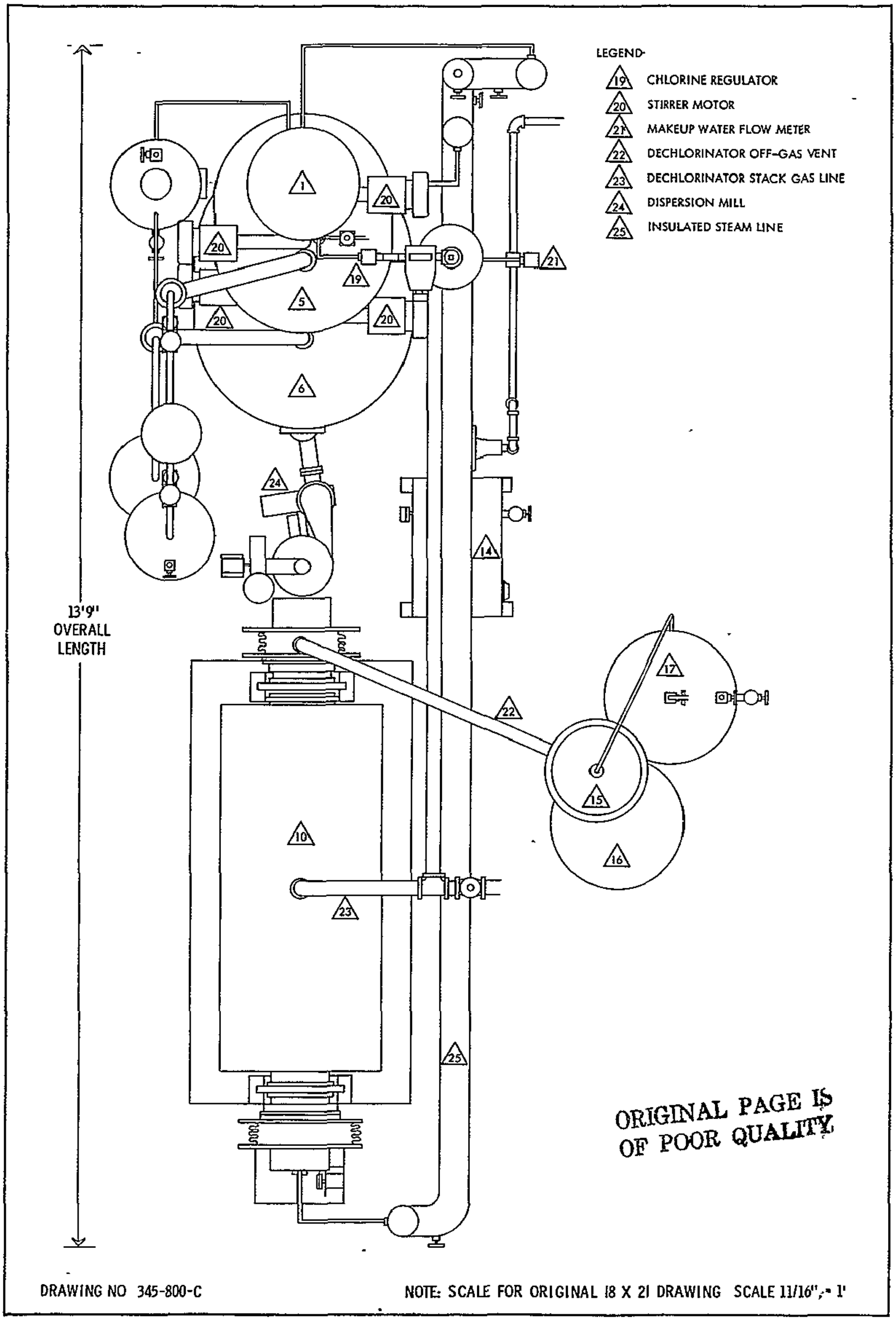

Figure 8. JPL coal desulfurization mini-pilot plant -- plan view

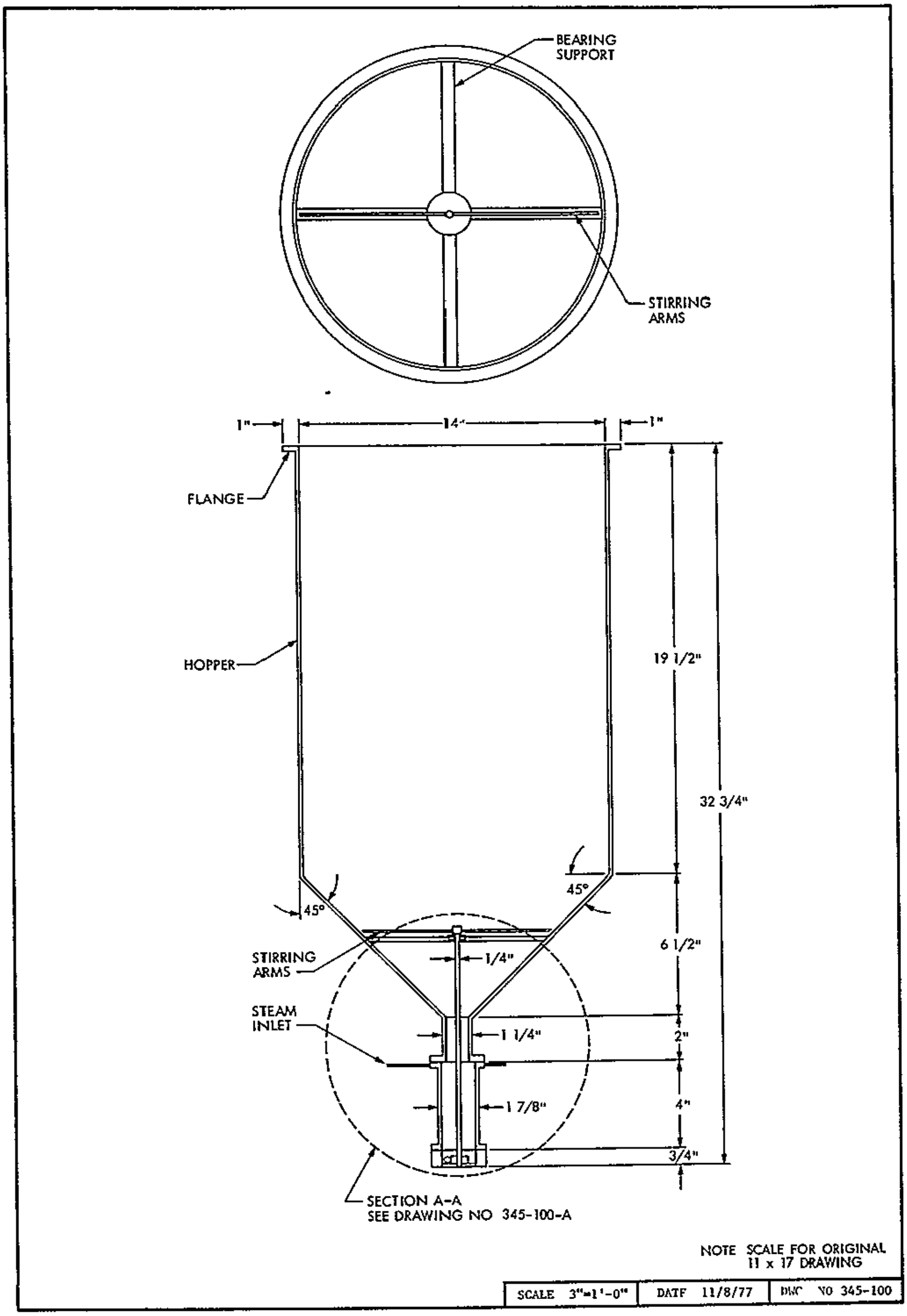

Figure 9. Ground coal hopper and blender

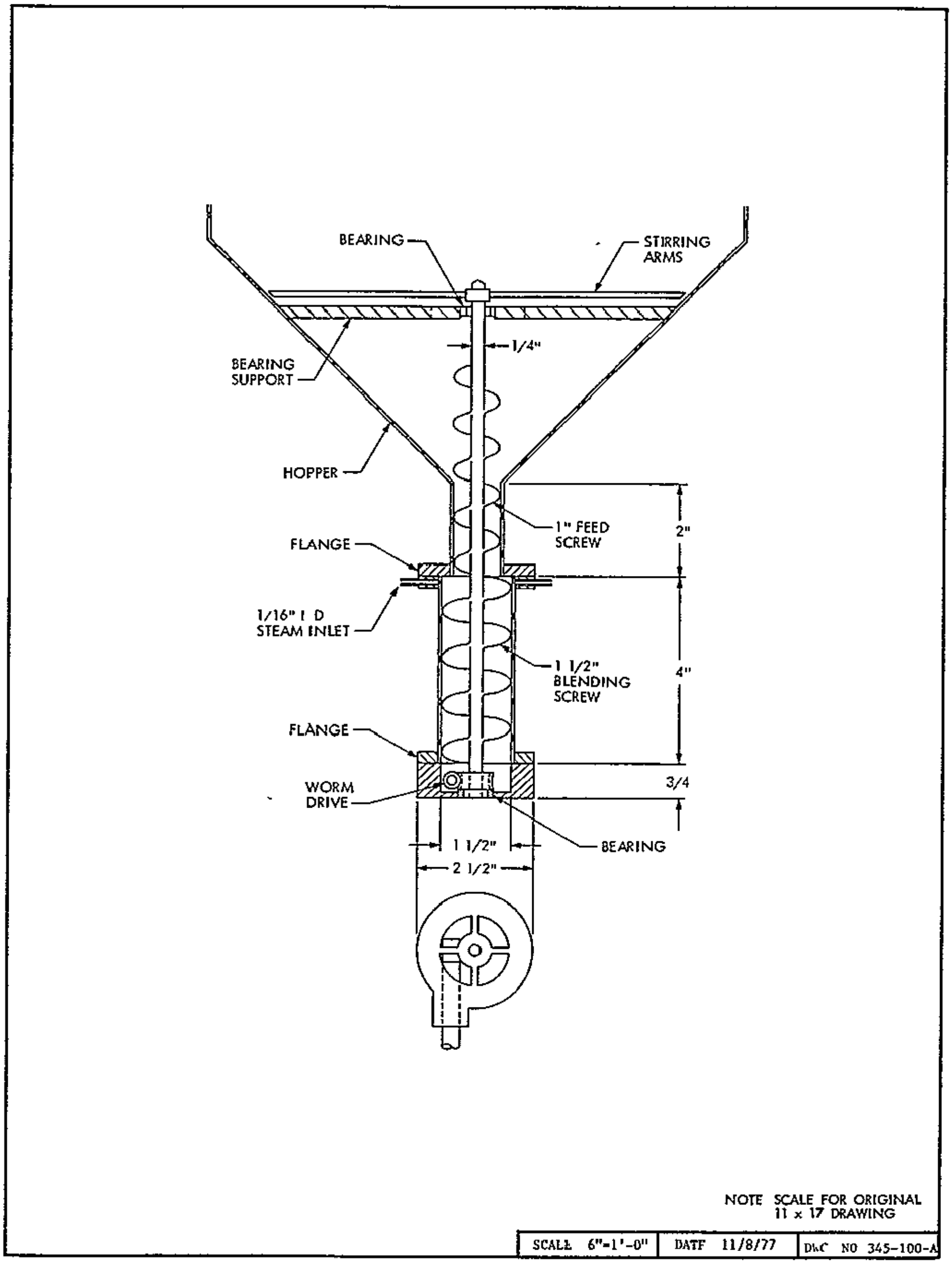

Figure 10. Ground coal hopper and blender -- Section A-A

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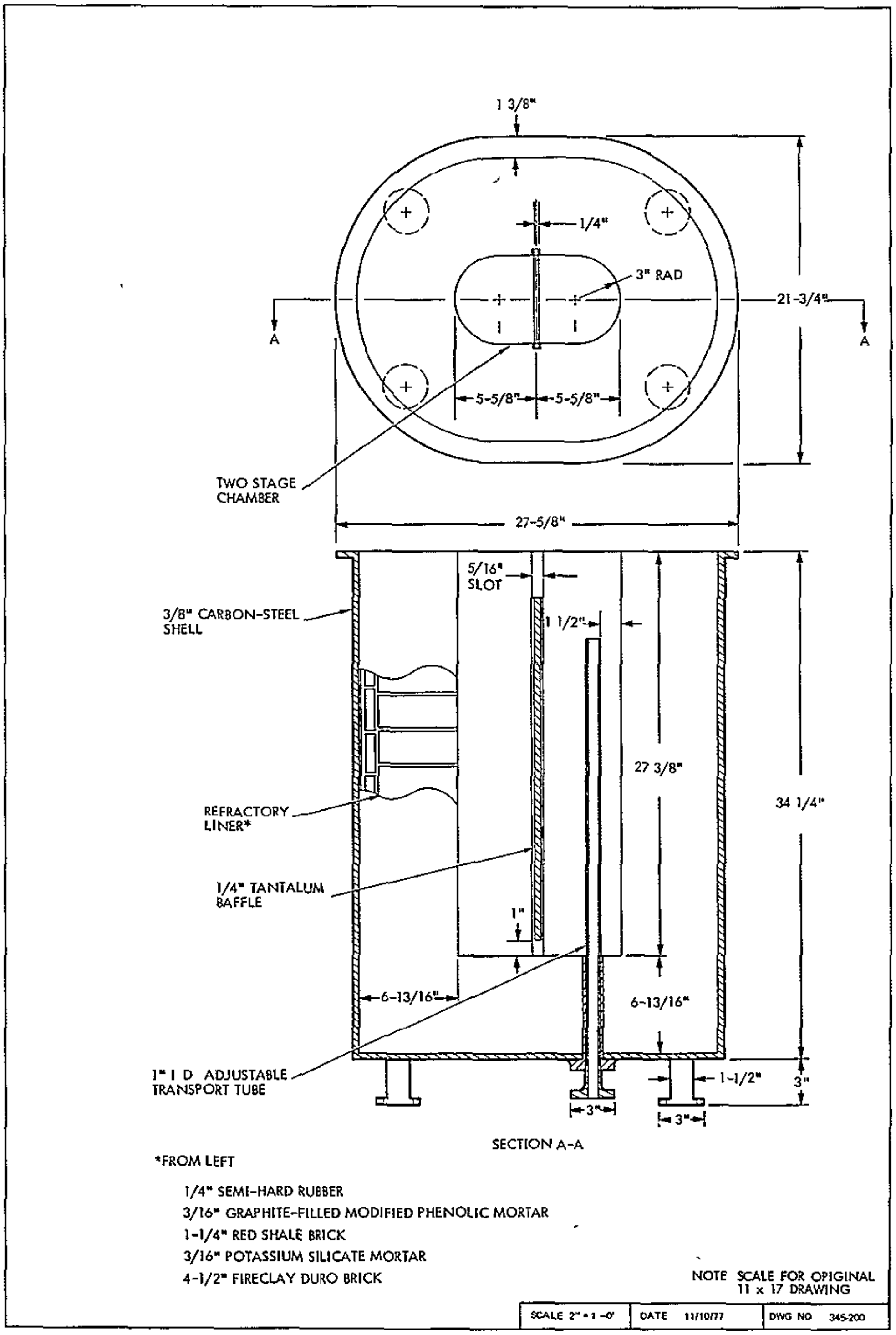

Figure 11. Chlorinator

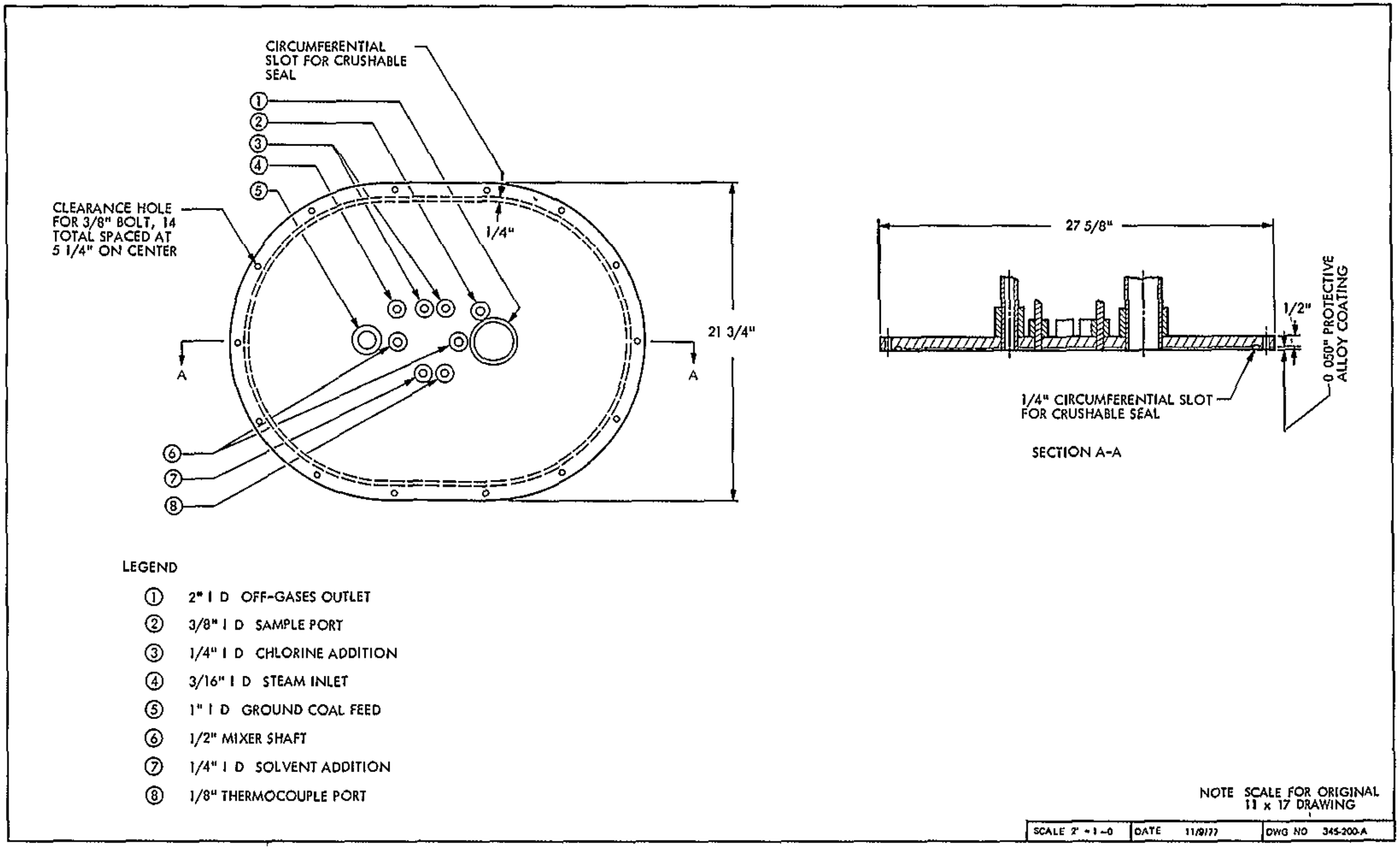

Figure 12. Chlorinator reactor head

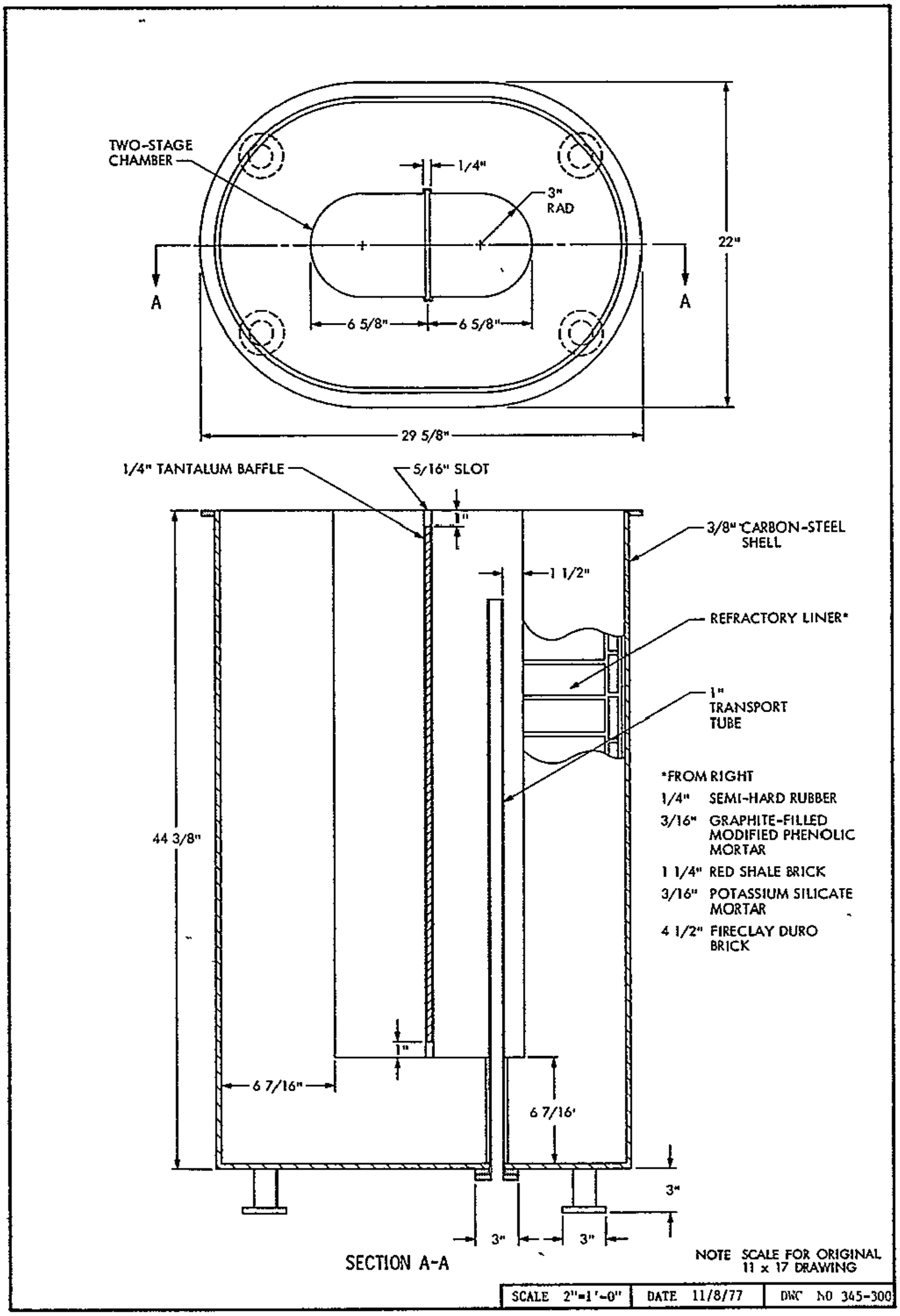

Figure 13. Hydrolyzer

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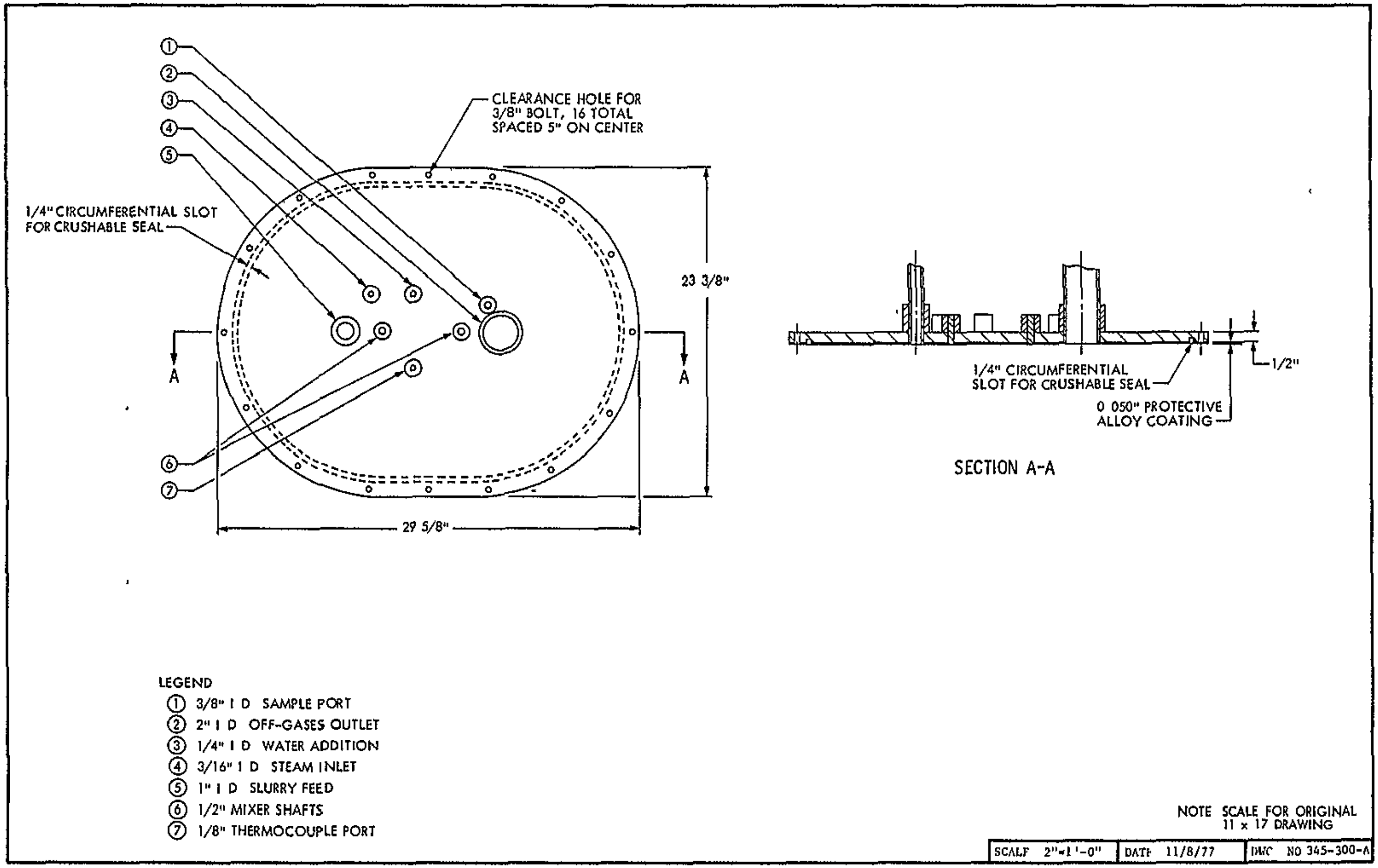

Figure 14. Hydrolyzer reactor head

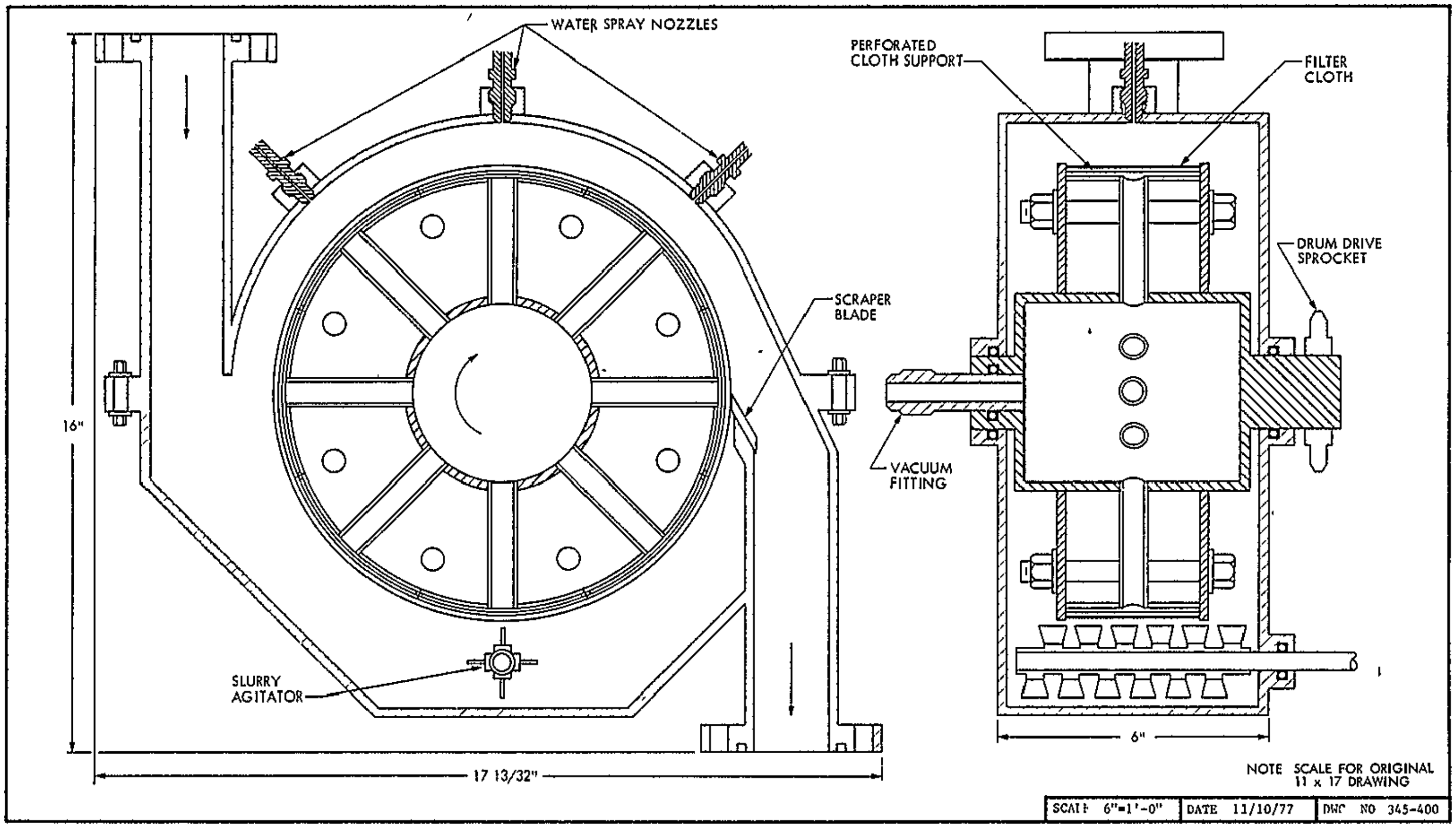

Figure 15. Rotary vacuum filter

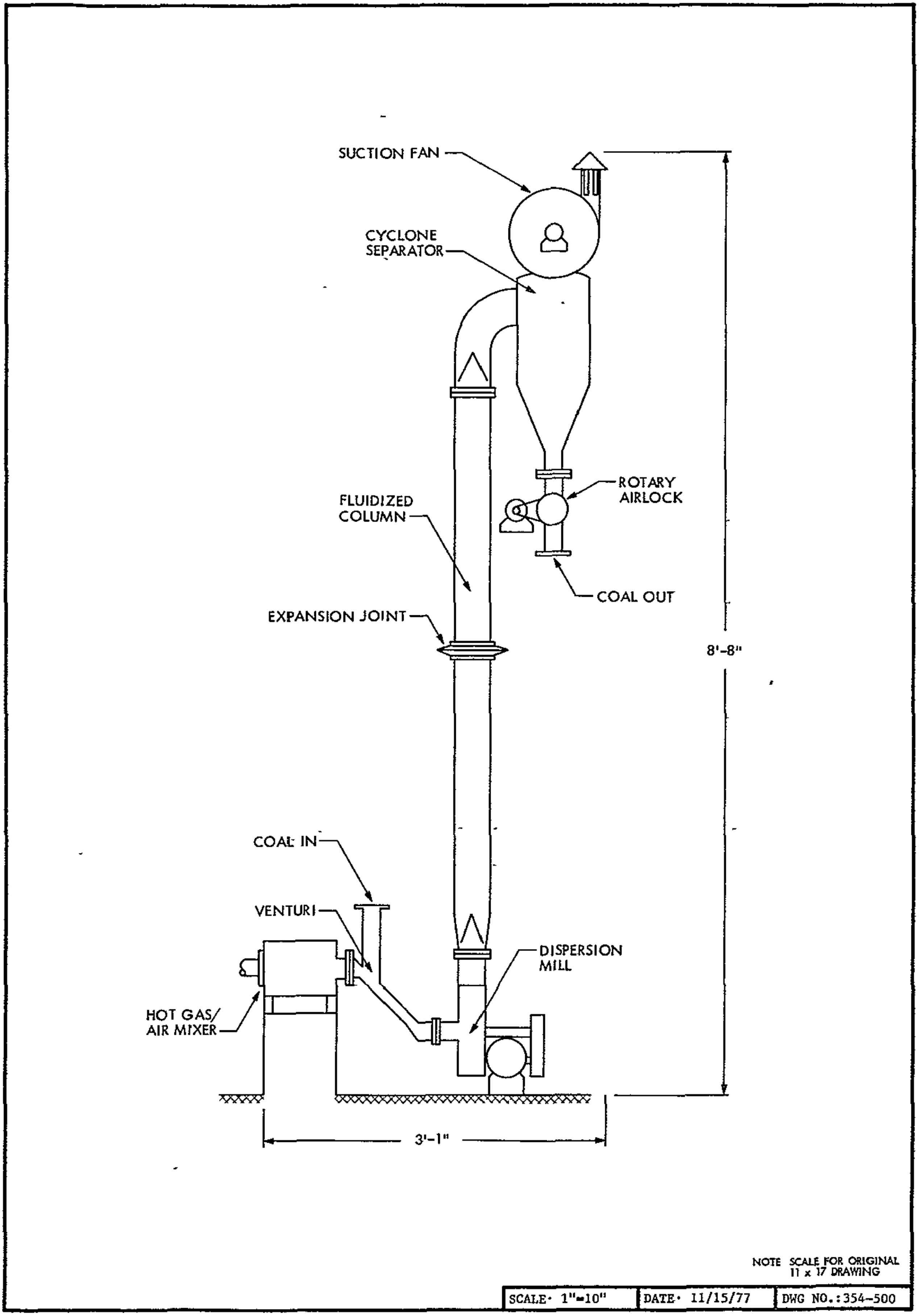

Figure 16. Flash dryer

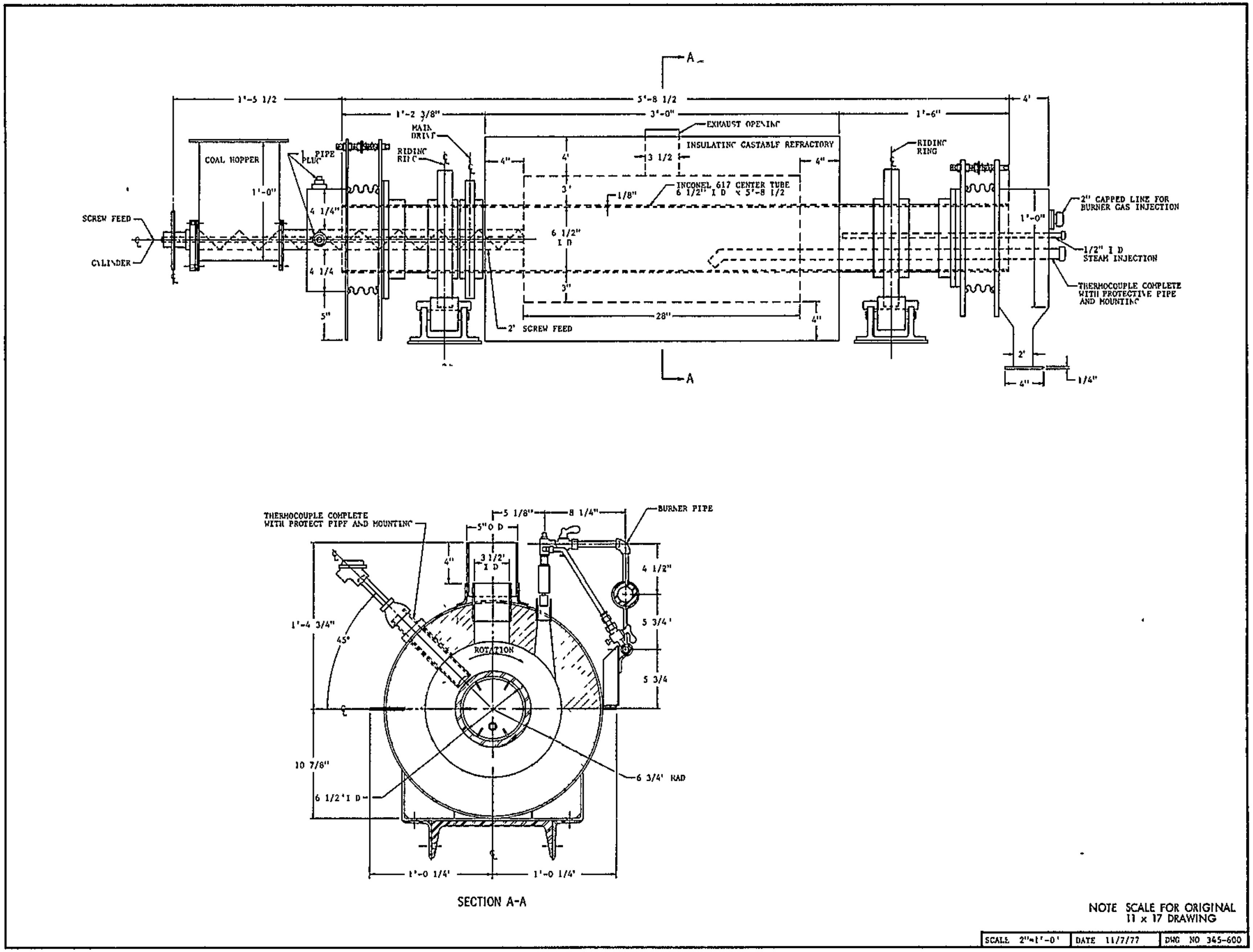

Figure 17. Dechlorinator

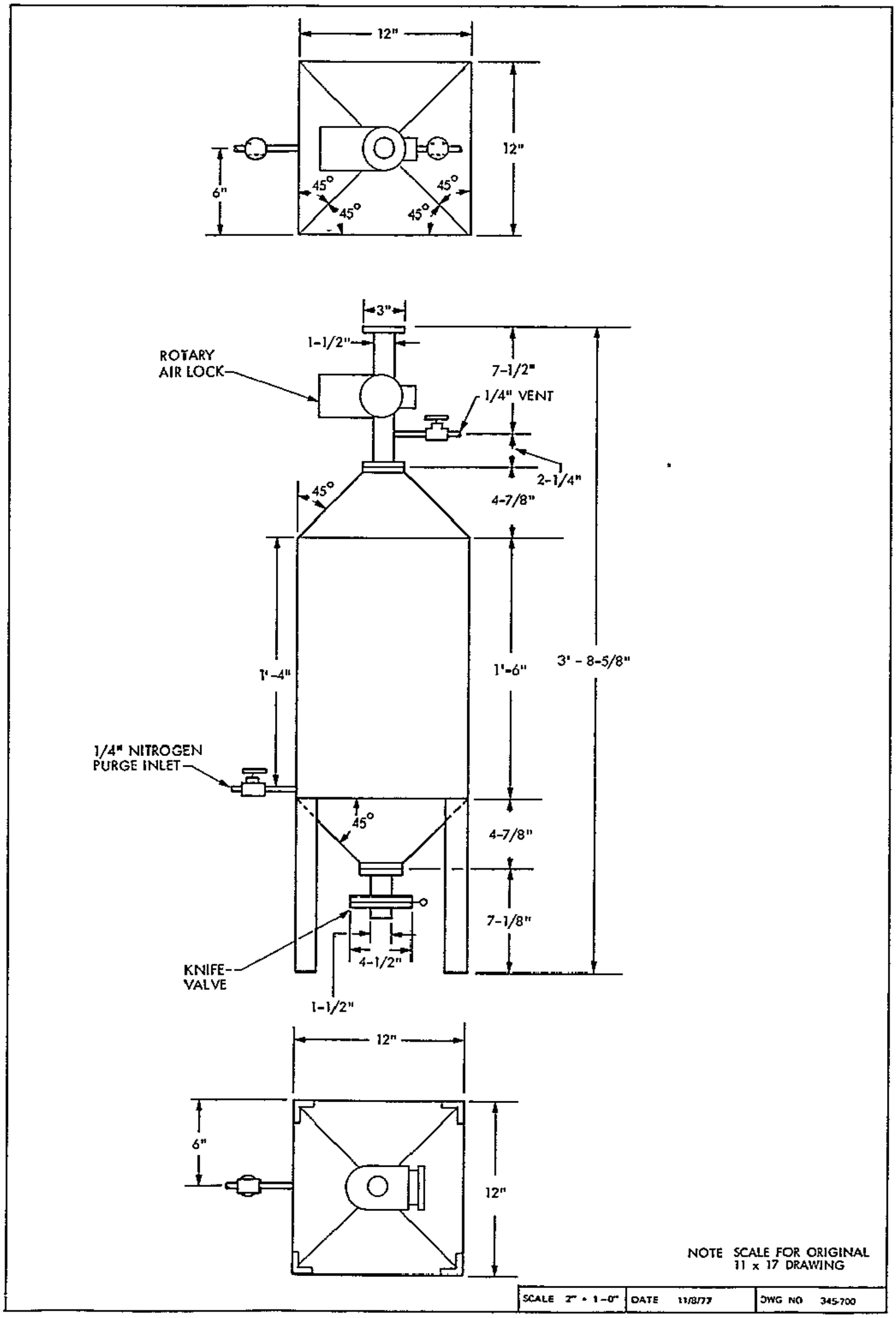

Figure 18. Clean coal storage hopper

which would have complicated HCl and product coal recovery with minimum losses. Therefore, an indirect fired calciner will be used. An existing unit that is indirectly heated by natural gas burners may meet the requirements for the dechlorinator.

Description of Major Units

The coal desulfurization mini-pilot plant is comprised of seven major units (Table 20, Figures 6, 7, and 8) and several auxiliary systems to support them. A parts list for the mini-pilot plant is included as Table 21. The seven major units, listed consecutively from start to completion of the desulfurizing process, are as follows:

1. Ground coal hopper and blender
2. Chlorinator
3. Hydrolyzer
4. Vacuum filter
5. Flash dryer
6. Dechlorinator
7. Clean coal storage hopper

Ground Coal Storage Hopper and Blender. This unit is a cylindrical bin with an air-tight lid and a conical bottom. Flanged to the bottom of the cone is the housing for two vertical feed screws and the worm and pinion that drives them. The screws are on a single shaft, with the smaller of the two extending into the cone of the storage hopper. It is supplied with ground coal by the rotation of sweeper arms attached above it to the same shaft. The upper screw feeds coal directly into the lower screw, which is larger in diameter but of the same pitch, so it does not operate at a choked or completely full condition. The increase of internal volume permits the introduction of steam through nozzles at the upper end of the large screw. The steam moistens the coal and is blended with it as it passes along the screw.

Chlorinator. The dechlorinator is an oval steel unit that is lined with refractory brick. The brick forms a narrow, deep retort in the center, which is separated into two equal parts by a baffle that leaves a clearance slot below the top and above the bottom. In one of the chambers formed by the baffle is a standpipe that extends through the bottom of the chlorinator to the next unit. The chlorinator is closed at the top with a sealed lid that allows entry, through various flanges and bosses, of wetted coal from the blender, steam, solvent, chlorine gas, stirring shafts, and a thermocouple, and that permits samples to be taken and evolved gases to escape.

Hydrolyzer. From the chlorinator, the coal, now in a slurry with water and solvent, passes vertically downward to the hydrolyzer. This is another, larger, oval steel unit lined with refractory brick and divided into two compartments by a baffle. The hydrolyzer has a standpipe through the bottom and a sealed lid similar to that of the chlorinator. Introduced through the lid are the slurry of chlorinated coal, water, steam, stirring shafts, and a thermocouple. Ports are also provided for sampling and for escaping vapors and gases.

Table 20. Major units -- coal desulfurization mini-pilot plant

System/Treatment	Manufacturer/Availability	Specifications
Ground Coal Storage Hopper and Blender		
Coal storage and initial wetting	JPL/4 weeks	Two ft^3 cylindrical hopper equipped with stirring arms to prevent bridging
Blender	Bay City Fabrication, Inc Long Beach Ca 8-10 weeks	One inch diameter vertical feed screw from hopper to blender nominal feed rate, 2 kilograms/hr One and one half inch diameter vertical blender screw Two steam jets at junction of feed screw and blender screw
Chlorinator		
Agitation of ground coal in a heated solution of water solvent and chlorine	Pennwalt Corp, Philadelphia Penna	Steel vessel lined with acid resistant brick Two chambers Separable head ported for wetted coal solvent steam chlorine mixers thermocouple sampling and off gases One inch standpipe in downstream chamber for flow to next unit Working volume 72 ft^3 Coal residence 30 to 120 min Maximum operating temperature, 100°C Maximum operating pressure 100 psig
Hydrolyzer		
Agitation of chlorinated coal slurry in a hot water bath Recovery of evolved solvent	Pennwalt Corp Philadelphia Penna	Steel vessel lined with acid resistant brick Two chambers Separable head ported for coal slurry water steam mixers, thermocouple sampling and off gases One inch standpipe in downstream chamber for flow to next unit Total volume 1 43 ft^3 Coal residence 30 to 120 min Maximum operating temperature 100°C Maximum operating pressure atmospheric
Vacuum Filter		
Filtration of coal slurry Clean water wash of filter cake	Jackson Enterprises Orillia Ontario Canada 16 24 weeks	Cloth covered drum 3 inches wide, 10 inches diameter, partially submerged in coal slurry Drum center evacuated filtrate stored in receiver Slurry flow 21 to 71 ft^3/hr Case is side ported to allow air flow to drum
Flash Dryer		
Hot Gas/Air Mixer	JPL Pasadena Ca 3 weeks	Mixing chamber with thermostatically controlled damper to blend dechlorinator flue gases and air
Venturi	JPL Pasadena Ca 3 weeks	Wide narrow throat cross sectional area 75 in^2
Dispersion Mill		High RPM center fed combination chopper/fan Case width and outlet diameter 3 inches
Fluidized column	JPL Pasadena Ca 2 weeks	Four inch diameter vertical tube Expansion bellows in center Upper end in 90° curve to enter separator
Cyclone Separator		Tangential entry separator Bottom center exit for product Top center exit for air Blower capability 27 scfm
Dechlorinator	C E Raymond/Bartlett Snow Chicago Ill Available	Two concentric tubes outer lined with castable firebrick inner rotatable Inner tube diameter 6 1/2 inches length 85 1/8 inches Inside tube heated by seven natural gas burners Maximum operating temperature 2000°F Rotational speed of inside tube variable from 1 16 to 11 6 RPM Cylinder material Inconel 617 Feed screw capability 4 kilograms/hr 0 37 to 7 29 RPM
Clean Coal Storage Hopper	JPL Pasadena Ca 4 weeks	Two ft^3 rectangular hopper equipped with nitrogen purge and blade type emptying valve

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Table 21. Parts list -- coal desulfurization mini-pilot plant

Ground Coal Hopper/Blender
- Head
- Hopper
- Bearing Support
- Bearing
- Sweeper Arms
- Feed Screw
- Blending Screw
- Steam Nozzles
- Screw Housing
- Steam Control Valve
- Steam Flow Meter
- Screw Drive Gears
- Screw Drive Motor

Chlorinator
- Head
- Connecting Tube (to Hopper)
- Mixer Motors (2)
- Stirring Shafts & Paddles (2)
- Steam Control Valve
- Steam Flow Meter
- Sampling Valve
- Thermocouple
- Chlorine Cylinder
- Chlorine Regulator
- Chlorine Feed Lines
- Solvent Return Line
- Condenser Feed Line
- Condenser
- Refrigerated Gas Trap
- Gas Collector
- Gas Trap Isolation Valves (2)
- Gas Trap Drain Valve
- Water Supply Valves (2)
- Gas Collector Water Fill Valve
- Gas Collector Bleed Valve
- Gas Trap Connecting Lines (2)
- Water Lines
- Chlorinator Vessel
- Baffle
- Rotary Valve

Hydrolyzer
- Head
- Connecting Tube (to Chlorinator)
- Mixer Motors (2)
- Stirring Shafts & Paddles (2)
- Steam Control Valve
- Steam Flow Meter
- Sampling Valve
- Thermocouple
- Water Control Valve
- Water Flow Meter
- Condenser Feed Line
- Condenser
- Gas Collector
- Gas Collector Isolation Valve
- Gas Collector Connecting Lines
- Water Supply Valves (2)
- Water Lines
- Gas Collector Water Fill Valve
- Gas Collector Bleed Valve
- Solvent Return Valve
- Solvent Return Lines
- Solvent Tank
- Solvent Tank Outlet Valve
- Solvent Pump
- Solvent Bleed Valve
- Hydrolyzer Vessel
- Baffle
- Rotary Valve

Vacuum Filter
- Vacuum Filter (1 Unit)

Steam Boiler
- Steam Generator (1 Unit)
- Insulated Steam Lines
- Bleed Valve

Miscellaneous
- Water Lines
- Electrical Panel & Wiring
- Flanges
- Pipe Fittings
- Tube Fittings
- External Insulation

Flash Dryer
- Hot Gas/Air Mixer
- Venturi
- Dispersion Mill
- Fluidized Column
- Cyclone Separator
- Blower
- Rotary Valve
- Combustion Gas Bleed Valve
- Combustion Gas Feed Line

Dechlorinator
- Dry Coal Hopper
- Feed Screw
- Feed Screw Motor
- Dechlorinator (1 Unit)
- Closed Funnel
- Rotary Valve
- Off Gas Exhaust (to Neutralizing Column)
- Steam Control Valve
- Steam Flow Meter
- Thermocouple
- Trunnion Elevator Screw
- Trunnion Pivot

Clean Coal Hopper
- Hopper (1 Unit)
- Knife Valve

Off-Gas Neutralizer
- Neutralizing Column
- Caustic Solution Supply Tank
- Neutral Solution Holding Tank
- Caustic Solution Pump
- Connecting Line (to Holding Tank)
- Neutral Solution Drain Valve
- Caustic Solution Bleed Valve
- Caustic Solution Fill Valve
- Connecting Line (from Caustic Tank)

Vacuum Filter. The coal slurry, now consisting largely of coal and water, flows through the hydrolyzer standpipe and is partially dried in the vacuum filter. The filter consists of a vertical drum rotating one-third submerged in the slurry from the hydrolyzer. The ends of the drum are solid, and the cylinder is perforated and covered with porous filter cloth. The perforated cylinder is supported by hollow tubes leading to a vessel in the center of the drum. The central vessel can be evacuated, thus drawing the slurry to the porous filter cloth and extracting water from it as the drum rotates. The partially dried cake of coal is removed from the drum by a blade and is carried by gravity to the next unit.

Flash Dryer. The flash dryer, which receives the coal cake from the vacuum filter, consists of five sub-units: a hot gas/air mixer, a venturi, a dispersion mill, a vertical fluidized column, and a cyclone separator. The hot gas/air mixer provides a stream of heated gases to the venturi, into which the coal cake is discharged. The venturi reduces the gas pressure and increases its velocity to carry the coal cake into the dispersion mill. This mill is a center-fed chopper and fan that pulverizes the cake and throws it out a tangential tube into the fluidized column, where the hot gases that carry it upward complete the drying process. From the fluidized column the coal, now as dried particles, enters the cyclone separator. There it is thrown against the walls of the separator and falls into the dechlorinator hopper, while the hot gases are drawn to the center and exhausted through a blower.

Dechlorinator. The dechlorinator consists of an abrasion, corrosion and heat resistant cylinder (Inconel 617 or other stainless steel alloy) that rotates inside a larger stationary cylinder having a refractory-brick lining. Between the two cylinders is a toroidal cavity that serves as a fire box to heat the inner one. Heating is accomplished by injecting a mixture of natural gas and air through nozzles and burning it in the cavity. Dried ground coal is moved into the inner cylinder by a feed screw and moves through it because the dechlorinator is inclined slightly toward the clean coal hopper. The coal is tumbled, as it moves, by flights along the inner wall. At the end of the cylinder it falls into a closed funnel through a rotary air lock and drops to the clean coal storage. The closed funnel serves as a closure for the dechlorinator and is ported for the entry of steam, combustion gases and a thermocouple.

Clean Coal Storage Hopper. This is a closed bin, isolated from the dechlorinator by a rotary valve and having a knife valve at the bottom to remove clean coal. It is provided with a nitrogen purge to prevent oxidation of the coal heated in the dechlorinator.

Process Equipment Operation

Start-up of the mini-pilot plant requires bringing the equipment to operating temperature and introducing flow through the chlorinator and into the hydrolyzer and dechlorinator to establish the equipment inventories of material preliminary to establishing steady-state operating conditions. Phasing of feed materials may be required to avoid problems of caking, etc. Mechanical operation of the equipment is described as follows:

As the coal enters the blending screw it is wetted with steam from small nozzles in the blender flange. Steam cannot enter the ground coal hopper because the feed screw, operating choked, blocks the passage with coal. The steam mixes with the coal in the blender and the warmed and wetted coal is dropped straight down a tube into the chlorinator.

The chlorinator has two chambers, separated by a baffle that allows gas pressure to equalize at the top and liquid to flow across the bottom. One chamber receives the coal from the blender, the other discharges it through a standpipe to the hydrolyzer below. In the chlorinator the coal falls into a liquid composed of water, solvent and chlorine, where it is continously agitated by mixers. The blades of these mixers are so arranged that they contribute nothing to the general flow of the slurry. The flow, and thus the residence time of the coal in the total system, is governed by the rate at which water and solvent are added and by the height of the standpipe. In this step of the process, the sulfur contained in the coal is oxidized by the chlorine to water-soluble sulfate compounds. The reaction occurs at temperatures in the range of 50 to 100°C, with heat supplied by steam injection. Solvent vapors, HCl and Cl_2 are contained in the chlorinator by a water-cooled reflux condenser. Gases escaping the reflux condenser are contained by a refrigerated cold trap. A small amount of inert gas passes into a gas holder for sampling and analysis.

The chlorinated coal slurry leaves the chlorinator by overflow into a stand-pipe that connects to the hydrolyzer. Before reaching the next major unit, the hydrolyzer, the coal slurry passes through a rotary air lock that blocks the backflow of hydrolyzer off-gases to the chlorinator.

The hydrolyzer provides a hot-water treatment of the coal slurry in which the chlorine/sulfur compounds are washed from the coal and the solvent is flashed from the slurry. As in the chlorinator, the slurry is constantly agitated and steam heated. Retention times are controlled by water and coal slurry feed rates. The temperature of the hydrolyzer is controlled by steam injection to flash the organic solvent to a condenser and into a solvent recovery tank for recycle to the chlorinator.

The washed desulfurized coal flows through a stand-pipe, through a rotary air lock to a rotary vacuum filter. The rotary air lock is required to seal the vacuum filter from the flashed solvent vapors.

In the vacuum filter the coal-water slurry enters a bath in the lower third of the filter case. The slurry is continuously agitated to keep the coal in suspension, so that it will be drawn to the filter drum in the vacuum-induced flow. As the drum rotates, the coal adheres to it and forms a thin, damp cake. This cake is flushed with fresh water at the top of the rotation to displace sulfate-containing wash water from the coal. The coal is redried as rotation continues and is finally scraped off the surface of the drum and dropped into the flash dryer venturi.

The coal is carried through the flash dryer, in a mixture of combustion gases and air, drawn by means of a blower fan at the exit of the cyclone separator. The mixture of wet coal and gases is directed to a dispersion mill. There, large lumps are pulverized and thrown upward into a vertical duct and then passed to a cyclone separator for separation of coal particles and gases. Most of the drying occurs in the dispersion mill, with drying completed in the vertical duct. A rotary air lock below the separator prevents back-flow of gases from the dechlorinator feed hopper.

At the bottom of the hopper is a feed screw that moves the dried coal into the dechlorination tube. Here the coal enters an atmosphere of dry steam, where it is dechlorinated at temperatures up to 500°C. The dechlorination tube rotates and tumbles the coal grains so that all are exposed to the dry steam. The tube is indirectly fired by gas burners to provide required temperatures. Retention time of coal in the calciner, nominally 20 minutes, is governed by the tube inclination and rotational speed. Off-gases consist primarily of a mixture of steam and HCl. A caustic scrubber will contain the HCl in the pilot plant. In a commercial unit, the HCl would be recovered for recycling to a Kel-chlor plant. Flue gases from the calciner are directed to the flash dryer, providing high thermal efficiencies for the combined calciner - flash dryer operation.

Coal from the calciner is discharged through a rotary air lock to a coal hopper and contained under a nitrogen blanket. Quantities are small enough that the hot coal will be cooled by natural convection of air to the coal hopper.

Bench-Scale Equipment for Batch Tests

A batch-mode screening program will parallel the procurement and construction of the mini-pilot plant. The effects of chlorinating under pressure will be studied, as well as solvent-to-coal ratios, and chlorination temperatures. Coal particle sizes will be varied in conjunction with changes in residence times, to find the most economical grind for desulfurization.

Two batch-mode programs will require at least one more vessel of the steel-walled, brick-lined type, that will accept batch amounts of 2 kilograms of coal. Chlorination and hydrolysis can be accomplished in sequence in the same vessel. Batch filtration followed by operation of the calciner in a batch-type operation will be utilized for the bench-scale experiments.

The chlorinator-hydrolyzer unit(s) can be obtained from the vendors and installed within 3 months after the start of the program in Phase I. The existing calciner will be modified to fit the continuous-process system and also be used in batch-process testing if possible. Testing and construction will thus be parallel, providing maximum development of the process in a short time.

Immersion Tests

Immersion testing of four types of brick and two mortars was successfully completed. The evaluation continued over a 6-week period during September and October. The list of materials tested covered only the face courses of the chlorinator and hydrolyzer vessel designs solicited from the Stebbins Engineering and Manufacturing Company and Pennwalt Corporation.

This work was undertaken to support the 4-month design period of Task 1.2. Equipment specifications are that acid resistant brick construction is to be used, in conjunction with a plastic or rubber membrane between the brick and the steel. The specifications further provide for acceptance testing of specific materials under reactor conditions of acid and organic solvent.

The scope of the testing program was set jointly by JPL and the vendors. Both engineering firms had many years of experience in process design involving chlorine and were confident in their acid-resistant brick designs for application to the JPL coal desulfurization process. Some inexpensive materials were specified in the hope that their adequacy would be proven by tests; for example, K14 mortar in lieu of Stebbins mortar AR20C. Also, since maximum temperature and pressure for the chlorinator and hydrolyzer were relatively low, other materials were feasible for construction and needed to be tested. The consideration of redshale brick for the face course as well as the second course is a case in point.

Immersion testing is a form of acceptance testing in which materials are submerged in process fluids, and afterwards examined for changes in structural properties. For bricklined vessel construction, the method is based on the premise that easily measured changes, occurring during a 3-week to 6-month exposure, in materials taken individually, allow a valid estimate of whether the vessel will last 15 to 20 years, or fail much sooner.

The material properties examined after immersion are multiply related to the functions the brick and mortar must serve. The face course must possess abrasive and chemical corrosion resistance and thermal insulating properties. The backing brick is to balance the stresses of internal/external gas pressure differences, thermal expansion, solvent swelling, and membrane compression. The membrane must match the brick to the steel and provide the final solvent barrier. The carbon steel shell has the direct function of structural support. Five relevant tests are sample appearance, immersion medium appearance, weight loss, compressive strength loss and porosity.

JPL's post-immersion testing followed the most current procedure set by the ASTM (C267-71) for chemical-resistant mortar. Both vendors have the experience necessary to judge fresh-cut brick by the same method, given the absence of an ASTM evaluative procedure for chemical-resistant brick. The procedure was modified to optimize the test procedures by reducing the number and duration of tests from the suggested 1/7, 1, 2, 4, 8 and 12 week schedule to 2, 4, 6, 8 weeks and 1, 3, 6 weeks, by Pennwalt and Stebbins, respectively. The alternative of accelerated testing to immersion testing was rejected for lack of established correlations to normal behavior.

The two quantitative tests, changes in weight and compressive strength, can each demonstrate three trends. First, there can be no change in the original values. Second, there can be a decrease to a plateau at some lower value, which may or may not be acceptable. Last, there may be a continuous decline. An acceptable trend that holds for months should hold for a 15-year vessel life. The two tests are somewhat independent, and each alone is a minimal barometer of success. With the use of a coal slurry, the medium appearance can only confirm the loss of full grains of material. Sample appearance gives a sensitive check on surface grain loss and points up chemical reactivity in cases where the reaction changes the grain color.

The immersion medium for the test of both chlorinator and hydrolyzer vessel materials was a coal slurry maintained under chlorination conditions. The base is BOM-approved PSOC-276 coal, which has a balanced organic and pyritic sulfur content for a total of 5.15 percent. The coal was initially wetted with water at a 0.5/1 water/coal ratio and slurried in methylchloroform at a solvent/coal ratio of 2/1. Chlorine was bubbled through the slurry until it was saturated. The first slurry had a balanced particle size distribution between fine and coarse coal: 44 percent of 12-35 mesh, 11 percent of 65-100 mesh and 44 percent of +200 mesh. The size distribution of the replacement slurry at the midpoint of testing was changed as a result of the quick attrition of the larger size range noticed in the original slurry. The second slurry contained 51 percent of 12-35 mesh. 33 percent of 35-65 mesh and 12 percent of 100-200 mesh, along with 4 percent of 65-100 mesh and no fines. The operating temperature was 74°C, and the pressure slightly above atmospheric. Agitation power was on the order of 0.01 horsepower/sample.

The entire immersion test was broken into four periods of from 4 to 9 days, or from 4 to 12 days, if defective operation is included at face value. Cumulative times ranged from 17 to 28 days for one immersion tank and from 27 to 36 days for the second.

Solvent, water and dissolved chlorine losses were made up at the end of every period and sometimes more frequently.

The following table summarizes the findings of Pennwalt and Stebbins, who performed their own analyses. For each test, the degree of acceptability is indicated. For the quantitative tests, the nature of the trend is indicated as well. Complete information in each test category was provided to the vendors; however, their conclusions on all categories have not yet been received. Such deficiencies are marked NA (not available).

Material	Overall	Sample Appearance	Weight Change (Trend-Quality)	Compressive Strength Change (Trend-Quality)
HB mortar	B	NA	5 - I	2+4 - P
Duro brick	F	NA	1 - A	3+4 - F
Redshale brick	E	NA	NA	2 - E
K14 mortar	A	A	6 - B	6 - E
Visil brick	E	E	6 - E	6 - E
Carbon brick	E	E	6 - E	6 - E

Trend:		Quality:	
	1-no change		E-excellent
	2-plateau		A-adequate
	3-decreasing		B-borderline
	4-erratic		P-poor
	5-inconclusive		F-failure
	6-not followed		I-inconclusive

Tables 22 and 23 provide the data from which these trends and conclusions are drawn. Table 22 gives initial and final dry weights, and percent change. Table 23 contains similar information on compressive strength. Interim wet weights are not shown. Figure 19 indexes five pages of representative photographs of each material.

The following observations are provided on the immersion test data:

1) The surface behavior of each of the six materials was quite similar. In no case, out of 47 samples, did abrasion cause enough change in edge shapes, grain patterns, characteristic visual impurities (specks), or voids, that the sample was unrecognizable. Yet all samples were discolored and suffered minor changes.

2) Wet weights were the only weight change data available after the first three periods for the K14 mortar, Visil, and Carbon bricks. The graphs of wet weight change for these samples were inconclusive, as the mass of liquid and coal retained (5 to 10 percent) after air drying was comparable to the weight loss. They did, however, show reproducibility with a standard deviation of less than 2 percent between samples of similar material.

3) Dry weight changes were biased to varying extents, depending on porosity, by deep penetration of coal fines. This could reach several percent in cases where cleaning is ineffective. For this reason the weight change data were given much less credence than the compressive strength trend. A loss of even a few percent is significant. It is normal for bricks to have a large scatter in weight change after the second period.

4) An acceptable compressive strength loss is defined by its magnitude and by the absolute value of the remainder. A 10, 20, or 30 percent loss may be tolerated. Maximum stress in bad weather may cause 5000-psi stresses in multilayer brick construction, which sets a lower limit. Typical literature values for compressive strength are tabulated below:

	Compressive Strength (psi)*
Silicate mortars (K14, HB)	~4200
Duro brick	6 - 8000
Redshale brick	16,000
Visil brick	4700
Carbon brick	6900

*Reference: Personal communication with Mr. Robert Pierce of Pennwalt Corporation, Philadelphia, Pennsylvania, November, 1977.

Table 22. Dryweight and weight change as a function of immersion time

Sample No	Duration of Immersion (Periods)†	Initial Mass (g)	Final Mass (g)	Change (%)	Period Average Change (%)	Deviation of Average (%)
Tank 1						
HB Mortar						
3	1	20 751	19.170	- 7.62		
11	1	21 001	19.521	- 7 05		
12	1	21.762	20 711	- 4 83	- 6 50	1.47
6	2	21.219	20 599	- 2 92		
7	2	20 915	20 030	- 4.23		
8	2	22 543	21 991	- 2 45	- 3 20	0 92
4	3	22 118	20 161	- 8 85		
10	3	21.240	19.719	- 7 16		
13	3	20 973	18 970	- 9 55	- 8 52	1 23
5	4	20 445	18 809	- 8 00		
9	4	21 273	19 820	- 6.83		
14	4	22 390	20.370	- 9.02	- 7 95	1 10
Redshale Brick						
15	3*	41 588	NA			
16	3*	41.508	NA			
17	3*	43 434	NA			
K14 Mortar						
1	4	25 291	23 589	- 6.73		
2	4	24 199	22 474	- 7 13	- 6̈93	(0 28)
Tank 2						
K14 Mortar						
16	4	25.990	22.876	-11 98		
17	4	24 285	21 393	-11 91		
18	4	26 094	22 905	-12 22	-12 04	0 16
Visil Brick						
1	4	31 932	32 012	+ 0 25		
2	4	31 839	31 944	+ 0 33		
3	4	32.147	32.202	+ 0 17	+ 0 25	0 08
Duro Brick						
4	1	39 178	39.280	+ 0 26		
14	1	36.927	36 990	+ 0 17		
15	1	39 110	39 212	+ 0 26	+ 0 23	0 05
5	2	38 341	38 421	+ 0 21		
11	2	37 050	37.149	+ 0.27		
13	2	39 564	39 679	+ 0 29	+ 0 26	0 04

Table 22. Dryweight and weight change as a function of immersion time (continued)

Sample No.	Duration of Immersion (Periods)†	Initial Mass (g)	Final Mass (g)	Change (%)	Period Average Change (%)	Deviation of Average (%)
Duro Brick (Cont)						
6	3	37 190	37 390	+ 0 54		
7	3	37 518	37 690	+ 0 46		
8	3	37 306	37 590	+ 0.76	+ 0.59	0.16
9	4	37 836	37.784	- 0 14		
10	4	39 122	39 110	- 0 03		
12	4	37 618	37 569	- 0 13	- 0 10	0 06
Carbon Brick						
19	4	26 018	26.247	+ 0 88		
20	4	26 230	26.466	+ 0 90		
21	4	25.941	26 291	+ 1 35	+ 1 04	0 27
Redshale Brick						
28	1*	43.043	NA			
29	1*	43 601	NA			
30	1*	43.644	NA			
25	2*	41.756	NA			
26	2*	43.042	NA			
27	2*	42.284	NA			
22	3*	43 139	NA			
23	3*	44 444	NA			
24	3*	43 491	NA			

† Average period is 5 days for tank one and 7 days for tank two
* Slightly shortened
NA Not yet available from vendor.

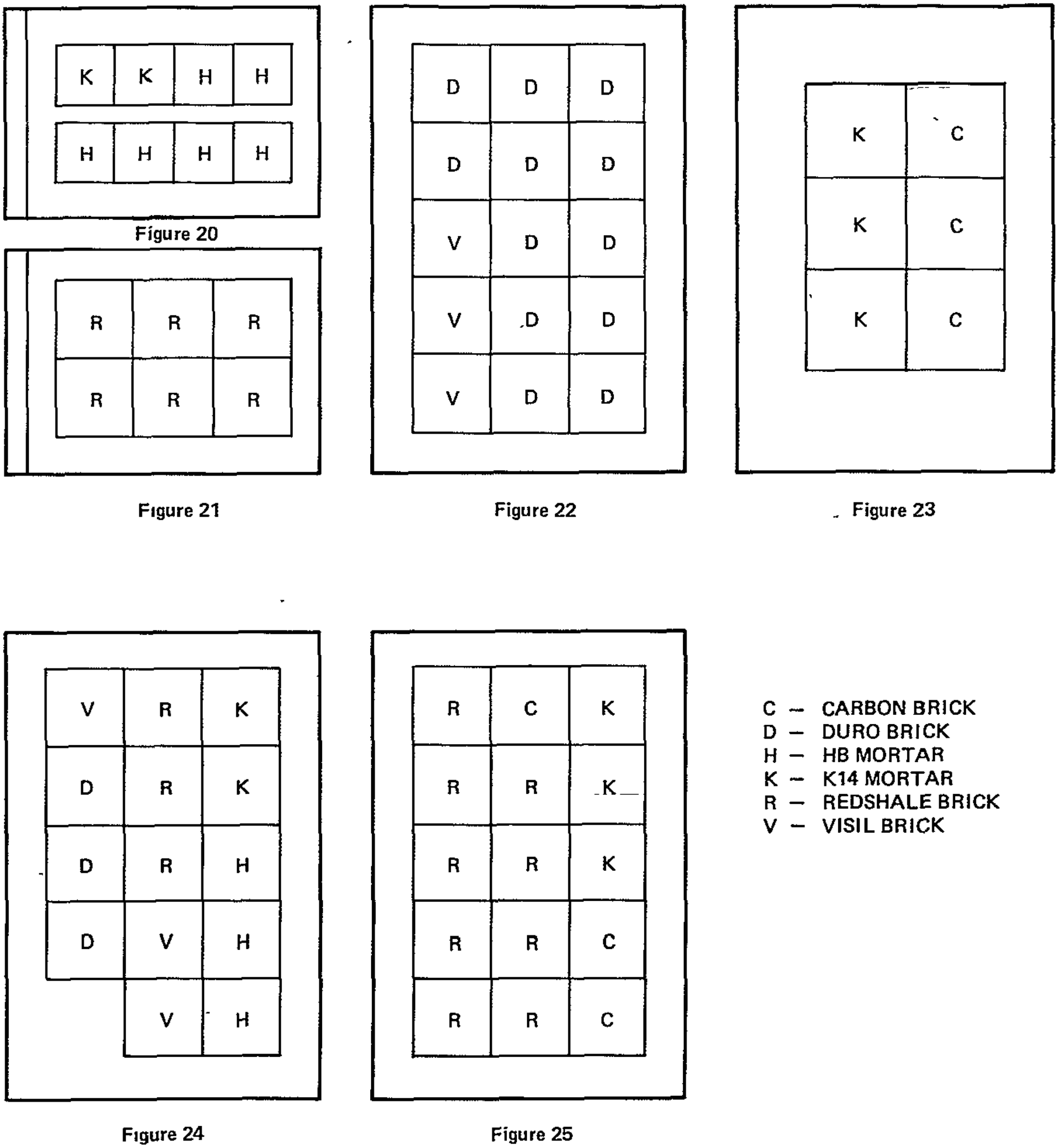
Table 23. Compressive strength change as a function of immersion time

Sample No.	Duration of Immersion (Periods)†	Control Compressive Strength (psi)	Footnote No.	Post-Immersion Compressive Strength (psi)	Change (%)	Period Average Change (%)	Deviation of Average (%)
Tank 1							
HB Mortar							
3	1	4998	1	4185	-16 3		
11	1		1	4495	-10 1		
12	1		1	3835	-23 3	-16 6	6.6
6	2		1	3960.	-20.8		
7	2		1	2980	-40.4		
8	2		1	3490	-30 2	-30 5	9 8
4	3		1	4205	-15 9		
10	3		1	4750	- 5 0		
13	3		1	3385.	-32 3	-17 7	13 7
5	4		1	3475.	-30.5		
9	4		1	3950	-21.0		
14	4		1	4140	-17 2	-22 9	6 9
Redshale Brick							
15	3*	22247.	2	20250	- 9 0		
16	3*		2	22900	+ 2 9		
17	3*		2	23600	+ 6 1	0.0	8 0
K14 Mortar							
1	4	4950	3,4,5	8550.	+72 7		
2	4		3,4,5	8550.	+72 7	+72.7	(0)
Tank 2							
K14 Mortar							
16	4	3372.	1	3550	+ 5 3		
17	4		1	3550	+ 5.3		
18	4		1	3905.	+15.8	+ 8 8	6.1
Visil Brick							
1	4	7100	3,5	7455	+ 5.0		
2	4		3,5	7100.	0	+ 2.5	(3 5)
3	4			Hold			
Duro Brick							
4	1	11830.	2	9535	-19 4		
14	1		2	9665.	-18 3		
15	1		2	9120	-22 9	-20 2	2 4
5	2		2	5680.	-52.0		
11	2		2	6930	-41.4		
13	2		2	9750	-17 6	-37 0	17 6

Table 23. Compressive strength change as a function of immersion time (continued)

Sample No.	Duration of Immersion (Periods)†	Control Compressive Strength (psi)	Footnote No.	Post-Immersion Compressive Strength (psi)	Change (%)	Period Average Change (%)	Deviation of Average (%)
Duro Brick (Cont)							
6	3		2	4130.	-65.1		
7	3		2	8315.	-29.7		
8	3		2	8825.	-25 4	-40 1	21 8
9	4		2	9240	-21 9		
10	4		2	13605	+15 0		
12	4		2	15700	+32 7	+ 8.6	27.9
Carbon Brick							
19	4	10295.	3,5	11715	+13 8		
20	4		3,5	11005	+ 6 9	+10.4	(4.9)
21	4			Hold			
Redshale Brick							
28	1*	22247.	2	17250	-22 5		
29	1*		2	13950	-37 3		
30	1*		2	12100	-45.6	-35.1	11 7
25	2*		2	23600	+ 6 1		
26	2*		2	23100	+ 3 8		
27	2*		2	20900	- 6 1	+ 1.3	6 5
22	3*		2	19400	-12.8		
23	3*		2	24097	+ 8 3		
24	3*		2	16173	-27 3	-10.6	17 9

† Average period is 5 days for tank one and 7 days for tank two
* Slightly shortened
1 Average of 2 control samples from the same batch
2 Average of 3 control samples from the same batch
3 Quality control samples off-the-shelf, the total number of samples unknown
4 Could be 4750 psi
5 Standard deviations based on two values in parentheses



C – CARBON BRICK
D – DURO BRICK
H – HB MORTAR
K – K14 MORTAR
R – REDSHALE BRICK
V – VISIL BRICK

THE FIRST FOUR PHOTOGRAPHS DISPLAY SEVERAL TYPICAL SURFACES OF EACH OF THE SIX MATERIALS BEFORE EVALUATION. THE FINAL TWO PHOTOGRAPHS COVER THE SIX MATERIALS AFTER FOUR PERIODS OF TESTING. NO ATTEMPT IS MADE TO GIVE BEFORE-AND-AFTER COMPARISONS OF THE SAME SAMPLES OR THE SAME FACES OF EACH SAMPLE.

Figure 19. Photograph index

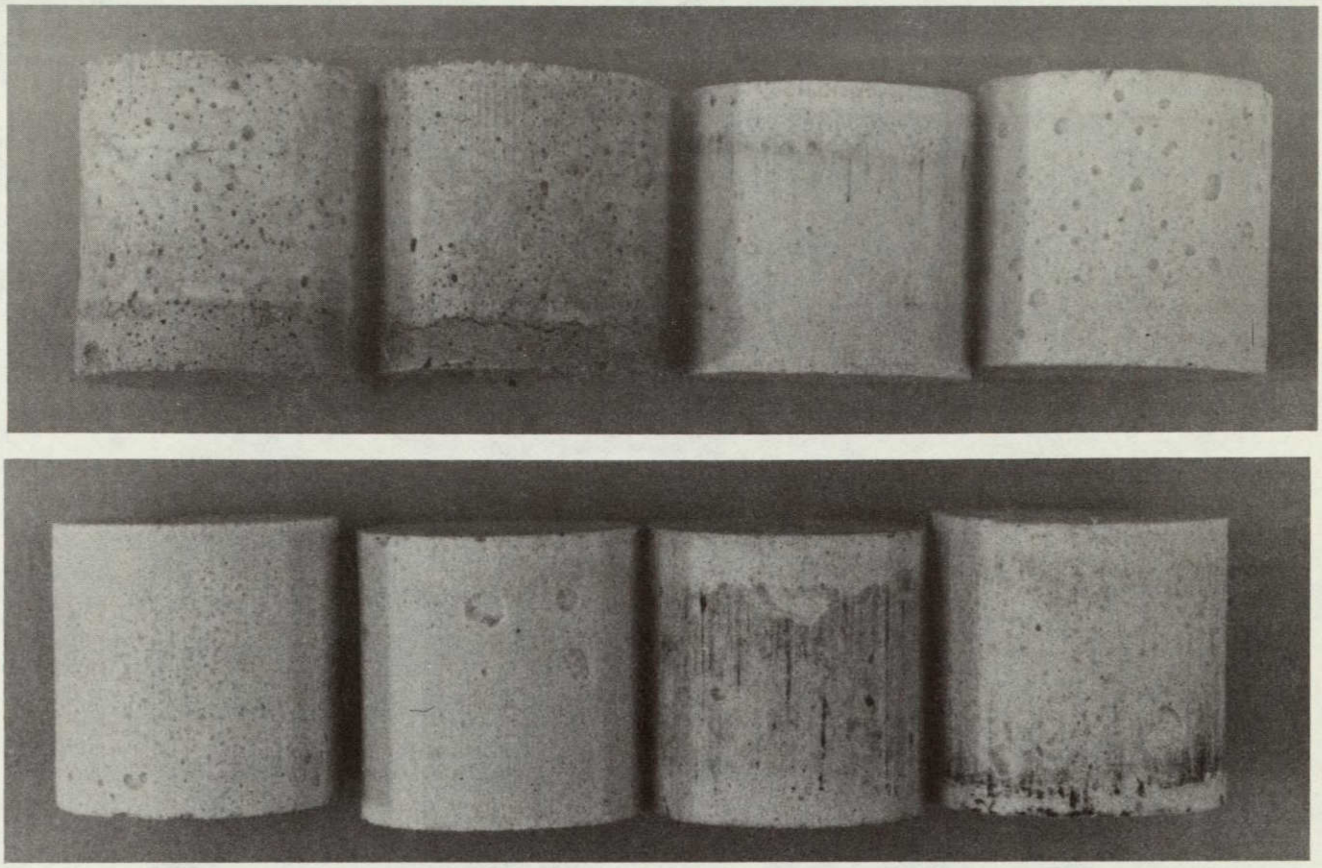


Figure 20. Pre-immersion K14 and HB mortar

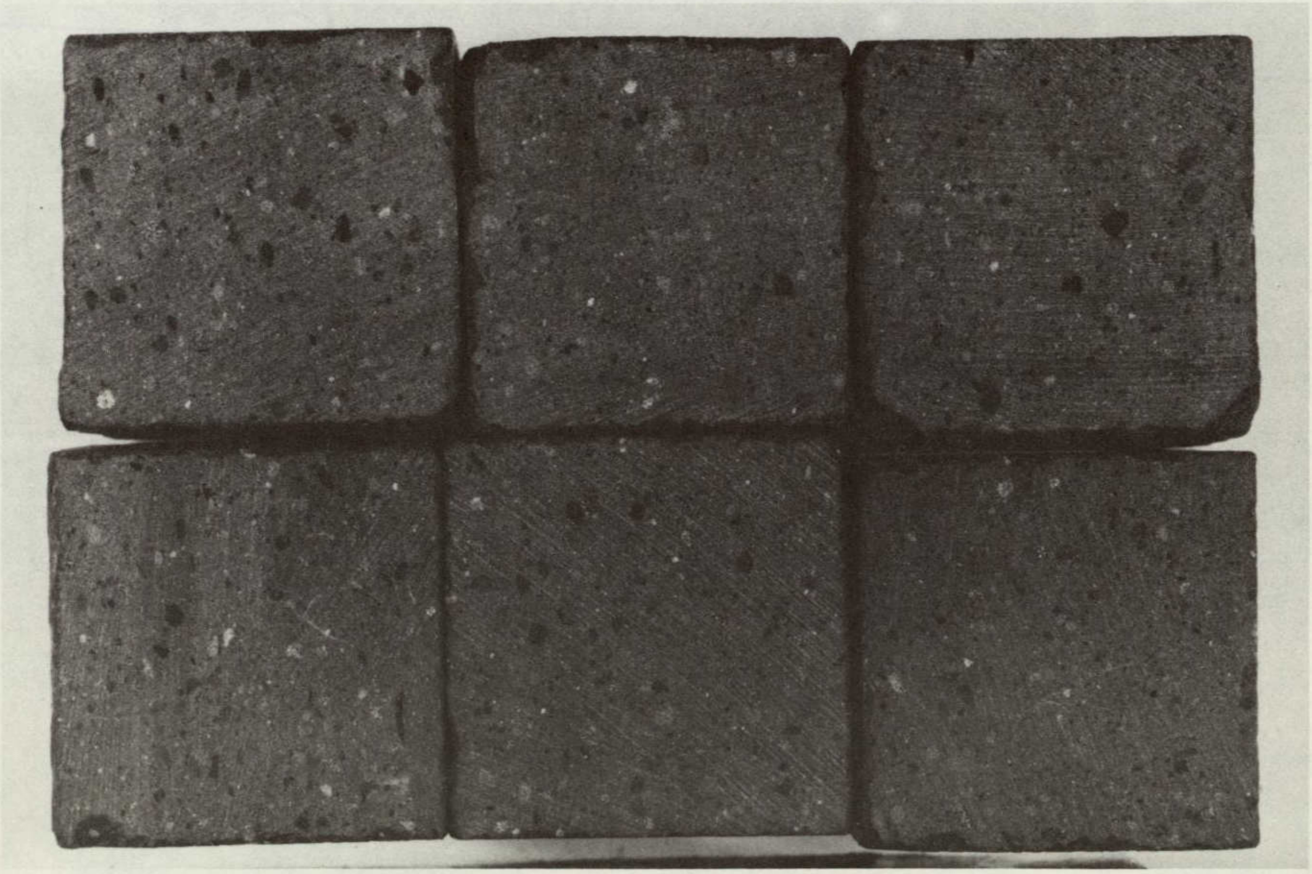


Figure 21. Pre-immersion redshale brick

C-2

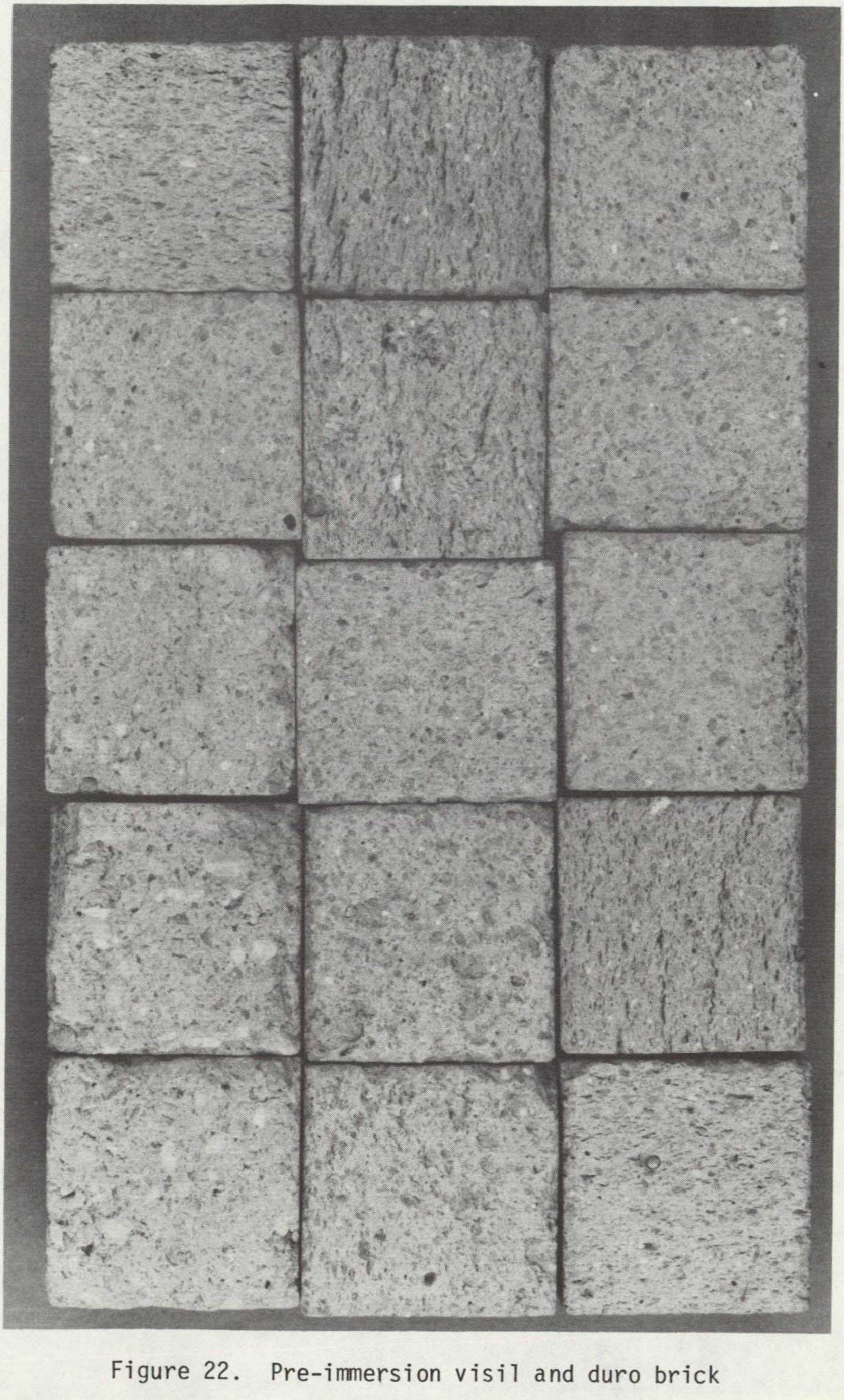


Figure 22. Pre-immersion visil and duro brick

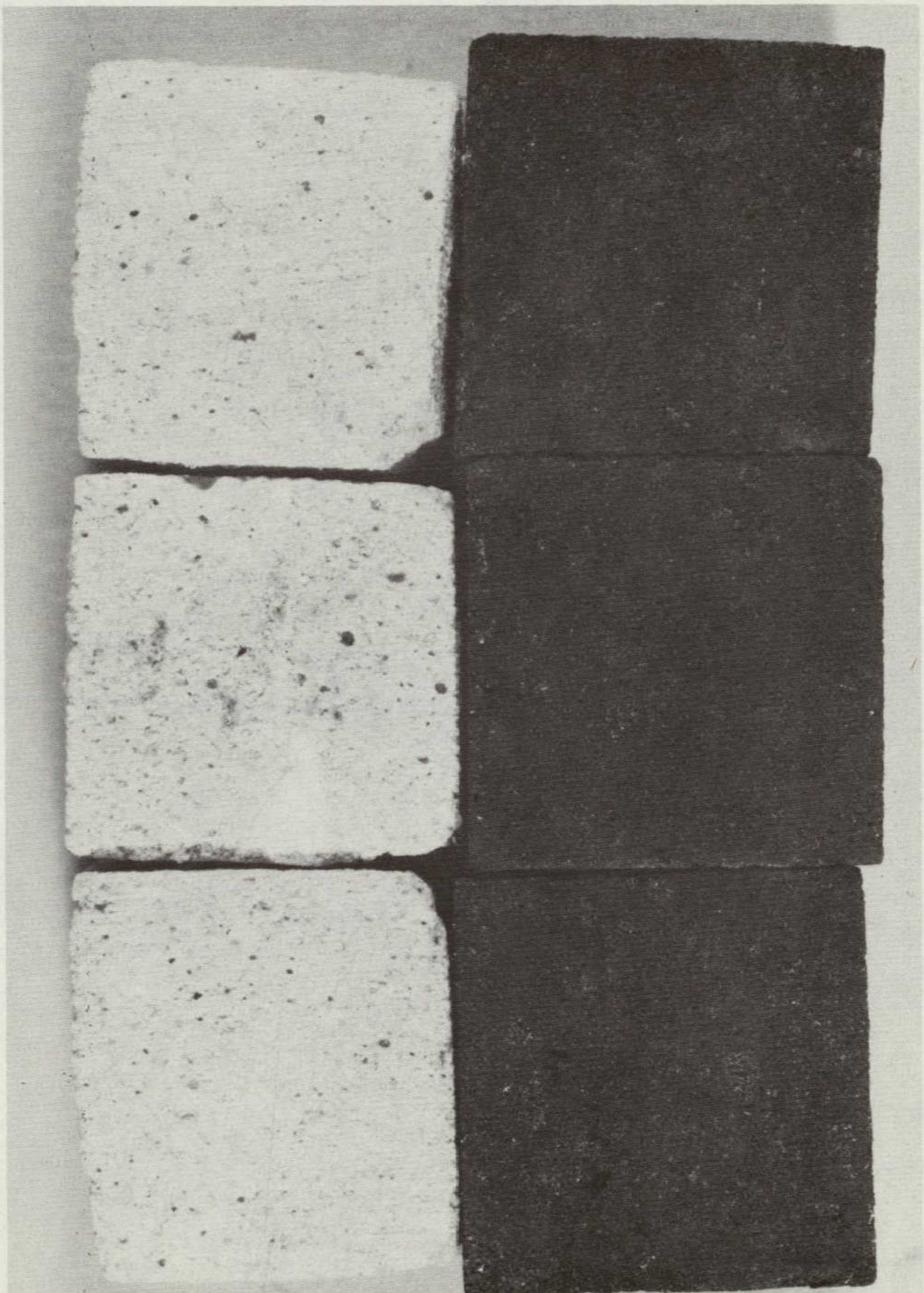


Figure 23. Pre-immersion K14 mortar and carbon brick

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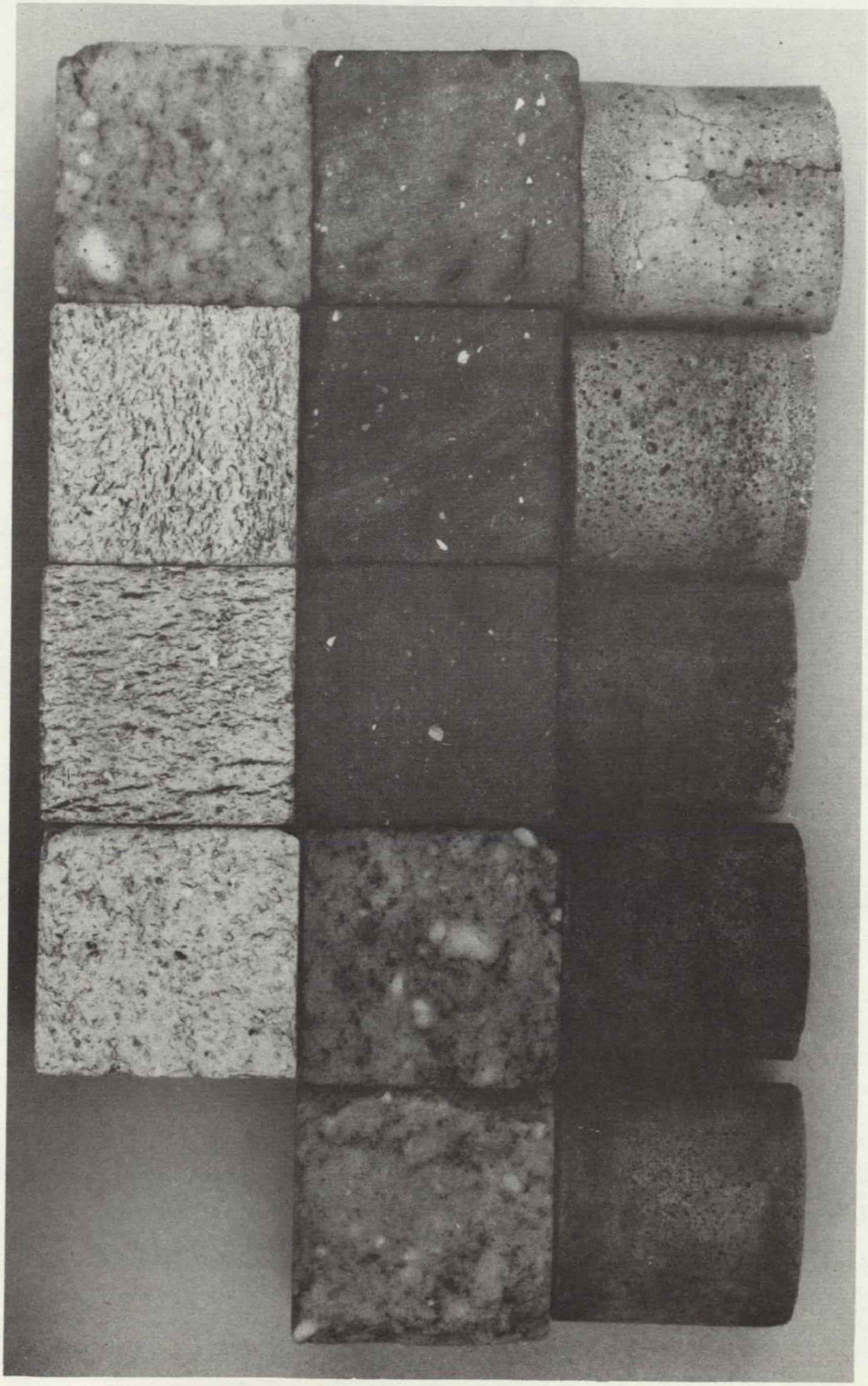

Figure 24. Post-immersion K14 and HB mortars, redshale, visil and duro brick

Figure 25. Post-immersion K14 mortar, carbon and redshale brick

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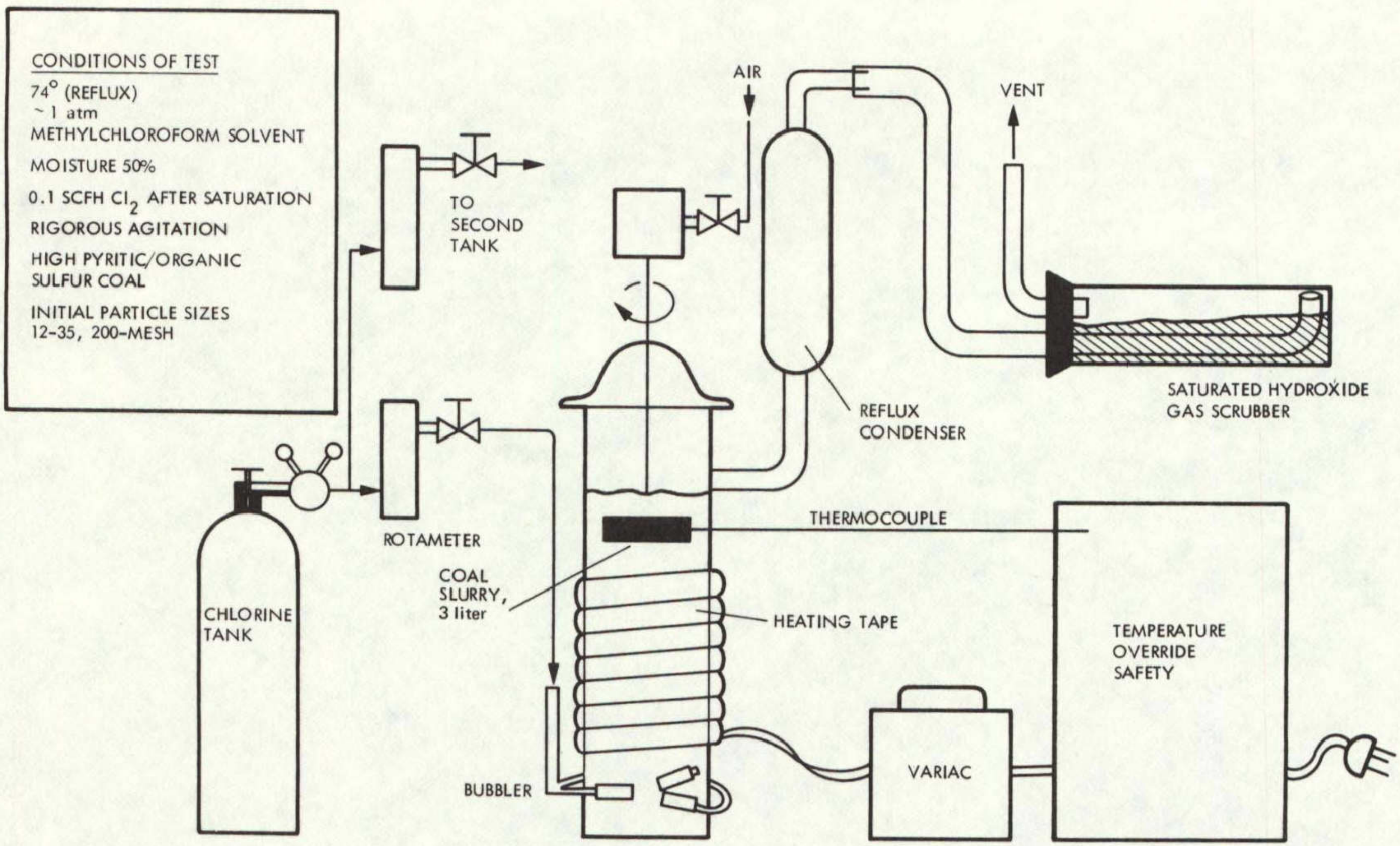
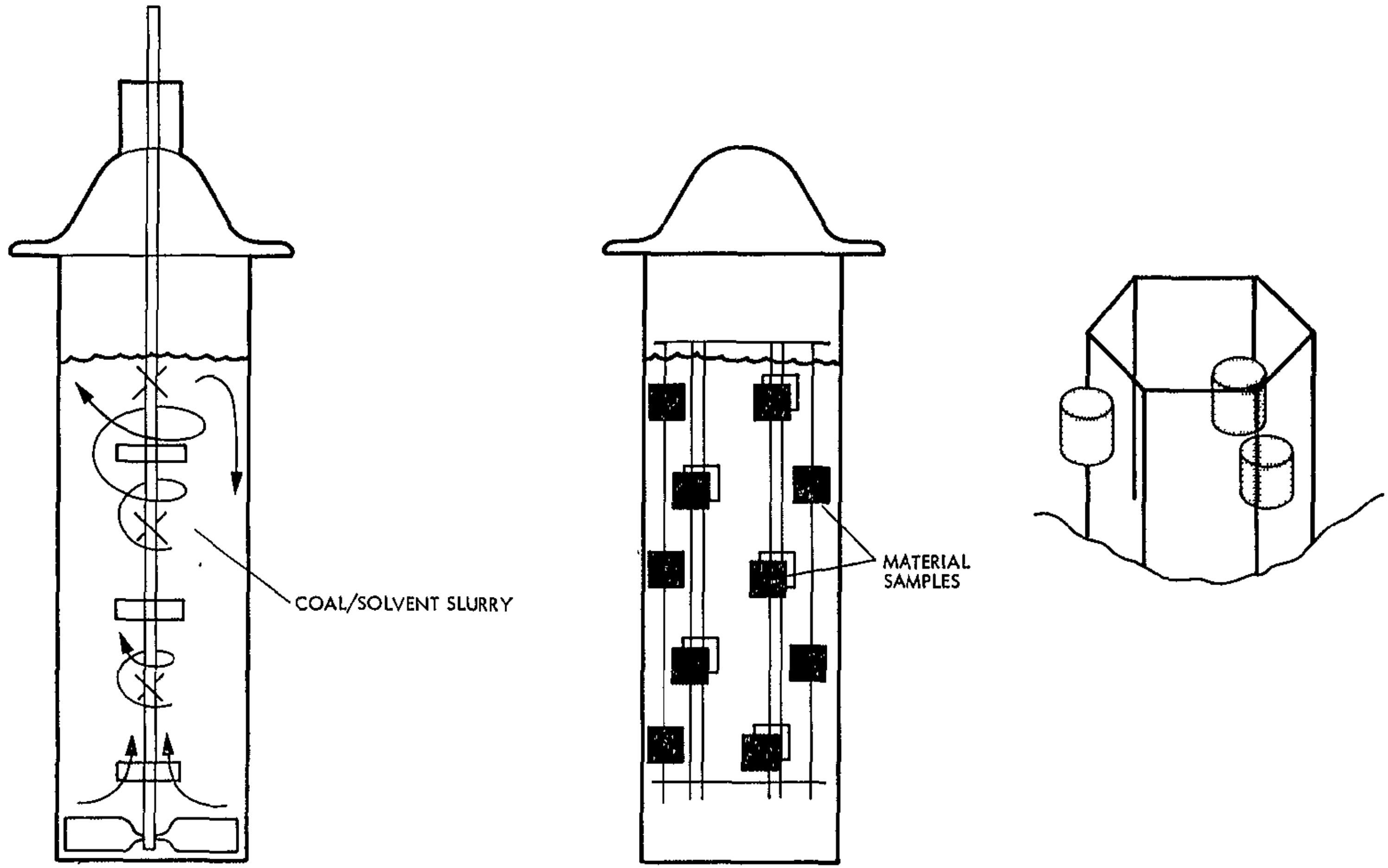


Figure 26. Apparatus for process reactor materials testing



(a) STIRRING AND FLOW PATTERN

(b) CAGE AND SAMPLE PLACEMENT

(c) CAGE DETAIL

Figure 27. Sample layout and flow details for reactor lining materials testing

Laboratory prepared blanks may test 50 percent higher than the literature value, which is the value expected in field use. A large loss after the first period is not erratic, but any other non-monotonic trend is erratic.

5) In determining the durations of submersion, periods of defective operation were weighted at between 0 and 50 percent of their face values by the different vendors.

6) Stebbins does not use trend evaluation of its samples. This makes it more difficult to gauge the borderline materials, though clear successes and failures are obvious from the final data point.

Two glass tanks were used to contain the 47 samples. Figure 26 displays the entire system. The chlorine delivery system feeds three bubblers at the bottom of the slurry with from 0.1 to 3 SCFH of chlorine gas. The reflux condenser provides solvent recovery as well as temperature control. It feeds into a sodium hydroxide chlorine scrubber with zero liquid head. Heat input was first provided by electrical filament tape as shown, and later by a water bath surrounding the tank. A thermocouple override and fume hood provided safety elements.

Figure 27 shows the glass cage and stirring structure within the first tank. This design allows no settling of the coarser particles and exposes almost the entire surface area of the samples to impingement attack.

Several major problems reduced the duration of proper operation by nearly one fourth. The stirring function was plagued by outages and a decrease in effectiveness when the slurry became finer. Maintaining the reflux temperature throughout the vessel without flashing near the heat source was difficult. Coal fines were found to coat the samples a few millimeters deep. Inadequate design tolerances resulted in frequent glass breakage. All these difficulties caused large errors in estimating the true duration of immersion. Periods were from 4 to 12 days long instead of 10.

As a result of JPL's Phase I acceptance tests, both vendors have modified their vessel designs. Pennwalt now recommends a redshale brick with modified phenolic mortar to replace its Duro-faced potassium silicate mortar design. If the silicate HB mortar were to be used, it might require repointing after 5-10 years service, which would require an adjustment in the joint widths and hence a careful redesign. Stebbins' general conclusion: "All materials appear substantially unaffected by chemical exposure. K-14 may be subject to abrasive attack by the slurry. Joints might require repointing after 3-5 years of operation." Stebbins suggests that a higher grade silicate mortar, AR20, used throughout the vessel would eliminate this potential problem. It is also possible that the alumina in the Visil brick will degrade after a long lead time. This would be detectable only with a full-length (3 to 6 months) immersion test. Stebbins will not guarantee the design as a whole until the steel-protective membrane is proven. Pennwalt maintains that rubber under compression is difficult to test and that this is not necessary.

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