

DEASHING OF COAL LIQUEFACTION PRODUCTS VIA PARTIAL  
DEASPHALTING. I - HYDROGEN-DONOR EXTRACTION EFFLUENTS

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INTRODUCTION

One of the more important problems to be resolved in the commercial development of coal liquefaction processes is that of deashing and solids separation.

An extensive laboratory program in a joint program with the Standard Oil Company of Ohio was carried out in the Research Division of Consolidation Coal Company in the period 1959 through 1963 to study the basic essentials of the hydrogen-donor extraction process. The major method of solids separation studied during this period was pressure filtration. The small scale of the experimental work, i.e., maximum feed rate of about 30 lb/hr, precluded the use of continuous filtration equipment. Consequently, all the work was conducted in batch cyclic equipment.

The experimental work was continued and expanded with the financial support of the Office of Coal Research.<sup>(1)</sup> One of the items covered in the OCR funded laboratory program<sup>(1)</sup> was further investigation of pressure precoat filtration. Filtration rates were "optimized" and relative freedom from filter blinding was secured by operating in the 500-600°F range. It was noted that some precipitation of dissolved extract occurred in this temperature range.

The OCR funded program culminated in the design and operation of a 20 TPD plant in Cresap, West Virginia.<sup>(2,3)</sup> The design of the pilot plant, based on prior laboratory data, included the use of continuous pressure precoat filters for solids separation. Mechanical difficulties, which were unresolved, precluded successful operation of the filter system. Filtration rates observed, however, were in the range predicted from prior laboratory data.

The filters were accordingly replaced by a hydroclone system which operated in the range of 500-600°F. Successful operation of the hydroclone system was achieved and the results have been reported.<sup>(3)</sup> Success was, no doubt, due in part to partial extract precipitation and resulting solids agglomeration on cooling the extraction effluent.

Batch laboratory experiments were conducted prior to operation of the Cresap pilot plant<sup>(1,4)</sup> on the deashing of extraction effluents by means of the addition of deasphalting solvents.

Laboratory experimentation has continued in both batch and continuous units on improvement of the deashing process. Some of this work has been previously reported<sup>(\*)</sup> where the preliminary results of deashing by continuous settling were presented.

The present paper is concerned with a more detailed account of the above work on deashing.

EXPERIMENTAL

Batch experiments on deashing with deasphalting were conducted in a one-gallon stirred autoclave, 5" I.D. x 12" high. The autoclave was equipped with an adjustable sample probe and a N<sub>2</sub> purged differential cell with the probe functioning as the high-side of the D/P cell. In combination, the probe and the D/P cell could be used to

find the liquid-gas interface within the autoclave. Once the interface was found, the probe was moved a prescribed depth into the liquid bed at which point small samples were taken at various time intervals to determine the settling rate of the interface between the settling solids and the clear liquid phase. As a result of the liquid samples withdrawn, there was a net decrease in the level of the gas-liquid interface. A height adjustment was used after each sample to maintain a constant immersion.

The experimental procedure involved stirring the autoclave contents at 890 r.p.m., heating to extraction temperature, holding for prescribed time at temperature, cooling to deashing temperature followed by addition of deashing solvent, if any. At this point the stirrer was stopped and the autoclave contents were allowed to settle.

Samples were taken immediately after stirring was stopped according to a prescribed time and depth schedule. The purpose, of course, was to determine the settling rate. A correction had to be applied in determining the settling rate to account for the perturbation induced by the unavoidable relatively large sample taken for analysis. The correction procedure was checked by applying it to a model system of coal settling in decane where the settling rate could be determined by visual observation.

The continuous unit used for continuous deashing is schematically illustrated in Figure 1. The extraction system itself is adapted to operation at slurry feed rates up to 30 lb/hr. The five-stage extractor contains a total inventory of 1.7 gallons. The construction and operation of the continuous extraction system has been previously described in detail.<sup>(2)</sup> The operation of the continuous settler without deasphalting solvent addition was also previously described.<sup>(5)</sup>

The flow sheet of Figure 1 also shows the system for continuous metering of additives to promote deashing. Two types of additives were used. The first consisted of n-decane which acted as a deasphalting solvent. The second consisted of 16% aqueous  $H_2SO_4$ . The purpose of the sulfuric acid was to remove soluble alkaline ash from the extract by conversion to sulfates. The stoichiometric feed rate was calculated based on conversion of the combined Na, K, Mg and Ca content of the feed coal to the corresponding sulfates. The extraction effluent is continuously mixed with the deasphalting solvent in the additive contactor. This is a one-gallon stirred, electrically heated vessel usually operated at the same temperature as the continuous settler. The contactor vessel was employed to precool the slurry feed to settler temperature in most cases, even when no additive was used.

A number of different size vessels were used for continuous settling in order to determine the effects of varying throughput rates in lb/hr-ft<sup>2</sup>, and of different residence times in the compaction zone below the feed inlet.

The dimensions of the settlers used are outlined in the table below.

Settler No.	ID in	Area, ft <sup>2</sup>		Vol. Below Feed Point ft <sup>3</sup>
		Above Feed Point	Below Feed Point	
1	7.98	0.328	0.347	0.405
2	4.60	0.096	0.116	0.135
3	4.60	0.096	0.116	0.251
4	3.15	0.045	0.054	0.117
5	3.15	0.045	0.054	0.179

The bottom two inches of the settlers were agitated by a rake rotating at 1-2 r.p.m. The bottom of the rake contained two perpendicular rows of 1/8-inch spikes.

A run was conducted by setting the coal slurry feed composition and rate, extraction temperature, additive feed rate and settler temperature at the prescribed values. The rate of withdrawal of underflow from the settler was also prescribed

in advance. The underflow rate was controlled manually by intermittent withdrawal through a 1/2-inch gate valve at 10 minute intervals into the underflow product receiver which was positioned on a weigh scale.

Steady operating conditions were achieved usually after three inventory changes through the system. A material balance period ensued in which samples of all liquid and gas streams were taken. Duplicate gas samples were normally taken at the beginning and end of the material balance period. The methods of analysis and calculational procedures were substantially the same as those previously described.<sup>(2)</sup>

A number of different coals were investigated including Pittsburgh Seam and Illinois No. 6 coals from the eastern-half of the United States and bituminous and subbituminous coals from the western part of the country. The detailed investigation was, however, confined to two coals, i.e., a Pittsburgh No. 8, a high-volatile bituminous C coal from the Ireland Mine of Northern West Virginia and a Utah subbituminous coal from the Browning Mine in Emery County. Only the work with the latter two coals will be described. Inspections of the two coals are given in Table I. Two batches of the Browning Mine coal were used. As noted, the second batch was significantly lower in sulfur content.

The Ireland coal is a strongly caking, highly fluid and highly caking bituminous coal. The Browning Mine coal is, however, substantially noncaking and exhibits little or no fluidity in the standard Gieseler plastometer test. Its classification by the ASTM system is borderline between high-volatile C bituminous and subbituminous B. Because of its noncaking properties, it is more logical to regard it as a subbituminous coal in rank.

The work with the Ireland coal was conducted with solvents derived from the Cresap pilot plant.<sup>(3)</sup> These were "natural oils" derived from hydrocracking of the extract in the ebullated-bed catalytic hydrocracker. The oils were of two types, namely, fresh make-up hydro oils derived from the hydrocracker and extraction recycle oils. The latter oils were derived from the make-up hydro oil by recycle through the extraction unit and naturally were more depleted in hydrogen donors than the former. They also contained some undisplaced start-up solvent which consisted mostly of mixed methyl-naphthalenes. Analysis of typical oils of this type are given in Table II. The identification of specific compounds by gas chromatographic analysis is somewhat tentative due to the complex nature of the mixture.

The work with the Browning Mine coal on the other hand was conducted mostly with synthetic solvents consisting of mixtures of methyl-naphthalene and tetralin.

The definitions given below are applicable to the discussion presented in the remainder of this paper.

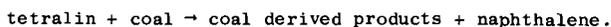
Extract in what follows is defined as that portion of the extraction effluent which boils above 240°C at 1 mm Hg and which is soluble in boiling cresol.

MAF extraction residue is the moisture and ash-free portion of the extraction effluent which is insoluble in boiling cresol.

Coal conversion is defined as

$$100 - \frac{100 \text{ lb MAF extraction residue}}{\text{lb MAF coal feed}}$$

Hydrogen transfer in the runs with synthetic solvents is determined as previously<sup>(1)</sup> from the amount of naphthalene produced via the reaction,



RESULTS AND EFFECTS OF VARIABLESA. Ireland Mine Coal1. Batch Results

A brief summary of the batch results will be given for comparison with continuous results. The average results of a series of runs with natural solvents are given in Table III. Two types of solvent mixtures were used, i.e., 25% Cresap hydro oil-75% recycle oil and 100% hydro oil. No deasphalting solvent was used in these runs. It is clear that a relatively high settling rate can be achieved without addition of a deasphalting solvent. The settling rate is drastically reduced, however, when the coal conversion is increased to 86% by use of 100% hydro oil.

The addition of deasphalting solvents was also studied previously<sup>(1,4)</sup> in batch experiments. Their effect on settling rates had not been determined. These experiments focused on a determination of the ash content of the clarified extract. All deasphalting solvents used were saturated hydrocarbons and all were effective in reducing ash content of the clarified extract. On a weight basis, all hydrocarbons tried, i.e., n-paraffins, n-butane through n-decane, cyclohexane and decalin were approximately equally effective in deasphalting and reducing extract ash. Their relative effect on rate of settling still remains to be determined, however, and such measurements are now in progress.

2. Continuous Results

The deashing of the extraction effluents from donor extraction using natural "Cresap" derived solvents via gravity settling was investigated in some detail. The products from four extraction conditions were used in this study. The conditions used and a summary of results are given in Table IV.

A series of settling runs were conducted using the effluent from Condition A (1.5 S/C ratio, 75-80% coal conversion) using settling temperatures of 550-600-650°F. No usable data were obtained at 550°F since massive deposits were formed in the settler walls. The settler was clean, however, at the two higher temperatures. The overflow rate in lb/hr-ft<sup>2</sup> was varied over a wide range by use of settlers with various cross-section areas as explained earlier. The underflow rate was not treated as a separate variable. In most runs, it was arbitrarily set at 20% of the feed rate to extraction. This usually gave an underflow stream containing 45-55 wt. % cresol-insolubles.

The effect of the throughput rate on percent ash breakthrough from these experiments is illustrated in Figure 2.

The effect of varying extraction conditions on settling behavior was also studied and results are summarized in Table V. A few runs are also shown in Table V, illustrating the removal of fine ash by sulfuric acid addition.

It is immediately noted that the settler throughput rate can be greatly increased, without sacrifice of deashing efficiency, by increase of the solvent-to-coal ratio from 1.5 to 2.

As was noted previously in batch results, increase of coal conversion from 77 to 86% greatly decreased deashing efficiency at 1.5/1.0 solvent coal ratio. It appeared to have little affect, however, when the solvent-to-coal ratio was increased to 2.

The effect of sulfuric acid addition is largely to reduce the fine ash of the extract. About one-third of the fine ash is removed, largely by conversion of soluble Ca to insoluble CaSO<sub>4</sub>. About 50% of the extract calcium is removed by sulfuric acid addition.

The extract recovery in the overflow is given in the last row of Table V. It is seen that the extract recovery in the overflow is in the range of 84-88%. Higher recoveries, of course, can be achieved by washing the underflow with feed solvent. Recoveries are also increased, without washing, by increasing coal conversion, increasing the solvent-to-coal ratio and by decreasing the amount of precipitated extract. The above statements follow from the material balance relationships in the system.

A small fraction of the extract is precipitated on cooling the extraction effluent to settler temperatures.

The amount of precipitated extract may be readily calculated from the observed extract-to-solvent ratios of the overflow and underflow streams, if it is assumed that all of the precipitated extract is in the underflow stream. The relationship is

$$X = (E_o/S_o) / (E_u/S_u) \quad (1)$$

where:

- X = the fraction of underflow extract which is in the soluble form.  
 $E_{o(u)}$  = lb of extract in overflow (underflow).  
 $S_{o(u)}$  = lb of solvent in overflow (underflow).

The fraction of total extract precipitated is then readily calculated by material balance.

Equation (1) was applied to the series of runs conducted at 600°F settler temperature at the two solvent-to-coal ratios, respectively, with results as shown below:

<u>Solvent-to-Coal Ratio</u>	<u>No. of Runs</u>	<u>Average Quantity Extract Precipitated, Wt. % MAF Coal</u>	<u>Standard Deviation</u>
1.5	14	2.3	1.5
2.0	3	5.5	1.2

The precipitated extract, was shown by solvent fractionation analysis, to consist, as expected, of the highest molecular weight fraction of the extract, i.e., the benzene insolubles.

## B. Browning Mine Coal

### 1. Batch Results

The batch experiments are summarized in Table VI. It is noted that in contrast to Ireland coal that the settling rate in the absence of additives was very slow. The addition of decane, however, was effective in providing acceptable settling rates.

Synthetic solvents consisting of methylnaphthalene alone or in admixture with tetralin were used. The viscosity of these solvents is lower than that of the higher boiling natural solvents such that the poor settling behavior with the Browning Mine coal cannot be ascribed to use of the synthetic solvent. An experiment in which the Cresap natural extraction solvent was used confirmed again the slow settling rate for this coal in the absence of additives.

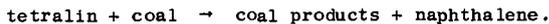
### 2. Continuous Results

The general extraction behavior of the Browning Mine coal in the continuous unit was assayed at the same time that the behavior of deashing via continuous settling

was explored. Synthetic solvents, i.e., methylnaphthalene-tetralin mixtures, were used in this work with the tetralin content of the feed solvent varied between 0 and 50%. The temperature was varied between 750-780°F and the solvent-to-coal ratio between 1.5 to 4.0 in this work.

The relationship between coal conversion and hydrogen transfer is shown in Figure 3. Like the results reported previously with Ireland Mine coal,<sup>(5)</sup> the experimental values fall on a relatively smooth curve independent of the experimental conditions.

Hydrogen transfer in these experiments is defined as the amount of hydrogen transferred to the coal via the general process,



A small amount of naphthalene is also produced via hydrogen-donor cracking of the solvent, i.e., by the reaction,



The naphthalene produced by the second reaction accordingly is excluded from that produced by the first reaction in calculating the amount of hydrogen transfer to the coal.

A kinetic relationship between the rate of hydrogen transfer and the extraction conditions as well as the tetralin content of the solvent was derived and previously presented.<sup>(5,6)</sup> The relationship was based on the extensive data with Ireland coal. It is interesting to note, that the rate of hydrogen transfer calculated using kinetic constants derived for Ireland coal, closely predicted the hydrogen transfer rates for Emery coal. This is illustrated by Figure 4. It should be emphasized, however, that the coal conversion versus hydrogen transfer relationship is very different for the two coals, i.e., a much higher coal conversion is achieved with the Ireland coal with the same amount of hydrogen transfer.

The exploratory survey of the variables in the continuous unit confirmed the need for addition of decane to achieve a high throughput rate in the continuous settler.

A survey of the effect of temperature and decane addition rates on deashing efficiency was made at 500, 600, and 650°F. The results of the 500 and 600°F runs are shown in Figure 5. No usable results were obtained at 650°F due to coke formation in the conditioner vessel. Thus, like the previous case of Ireland Mine coal without additives, the optimum settling temperature is near 600°F. The operability relationships versus temperature appear to be quite different for the two coals, however. The reason for the difference is not apparent.

Further details on some of the runs conducted with varying decane rates are given in Table VII.

The amount of extract precipitated was calculated by the procedure described earlier for the Ireland Mine coal case for continuous runs which were conducted over long operating periods, i.e., greater than 50 hours, to demonstrate operability. The results are indicated below:

Run No.	Conversion, Wt. % MAF Coal	Decane-to-Solvent Ratio	Wt. % Extract Precipitated
41	36	0.065	0
44	53.7	0.112	2.7
51	63.8	0.290	7.7

The runs in Table VIII are not included because of their short duration and accordingly less accurate material balances.

The extract recovery in Run 51, which was conducted under nominally the same conditions as Run 45-5 gave an extract recovery of 79%.

Experiments were conducted to recover "liquid" extract in the underflow by mixing the underflow with a portion of the extraction solvent and all of the feed decane, and passing the mixture through the settler again. Details have been previously presented,<sup>(7)</sup> and it was shown that 95% of the "liquid", i.e., unprecipitated extract, was recovered by this technique which simulates a two-stage continuous settler with back washing of the underflow from the first stage.

#### DISCUSSION OF RESULTS

##### Ireland Mine Coal

Batch settling may be regarded as proceeding through a hindered settling mechanism, i.e., where the settling rate is equivalent to the fluidization velocity of the given particle size distribution at a given level of compaction. For this reason, the initial settling rate is the highest and the rate decreases as the bed compacts.

The area required in a continuous settler can be approximated from batch results by the familiar "idealized" design equation<sup>(8)</sup> which is simply derived from material balance considerations:

$$A = \frac{Q (F - D)}{\rho R} \quad (2)$$

where:

- A = area required to produce clear overflow with underflow composition D and feed composition F.
- D =  $\frac{\text{weight of liquid}}{\text{weight of solids}}$  in underflow.
- F =  $\frac{\text{weight of liquid}}{\text{weight of solids}}$  in feed.
- $\rho$  = density of the liquid.
- Q = solids feed rate.
- R = rate of settling of slurry.

There is some uncertainty about the determination of R, but as a first approximation it should equal the initial settling rate of the feed slurry as determined from batch experiments.

Equation (2) should be valid as long as sufficient residence time is provided below the feed point to allow compaction of the underflow to the "material balance" concentration by hindered settling. In all of our experiments, with one exception, sufficient residence time was supplied to achieve the desired level of compaction. From this result, it was determined that the residence time required for compaction to 50% solids is less than 1.7 hours.

Equation (2) implies a sharp breakthrough of solids as upflow velocity in the settler is increased. Experimentally, as the results given in Figure 2 show, this is far from the case, i.e., there is a gradual increase in solids breakthrough as throughput is increased.

The experimental results at 600°F shown in Figure 2 were correlated by the empirical equation of the type,

$$Y = y_0 e^{KV/V_0} \quad (3)$$

where:  $Y$  = the percent of the ash reporting to the overflow.  
 $V$  = the actual upflow velocity of the overflow stream in in/min.  
 $V_0$  = initial settling velocity of feed slurry, in/min.  
 $y_0$  and  $K$  are arbitrary constants.

For the particular slurry used,  $S/C = 1.5$ , condition A, the empirical equation, determined as best fit to the data by the method of least squares takes the form,

$$Y = 1.106 e^{0.940 V} = 1.106 e^{1.88 V/V_0} \quad (4)$$

Thus, based on equation (4), if one operates the settler at an upflow velocity equal to the initial settling velocity as determined from batch experiments, one would obtain 93% of ash removal to the underflow. Accordingly, if one desires more efficient ash removal, the velocity must be reduced significantly below the initial settling velocity.

The settling behavior also has to be rationalized with respect to the size consist of the solids in the extraction effluent. These were determined by the Coulter counter method but only after dispersion of the residue particles in tetrahydrofuran. The extract is completely soluble in tetrahydrofuran and thus any particles agglomerated by precipitated extract are redispersed.

The results for two series of runs, i.e., at 76% and 85% conversion levels, respectively, are given in Figure 6. The median particle size is quite small, i.e., 28 and 16 microns, respectively. The higher conversion runs showed a small median particle size which is consistent with the lower settling rate for these extraction effluents.

Figure 7 illustrates the removal efficiency of residue particles as a function of size with the relative velocity  $V/V_0$  as parameter. It is noted that high efficiencies of collection of the +10 micron particles can be achieved at velocities up to  $V_0$ . Higher velocities cause a rapid decrease in efficiency of particle collection.

The reason for the relatively high rate of clarification remains to be discussed. The terminal velocities at settler operating conditions, for the median and 75% particle sizes were calculated by Stokes law. The densities and viscosities of the various materials used in these calculations were taken from correlations previously reported.<sup>(3)</sup> The results are shown in Table VIII.

It is immediately apparent, that efficient capture is achieved for many of the particles at upflow velocities greatly exceeding the terminal velocity. Part of this phenomenon is due to the capture or "trapping" of fine particles by the aggregate of particles in the "hindered" settling system. Experiments in model systems of fine Ireland coal dispersed and settling in hydrocarbons confirmed this phenomenon. However, the rate of settling of these slurries was considerably slower than that of the extraction effluents having similar nominal particle size distribution.

It is concluded that to a considerable degree, the high rate of settling is attributable to the agglomeration of the fine residue particles with the precipitated extract behaving as the "binder". The higher settling rate of the 2/1 versus the 1.5/1, solvent-to-coal ratio slurries, is consistent with this assumption and the observation that a higher percentage of extract is precipitated on cooling the higher solvent-to-coal ratio slurries.

Incidentally, the apparent anomaly of lower extract solubility at higher solvent/extract ratios is consistent with earlier measurements.<sup>(1)</sup>

#### Browning Mine Coal

The deashing results of the continuous operations, as in the previous case of Ireland coal, are consistent with the batch data, as can be seen by comparison of data in Tables VI and VII.

Particle size distribution of extraction residue for the Browning Mine coal is shown in Figure 6. Comparing results with Ireland coal, it is seen that the residue particles are finer and unlike the Ireland case, their size is relatively insensitive to extraction conditions.

The smaller particle size is one reason for the low settling rate of the Browning Mine extraction effluents. Another reason is that in contradistinction to the Ireland coal case, little or no extract comes out of solution on cooling.

The function of decane addition likely is to precipitate a portion of the extract and thus cause agglomeration of the fine residue particles to enhance their settling rate. Like the Ireland case, the precipitated extract is largely composed of benzene insolubles.

The efficiency of collection of fine particles is better for the Browning Mine coal after addition of decane than in the corresponding case for Ireland Mine coal where no decane is used. This can be seen by comparison of Figures 7 and 8. It is concluded that decane addition is very effective in causing agglomeration of fine particles.

Extensive data have not been obtained with other coals, but subbituminous coals from Montana behave similarly to the Browning Mine coal. Illinois bituminous coals show an intermediate behavior, i.e., settling may be achieved without additive, but not at as high a rate as in the Ireland coal case.

The extracts of lower rank coals appear to be lower in benzene solubles, and this may also explain the fact that extract precipitation does not occur on cooling but requires addition of a deasphalting solvent. A comparison, given below, of the residuum overflow extract for two "comparable" runs with Ireland Mine and Browning Mine coal illustrates the lower benzene insoluble content for the extract in the latter case:

Coal	<u>Ireland Mine</u>	<u>Browning Mine</u>
Condition or Run No.	Condition C	Run 45
Solvent/Coal Ratio	2	2
Extraction Depth	76	68
<u>Overflow Residuum Extract</u>		
Solvent Fractionation, Wt %		
Benzene Insolubles	33.2	20.1
Asphaltenes	39.4	48.5
Heavy Oil	27.3	31.4

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TABLE I

Analysis of Coal Feedstocks

Coal	Ireland Mine	Browning Mine	
		Batch A	Batch B
<u>Proximate Analysis, MF Wt %</u>			
Volatile Matter	42.4	41.1	40.9
Fixed Carbon	50.0	51.3	51.9
Oxidized Ash	7.6	7.6	7.2
<u>Elemental Analysis, MF Wt %</u>			
Hydrogen	5.36	5.05	5.03
Carbon	75.75	74.58	76.06
Nitrogen	1.44	1.39	1.31
Oxygen (difference)	6.67	10.32	10.06
Sulfur (total)	3.87	1.06	0.40
Sulfur (pyritic)	1.74	0.32	0.07
Sulfur (organic)	2.13	0.74	0.33
<u>Wet Screen Analysis, MF Wt %</u>			
+60 mesh	0	0	0
60 x 100 mesh	3.8	1.9	26.2
100 x 200 mesh	28.9	26.2	26.6
200 x 325 mesh	32.1	25.0	16.9
-325 mesh	35.2	46.9	30.3
Heating Value, Btu/Mf lb	13,407	--	13,402
Max. Gieseler Fluidity, DDPM at Temperature, °F	> 24,000 at 774°F	← < 1 →	

TABLE IIAnalysis of Natural SolventsSource

<u>Cresap Run No.</u>	<u>56</u>	
	<u>Hydro Oil</u>	<u>Recycle Oil</u>
<u>Ultimate Analysis, Wt %</u>		
Hydrogen	9.56	9.08
Carbon	90.04	89.92
Nitrogen	0.37	0.16
Oxygen (direct)	0.24	0.60
Sulfur	0.15	0.25
<u>TBP Distillation, Wt %</u>		
IBP °C	180	200
IBP x 250°C	34.2	35.4
250 x 300°C	27.9	48.9
300 x 350°C	20.0	9.5
350 x 474°C	8.9	3.6
+474°C	7.7	2.0
Holdup + Loss	1.3	0.6
<u>Gas Chromatographic Analysis, Wt %</u>		
Light Ends	23.0	12.9
Tetralin	2.7	0.9
Unidentified	20.0	11.2
Naphthalenes	3.1	3.6
Unidentified	5.9	2.3
Methylnaphthalenes	11.9	19.2
Higher Boilers	33.4	49.9

TABLE IIIBatch Extraction and Settling  
Results with Ireland Mine CoalA. Experimental Conditions

<u>Series</u>	<u>A</u>	<u>B</u>
Solvent	25% Hydro Oil 75% Recycle Oil	100% Hydro Oil
Solvent/MF Coal, Wt. Ratio	1.5	1.5
Extraction Temperature, °F	710	710
Extraction Residence Time, min.	60	60
Settling Temperature, °F	600	600

B. Experimental Results

Extraction Depth, % of MAF Coal	77	86
Initial Settling Rate, in/min	2.0	0.4
<u>Properties of Extract in Clarified Layer, Wt %</u>		
Cresol Insolubles	3.3	2.8
Total Ash	0.14	0.27
Fine Ash (-.45 $\mu$ dia.)	0.11	0.14

TABLE IV

Summary of Continuous Extraction ResultsA. Conditions

Condition	<u>A</u>	<u>B</u>	<u>C</u>	<u>D</u>
<u>Recycle Solvent</u>	3	0	3	0
Hydro Oil				
Solvent/MF Coal	1.5	1.5	2	2
Slurry Feed Rate, lb/hr	30.5	13.8	30.6	28.9
Extraction Temperature, °F	750	727	740	745
Residence Time, min	24	54.8	24.1	25.2

B. Results

<u>Yields, Wt % MAF Coal</u>				
Conversion	78.3	85.1	76.4	85.5
Extract + Polymer	84.9	86.3	77.0	85.0
Water	2.4	3.7	1.4	1.0
Gases + H <sub>2</sub> S + NH <sub>3</sub>	1.2		0.9	1.3
MAF Residue	<u>21.7</u>	<u>14.9</u>	<u>23.6</u>	<u>15.6</u>
Total	110.2	104.9	102.9	102.9
Polymerized Solvent Plus				
H <sub>2</sub> Transfer, Wt % MAF Coal	10.2	4.9	2.9	2.9

TABLE V  
Effect of Extraction Conditions  
on Settler Performance

Extraction Condition	<u>A</u>	<u>B</u>	<u>C<sup>(1)</sup></u>	<u>D</u>	<u>D<sup>(2)</sup></u>
Solvent/MF Coal, Wt Ratio	1.5	1.5	2	2	2
Coal Conversion, Wt %	78	85	76	84	85
Settler Temperature, °F	←————— 600 —————→				
Overflow Rates, lb/hr-ft <sup>2</sup>	247	239	516	489	524
<u>Wt % of Ash Out</u>					
To Overflow	2.5	33.2	1.5	1.4	2.0
To Underflow	97.5	66.8	98.5	98.5	98.0
<u>Extract Quality, Wt %</u>					
Wt % Total Ash	0.22	3.30	0.16	0.16	0.21
Wt % Fine Ash (-.45 μ)	0.08	0.12	0.04	0.09	0.06
Extract Recovery in Overflow, Wt % of Feed to Settler	86.6	88.3	84.9	84.2	85.6

(1) 135% stoichiometric H<sub>2</sub>SO<sub>4</sub> added.

(2) 106% stoichiometric H<sub>2</sub>SO<sub>4</sub> added.

TABLE VI

Batch Extraction and Settling Results  
 With Browning Mine Coal  
 Settler Temperature = 600°F

Extraction Temperature, °F	735	737	768	737	774
Time at Temperature, min.	30	30	30	20	21
Extraction Solvent	100% Methylnaphthalene	75% MN 25% tetralin	75% MN 25% tetralin	75% Sunoco FS <sup>(1)</sup> 25% tetralin	50% MN 50% tetralin
Solvent-to-MF Coal Wt Ratio	4.0	3.8	1.5	3.0	2.08
Extraction Depth, Wt % MAF Coal	36.1	36.1	58.1	53.7	67.4
<u>Decane</u> Extraction Solvent Wt Ratio	0	0.06	0	0.105	0.251
Initial Settling Rate, in/min	0	0.22	0.10	0.25	> 1.4

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(1) Petroleum derived solvent mainly consisting of mixed methyl- and dimethyl naphthalenes.

TABLE VII

Continuous Extraction and Settling  
Results with Browning Mine Coal  
 Extraction Solvent/MF Coal = 2.08  
 Feed Solvent = 49.4% tetralin-50.4% methylnaphthalene  
 Settler Temperature = 600°F  
 Extraction Temperature = 775°F

Run No.	<u>45-1</u>	<u>45-2</u>	<u>45-3</u>	<u>45-4</u>	<u>45-5</u>
Slurry Feed Rate, lb/hr	14.7	15.0	15.1	14.9	13.8
Residence Time, min	42.6	41.3	40.9	41.2	43.5
<u>Yields, Wt % MAF Coal</u>					
Conversion	69.6	66.8	68.2	69.4	67.4
Extract	58.5	51.9	52.8	55.0	60.5
Decane/Extraction Solvent, Wt Ratio	0.104	0.133	0.200	0.252	0.309
Overflow Rate, lb/hr-ft <sup>2</sup>	286	282	303	286	304
Settler Upflow Velocity, in/min	1.12	1.16	1.29	1.28	1.35
Wt % of Ash Fed to Settler in Overflow	30.7	14.0	5.9	1.1	0.8
Product Extract Ash Content, Wt %	4.05	2.35	0.95	0.24	0.12

TABLE VIII

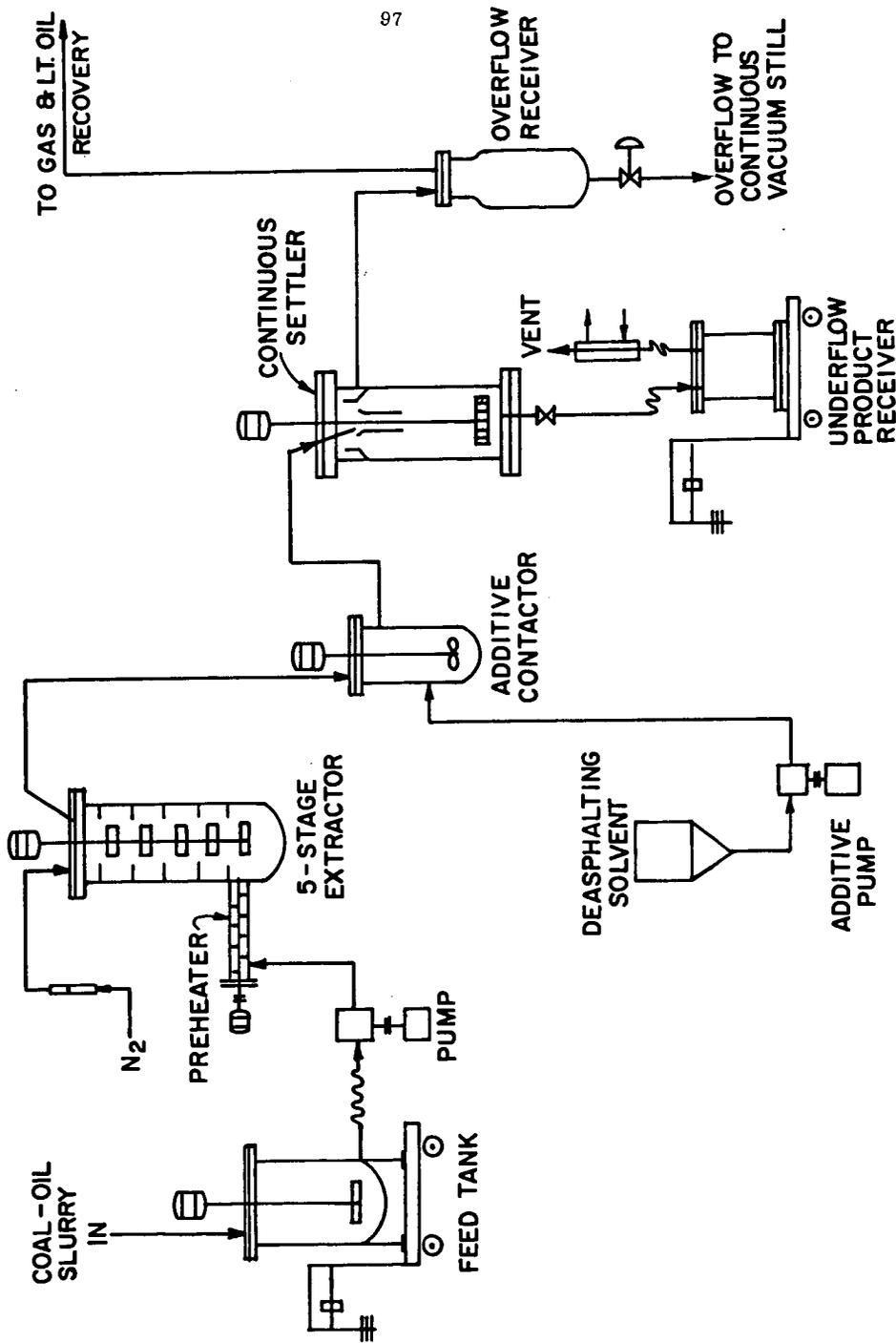
Terminal Velocities of Extraction  
Residue Particles

<u>Condition</u>	<u>A</u>	<u>B</u>	<u>C</u>	<u>D</u>
Solvent/Coal Ratio	1.5	1.5	2	2
Conversion	78	85	76	85
Median <sup>(1)</sup> Particle Size, $\mu$	20	12	28	16
Terminal Velocity, in/min	0.42	0.15	0.82	0.27
75% <sup>(2)</sup> Particle Size, $\mu$	54	22	70	34
Terminal Velocity, in/min	3.1	0.51	5.1	1.2
Initial Settling Velocity of Slurry	2.0	0.4	--	--

(1) 50% of particles smaller than size given.

(2) 75% of particles smaller than size given.

Figure 1  
Hydrogen Donor Extraction Unit With Settler



## Figure 2 Ash Breakthrough To Settler Overflow

1-1/2:1 S/C Ratio    70-80% Conversion    Condition A

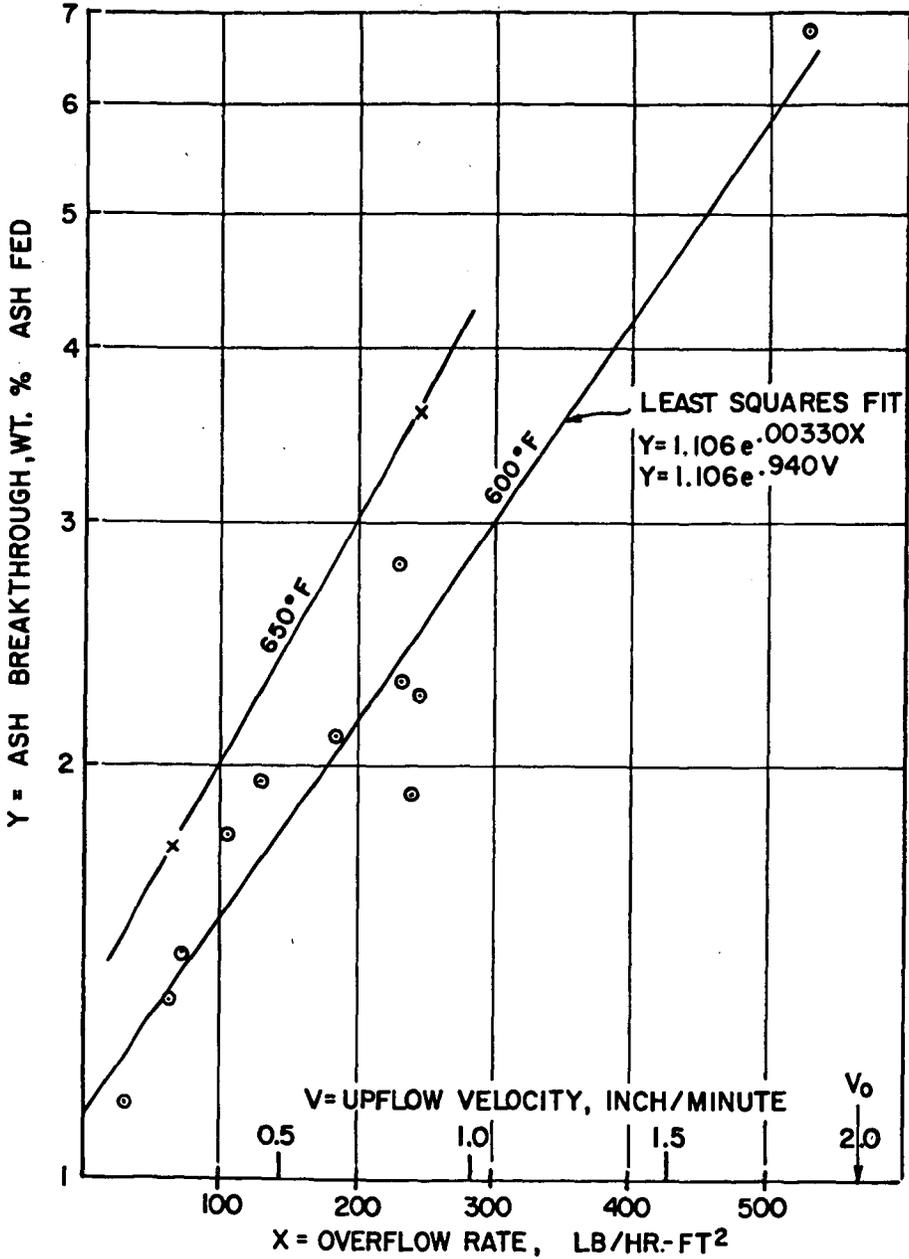


Figure 3

## Relation Between Coal Conversion & Hydrogen Transfer Browning Mine Coal

Point	Run 197.01	% Tetralin In Solvent	Temperature	Residence Time (Min.)	S/C Ratio	Coal Batch
1	41	0	749 °F	24.1	4	A
2	35	50	713	20.9	1.5	A
3	44	28.9	746	23.5	3	A
4	40	50	754	26.8	1.5	A
5	34	50	749	42.4	1.5	A
6	46	50	770	42.6	2	A
7	51	50	772	43.4	2	B
8	36	50	774	41.5	1.5	A

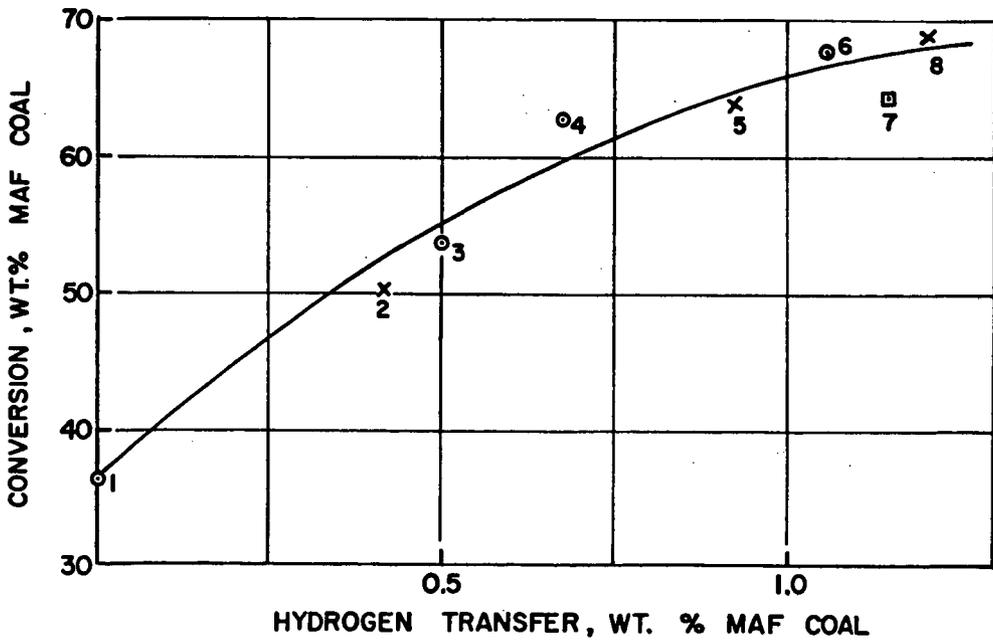


Figure 4  
Comparison Of Observed And Predicted  
Hydrogen Transfer From Continuous Extraction  
Runs With Browning Mine Coal

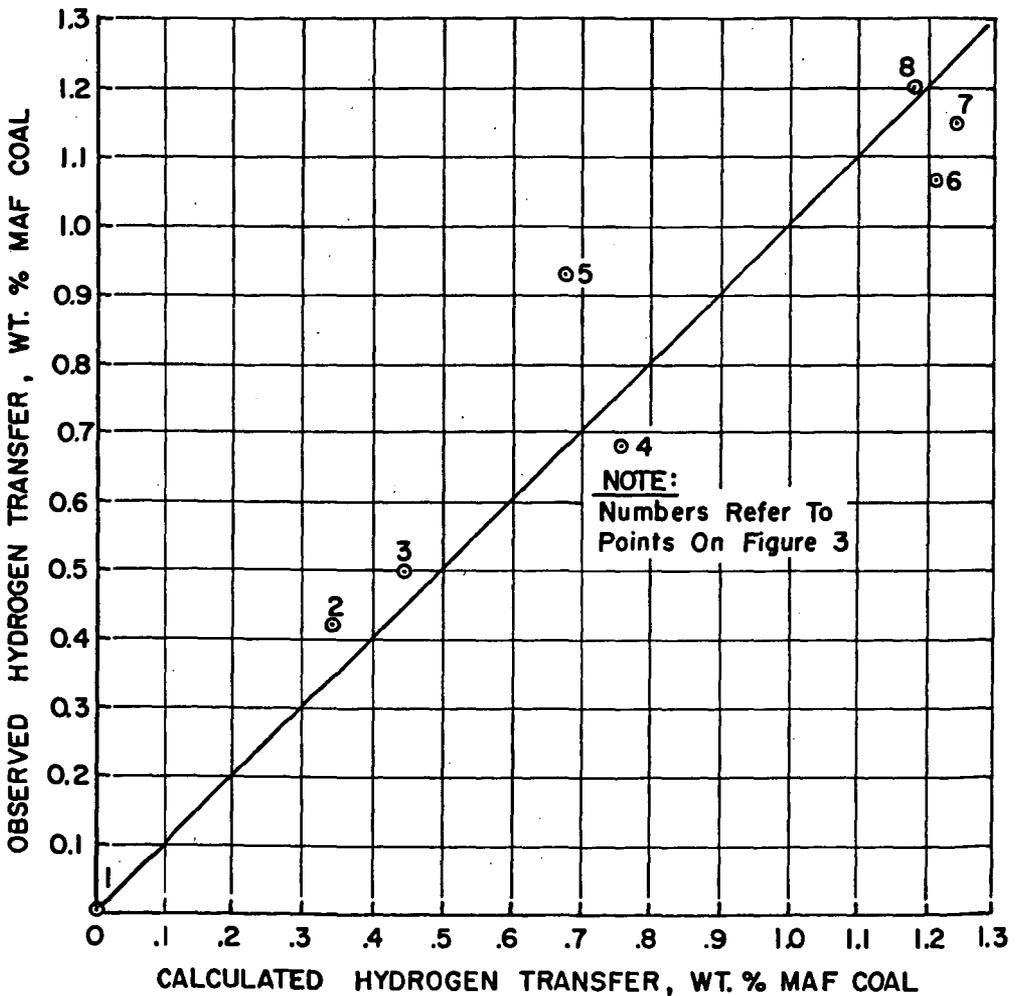


Figure 5

### Effect Of Temperature And Decane Proportion On Settler Separation Efficiency

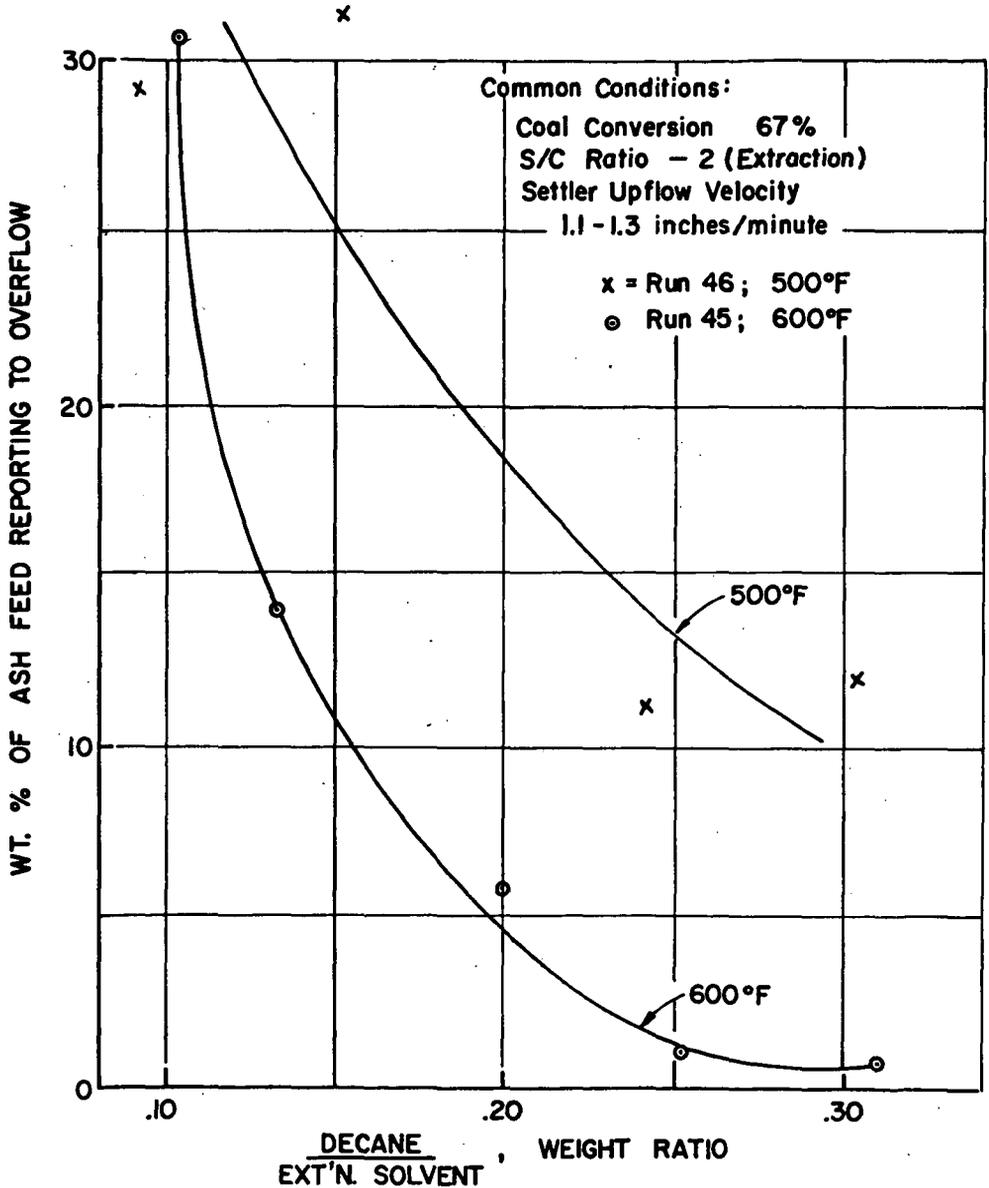


Figure 6  
Particle Size Of Insolubles

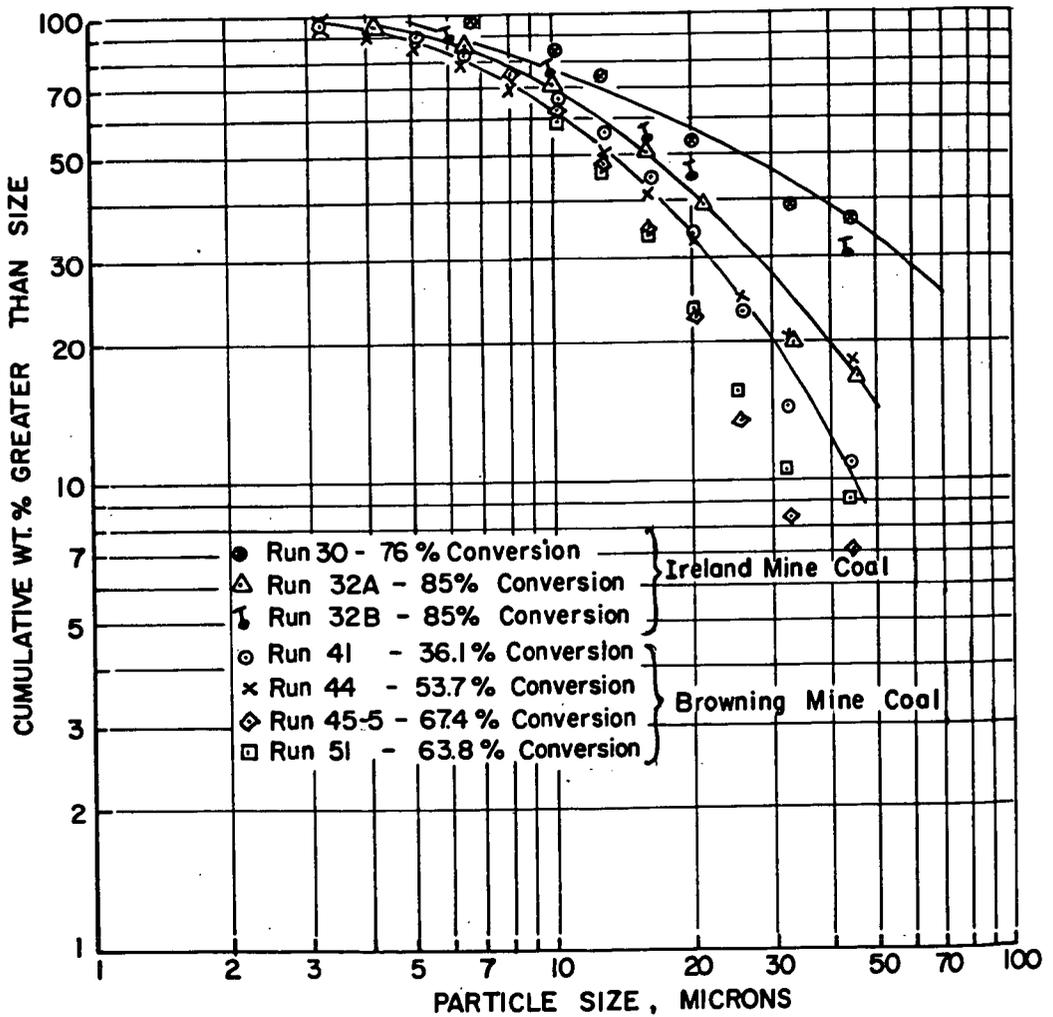


Figure 7  
 Fractional Removal Efficiencies Of Residue Particles  
 Ireland Mine Coal - Solvent/Coal Ratio = 1:5

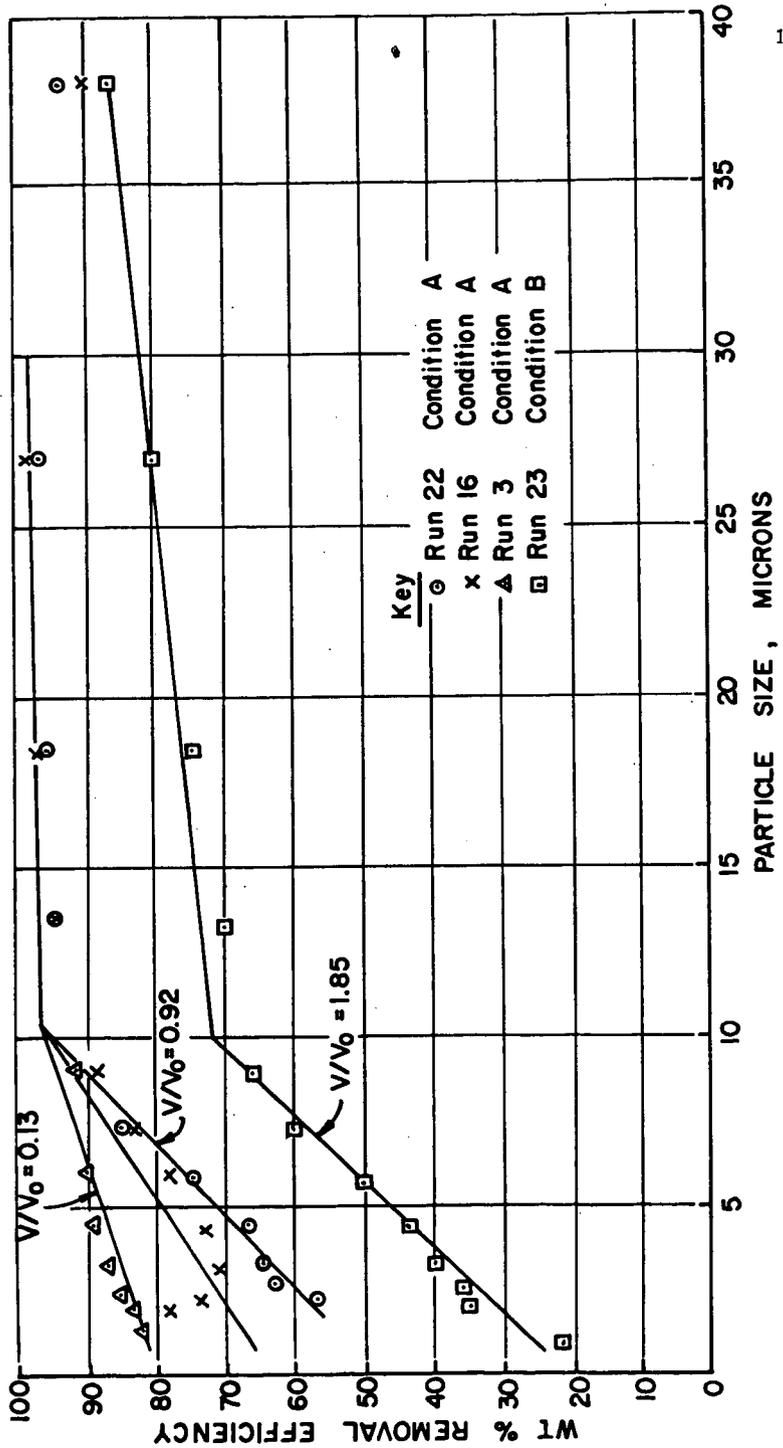


Figure 8  
 Fractional Removal Efficiencies Of  
 THF Insoluble Extraction Residue  
 Runs 41, 44 and 51

