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Paul W. Smith
Desulfurization of Coal using Freon-113
as a Dense Media Separation Fluid

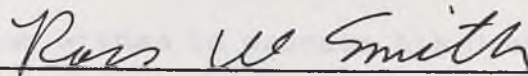
Frank Diehl
A thesis submitted in partial fulfillment of the
requirements for the degree of Master of Science
in Metallurgical Engineering

by

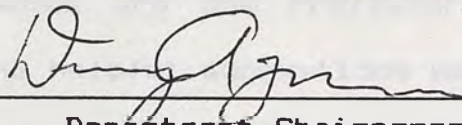
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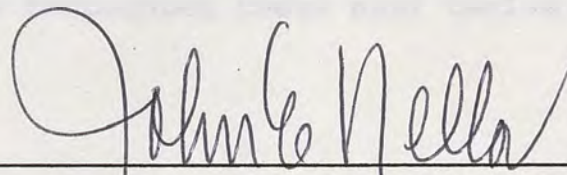
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ABSTRACT

An anhydrous heavy liquid gravity separation process for the beneficiation of fine coal was investigated. The parting liquid is Freon-113 (1,1,2-trichlorotrifluoroethane), a fluorocarbon having a low boiling point, low latent heat of vaporization, high density, and low viscosity. It is non-toxic, non-flammable and non-corrosive.

The fluorocarbon parting liquid can be recovered from the product coal and reject by filtration. The energy required for distillation or evaporation of the parting liquid is much reduced as compared to water due to the low latent heat of vaporization. The low viscosity of the parting liquid results in shorter separation times than are observed with magnetite suspensions.

The feed, product coal, and reject collected during float-sink tests were analyzed to determine the percent in ash, total sulfur, sulfate, pyritic and organic sulfur present. Fourteen size fractions ranging from 6M x 8M (3.35 x 2.33mm) to 400M x 0 (38 μ m x 0) were investigated.

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INTRODUCTION

In an approximate twenty year span, the growth of coal production tonnage in the United States progressed from 534 million tons in 1966 to a forecast by the National Coal Association of over 880 million tons in 1986 (Coal Age, 1986). According to the Mining Annual Review (February 1984), the U.S. at present possesses about 25% of the world's known total coal resources with estimated economically recoverable reserves of 364,000 million tons. This is sufficient to last for hundreds of years.

Tremendous changes in coal preparation technology have occurred over this period to result in more intensive and efficient methods of coal cleaning, especially in the finer sizes. McClung et al. (1979), Humphreys et al. (1979) and Wright (1985) indicate that the major factors contributing to the need for increased coal preparation are (1) depletion of the higher quality coal seams; (2) increased demand for quality brought on by environmental requirements, i.e., Clean Air Act of 1967; (3) increased extraneous dilution caused by mine health and safety laws, i.e., Coal Mine Health and Safety Act of 1969; (4) the Arab Oil Embargo of 1973-74; and (5) the secondary petroleum price increase of 1979.

McClung et al. (1979) states that the impurities present in coal are classified into ash forming and those that contribute sulfur. From the viewpoint of coal cleaning, both the ash

forming and the sulfur containing impurities are subdivided into two categories, i.e., extraneous and inherent. The extraneous impurities are segregated and can be eliminated by available cleaning methods to whatever extent is economically justified. The inherent impurities are inseparably combined with the coal.

The principal ash forming minerals present in coal were compiled by Nelson (1953), who reported that probably 95% of the mineral matter associated with normal marketable coals consists of species belonging to the shale, kaolin, sulfide, and chloride groups. Table 1 shows the minerals that may occur in bituminous coals. The species are listed in approximate order of the amount present within each group. Table 1 is presented in Appendix I.

Sulfur is recognized as one of the major impurities in coal. It is generally accepted that sulfur exists in coal as pyritic sulfur, organic sulfur, sulfate salts and elemental sulfur.

Pyritic and organic sulfur together account for almost the total sulfur content, occur in varying amounts in different coals, and may vary greatly in coal of the same seam. Sulfate salts occur mainly as gypsum and iron sulfate. The former is present in fresh coal and the latter results from the oxidation of the iron pyrite during the storage of coal. Sulfate sulfur usually accounts for less than 1% of the total sulfur content (Miller, 1964). The concentration of elemental sulfur in coal is also small, being generally less than 0.2% of the total sulfur content (Greer, 1979).

Pyrite, FeS_2 , the primary inorganic source of sulfur in coal, occurs in two crystalline habits, i.e., pyrite (cubic) and marcasite (orthorhombic). Pyrite is the more common so that pyrite and marcasite are often referred to collectively as pyrite (Morrison, 1981). According to McClung et al. (1979), macroscopic pyrite occurs predominately in coal in four forms:

VEINS: generally thin and filmlike along the vertical joints but may be as much as several inches wide and contain large pyrite crystals with well developed crystal faces.

SEAMS: extremely variable in shape and size but generally flattened and elongated in cross section, ranging in size from a fraction of an inch thick by several inches in diameter to several inches thick and hundreds of feet in lateral extent.

NODULES: roughly spherical in shape and from inches to several feet in diameter.

PYRITIZED PLANT TISSUE: often included with carbonate minerals in a coal ball, which is a portion of the coal in which the plant material has undergone replacement by inorganic material rather than coalification.

In addition to the macroscopic pyrite, much of the pyritic sulfur in coal is present in forms that can be observed only with the aid of optical or electron microscopes.

One of the earliest studies of the occurrence of pyritic sulfur in coals was done by Thiessen (1920). In general, he indicated that all coals contained microscopic grains of pyrite which are distributed at random and usually occurred in colonies. Furthermore he suggested that much of the pyritic sulfur could be in amicroscopic form (not visible with an ordinary microscope). Although, other studies such as, McCartney et al. (1969) offer somewhat different findings, i.e., the fine pyrite is much more uniformly distributed than coarse pyrite.

By means of scanning electron microscopy very detailed results have been obtained concerning occurrence and distribution of pyritic sulfur in coal. This provides accurate and precise fundamental information which is obtainable by no other technique. According to Morrison (1981) a significant portion of microscopic pyrite in coal occurs as:

NODULES: these consist of framboids (after framboise, the French for raspberry) which range from a few micrometers to several hundred micrometers in diameter, with most of them being approximately 10 to 20 micrometers in diameter. The framboids are assemblies of octahedral crystals.

DISCRETE CRYSTALS: these individual pyritic crystals may be widely dispersed throughout the coal. They occur predominantly in a size range of about 1 to 40 micrometers in diameter, with the large majority of the crystals being between 1 and 2 micrometers in diameter. This form of pyrite is not removed completely by physical coal cleaning methods as it is uneconomic to grind coal to this order of size.

Little is known about the organic sulfur in coal. However, it is generally assumed that the organic sulfur can be described by the following classification (McClung et al., 1979; Morrison, 1981):

- Aliphatic or aromatic thiols
(mercaptans, thiophenols) $R-SH$, $Ar-SH$
- Aliphatic, aromatic or mixed sulfides
(thioethers) $R-S-R$, $Ar-S-Ar$, $R-S-Ar$
- Aliphatic, aromatic or mixed disulfides
(bisthioethers) $R-S-S-R$, $Ar-S-S-Ar$, $R-S-S-Ar$
- Heterocyclic compounds of the thiophene type
(dibenzothiophene)

Organic sulfur is chemically locked to the coal structure and therefore is very difficult to remove from the coal.

However, it is not possible to remove the organic sulfur without chemically altering the coal. Chapman and Jones (1954) state: "Organic sulfur, unlike pyritic sulfur does not exist as discrete particles, but is intimately associated with the coal structure, and as such it is not possible to remove it or reduce its concentration by physical means."

Organic sulfur removal is widely expected to be much more difficult than pyrite removal, at least in processes that do not significantly change the form of the coal. Furthermore, when discussing organic sulfur removal, one enters a gray area of technology where it is difficult to draw a line between simple coal desulfurization and chemically more radical coal conversion.

Any success in removing sulfur has come about by removing pyritic sulfur by physical means. The physical methods are based on the fact that pyrite is heavier than coal. The coal is broken down into small pieces and then contacted with water or some other liquids; the difference in specific gravity between ash (or pyrite) and coal allows float and sink separation. But there is a pyrite removal dilemma: If the pyrite content is finely dispersed, it is necessary to crush the coal finely. However, the more the coal is crushed, the more coal will be subsequently lost with the pyrite reject fraction. Therefore, removal of sulfur in the pyritic form is the subject of this investigation.

It is useful to develop a bridge between the pyritic sulfur

distribution in coal and methods to remove the pyrite. A considerable amount of the pyrite in coal can be individual pyrite crystals, each generally of the order of a micrometer in diameter, or as small framboids. The more efficient use of coal relies on considerable improvement in rational methods of preparation. It is well accepted that as the size composition of most coals is decreased, the potential for pyritic sulfur reduction and ash removal increases. A coal particle size of 6 to 10 micrometers would be excellent, and a particle size of the order of 1 to 2 micrometers would probably be optimum.

It is not surprising that coal with 50 percent or more of the sulfur present as colloidal or framboidal pyrites would be difficult to clean with so much pyrite being present in a very small size range. The amount of pyrite present of a size of 76 micrometers (200 mesh) or less in diameter is of direct practical interest. Most utilities in the U.S. crush coal to <200 mesh just prior to combustion. Therefore, the significance of this information relates to the potential for additional sulfur removal at a power plant after final coal crushing. There may be a very important potential present to decrease the coal sulfur content by removal of the very fine sulfur bearing phases (predominantly pyrite) at this stage.

Currently more than 80% of American electricity is generated by coal. Approximately one-fifth of this total utility burn of steam coal went through some type of cleaning process. The Electric Power Research Institute projects that

about 900 million tons of coal will be burned in the U.S. for electricity by 1990. However, the percentage of coal burned without benefit of precombustion cleaning will decline due to higher emission standards for sulfur dioxide. The most room for precombustion cleaning is in the area of coal fines (Wright, 1985).

The relationship between the specific gravity and the particle size is given by the following equation:

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where ρ_p is the particle density, ρ_f is the fluid density, d_p is the particle diameter, and τ is the settling time. The relationship between the specific gravity and the particle size is given by the following equation:

The specific gravity of the coal is given by the following equation:

THEORETICAL CONSIDERATIONS

According to Taggart et al. (1945) sink-float separation is the simplest method of gravitational separation available. The method is particularly suitable for coal because with any given coal the ash content has a direct and substantially constant relation to the specific gravity.

The float-sink, heavy-liquid, or heavy-media process is as simple as the names indicate. If a vessel contains a liquid whose specific gravity is intermediate between two solids that are insoluble in the liquid, a separation is possible by merely adding the solids to the liquid, stirring the slurry and removing a sink (reject) and float (product coal) fraction.

Perhaps the most significant advance in heavy-media processing involves use of the cyclone. Heavy-media cyclones use slurries that have been further thickened by finely ground magnetite that raises the specific gravity. The Department of Energy has sponsored research into fine coal heavy-media cyclone cleaning. However, the U.S. Bureau of Mines and Dow Chemical have recently done work with heavy organic liquids and the heavy-media cyclone (Roberts et al. 1970; Wright 1985).

The specific gravities of the usual impurities associated with coal compared with those of coal are shown in Table 1. Taggart (1945) indicates that a heavy-media of specific gravity between 1.50 and 1.65 would put all the distinct impurities into the sink fraction. Since, the specific gravity of raw coal

ranges in the literature from 1.23-1.72 (Leonard 1975) or 1.17 to 1.35 (Taggart 1945), depending on moisture, rank, and ash content, one would expect product coal liberated from mineral matter to float. All coal-cleaning methods in general use are methods of gravity concentration with the exception of froth flotation.

An ideal organic liquid used for gravity separation of coal should not react with the coal, be non-polar, have the desired density range, have low viscosity and a low boiling point. Further the liquid should be non-toxic and not cost prohibitive. There are many available liquids that meet one or more of the above criteria (see Table 2), but few meet all of them.

For this investigation, the organic liquid should have a density in the range of 1.2-1.7 g/cc, a boiling point between 23°C and 50°C, and a viscosity below 2.0 centipoise. While the density and viscosity ranges can vary somewhat, it is essential that the boiling point of the candidate liquid be low enough to effect an energy-efficient and complete recovery. Since even the least expensive organic liquids are relatively expensive, process economics demand the ability to effect complete recovery.

Five candidate liquids conform with the above criteria. They are:

	Density g/cc	Viscosity Cp	Boiling Point °C
a) Freon-11	1.500	0.430	24.0
b) Freon-113	1.565	0.680	47.6
c) Dichloromethane	1.320	0.450	39.8
d) Carbon Tetrachloride	1.600	1.038	76.8
e) Trichloroethylene	1.460	0.566	87.0

Of these liquids, only Freon-11 and Freon-113 are non-toxic and only carbon tetrachloride and dichloromethane are non-polar. Ideally a non-toxic liquid is desirable. The density of each liquid is slightly different, however, they all fall into the range of 1.2 to 1.7 g/cc. The viscosity of each liquid is under 2.0 cp. If there are no interactions between the liquid and the coal, then the base viscosity of the liquid will remain a constant and will be independent of the solids concentration. Carbon tetrachloride and dichloromethane have zero dipole. The others have a dipole of about 0.3 Deby's. Since the dipoles are very small and close to each other it is difficult to quantitatively make any judgements as to the effect this may have on float-sink behavior (Keller 1980). The choice of Freon-113 over Freon-11 is a matter of experimental convenience based on the higher boiling point of Freon-113. The Freon-11 vaporized spontaneously in the non-enclosed apparatus used. The cost of Freon-11 or Freon-113 is approximately \$10.00 per kilogram (Aldrich 1985).

The separation behavior under ideal conditions is

generally defined by Stokes's law:

$$V = \frac{2gr^2(\rho_2 - \rho_1)}{0.09\eta}$$

where the velocity of separation, V in cm/s, is directly proportional to the radius r (cm) of the particle squared times the difference in specific gravity between that of the particle (ρ_1) and that of the parting liquid (ρ_2). The velocity is also inversely proportional to the viscosity of the parting liquid η (centipoise). The proportionality constant is $2g/0.09$ where g is the gravitational constant (980.6 cm/s^2). The parting liquid used (Freon-113) is completely immiscible with water; thus if a water droplet were present in that parting liquid it would rise according to Stokes's law to the top of the bath and float. Furthermore, a piece of mineral matter encased in a liquid water film will rise or sink according to the same equation based on whether or not the average density of the mineral and water composite is more or less than the parting liquid.

Since the driving force for separation, $r^2(\Delta\rho)$, is dependent on the particle radius (squared), a separatory funnel containing the parting liquid when fed a distribution of different particle sizes of raw coal must act as a form of classifier. That is, in a given instant the large particles entering the funnel can be immediately removed whereas the ultrafine materials may require minutes or even hours to become

either float or sink material. Given sufficient time and equilibrium conditions, however, all particles will either float or sink irrespective of size above the limit for Brownian motion.

In the Otisca process, according to Keller et al. (1977), fine size coal can be separated nearly as effectively as coarse coal. The low viscosity of the parting liquid permits even small refuse particles to sink fairly rapidly. As a consequence, coarse and fine size coal can be cleaned together in the same bath.

However, if prior to float-sink test, raw coal particles are distributed into size fractions differing only by a ratio of the square root of two to one. A determination could be made if ash or pyritic sulfur was concentrating in a specific size fraction. Also, the lower limit for static separation of raw coal fines would be established.

A measure of the coal cleaning performance of a laboratory float-sink study is accomplished through efficiency of separation. Efficiency can be evaluated in a variety of ways based on utilization of some measure of quantity, such as recovery, and quality, such as ash or sulfur content of clean coal and refuse.

According to Schulz (1970), the basic efficiency expression for the evaluation of the concentration process applicable to the separation of a two component system is the separation efficiency, E_s . The separation efficiency (E_s) is

based on the quality of coal being improved through ash or sulfur reduction and the quantity of float particles and clean coal being recovered. In the beneficiation of fine coal, for example, the valued constituent is the clean coal and the waste is ash or sulfur compounds.

The separation efficiency, (E_s), using ash as an example, is expressed by the following (Hancock, 1947):

$$E_s = \frac{(A_f - A_r)}{(A_c - A_r)} \left[\frac{(100 - A_c)}{(100 - A_f)} - \frac{A_c}{A_f} \right] 100$$

Where A_c = % ash content of clean coal

A_f = % ash content of feed coal

A_r = % ash content in refuse

Separation efficiency, E_s , is applicable to the separation of coal and its various impurities distinguishable by their compositions on any basis, chemical or physical, with complete freedom of definition of impurities, i.e., product coal and ash or the various sulfur compounds whether organic or inorganic. The maximum value of E_s for the float-sink process must occur when the grade of impurity being removed equals that in the feed coal. A negative sign on an efficiency value signifies that the impurity being considered was concentrated in the float coal.

EXPERIMENTAL PROCEDURE

SAMPLE PREPARATION

Two sealed, 5 gallon buckets of Illinois #6 bituminous coal were received from Freeburg, Illinois. The samples were set out on plastic sheets allowing ambient moisture equilibrium to be reached. After overnight drying, ten passes through a chipmunk jaw crusher reduced the gross sample particle size sufficiently to allow a fine coal distribution throughout the fine size fractions. Fourteen separate size fractions of raw coal were prepared by dry screening the reduced gross sample in a Ro-Tap:

6M x 8M* (3.35 x 2.36 mm)	50M x 70M (300 x 212 μ m)
8M x 12M (2.36 x 1.70 mm)	70M x 100M (212 x 150 μ m)
12M x 16M (1.70 x 1.18 mm)	100M x 140M (150 x 106 μ m)
16M x 20M (1.18 x 850 μ m)	140M x 200M (106 x 75 μ m)
20M x 30M (850 x 600 μ m)	200M x 270M (75 x 53 μ m)
30M x 40M (600 x 425 μ m)	270M x 400M (53 x 38 μ m)
40M x 50M (425 x 300 μ m)	400M x \emptyset (38 μ m x \emptyset)

*M = U.S.A. Standard Sieves (I O S Standard Mesh No.)

The fourteen samples were stored under nitrogen in amber glass jars to prevent further oxidation and any photochemical reactions.

FLOAT-SINK TESTS

Float-sink tests were performed in 40 ml. glass centrifuge

tubes, each containing 5 grams of raw coal and 30 ml. of Freon-113. The float-sink tests were repeated until sufficient product coal and reject were obtained for subsequent analysis. Testing fraction 6M x 8M (3.35 x 2.36 mm) through 140M x 200M (106 x 75 μ m) was readily accomplished. However, it was impossible to statically obtain separation between float and sink through fractions \leq 200M x 270M (75 x 53 μ m). A centrifuge was used to hasten separation on these difficult size fractions. In all cases, sufficient time was allowed for complete separation before removal of the float layer.

After separation, the product coal and reject were air dried for at least 16 hours in a well ventilated area to ensure that all of the parting liquid had evaporated. At this point, the product coal and reject were essentially at equilibrium with ambient moisture and their weights could be recorded. The weight of product coal and reject obtained from each 5 gram sample of raw coal was not recorded. The total weight of product coal and reject collected from the centrifuge tubes for each size fraction was recorded. After weighing, the product coal and reject samples were stored in amber glass jars under nitrogen for future ash and sulfur determinations.

The parting liquid (Freon-113) was filtered and saved for future I.R. analysis.

ANALYTICAL SAMPLE PREPARATION RAW COAL

Analytical samples were prepared from each size fraction of raw coal by a combination of size reduction and sample splitting.

according to standard ASTM procedure. All analytical samples were stored in amber glass jars under nitrogen.

CHEMICAL ANALYSIS

All samples were analyzed for ash by standard ASTM method (please refer to Appendix III for further discussion of these analytical procedures). Total sulfur was determined by the Eschka method according to ASTM procedure. Sulfate sulfur and pyritic sulfur were determined by the methods described in U.S. Bureau of Mines Bulletin 638. In all cases, each sample was analyzed in triplicate and the average reported. The accuracy of the analytical results was compared against a standard reference material (1632a) from the National Bureau of Standards which has a certified ash and sulfur content.

INSTRUMENTAL ANALYSIS

Standard methods were followed when using infrared spectroscopy (Beckman IR-4240 Spectrophotometer), electron probe microanalysis and X-ray diffraction (Copper $K\alpha$).

RESULTS

The weight distribution of feed coal between float and sink fractions is shown in Figures 1 and 3 using 100 grams of feed coal as a basis. The feed coal distribution between float and sink is essentially constant until the 270 mesh (53 μ m) fraction. Then an irregularity presents itself as an increase in weight-percent sink. This irregularity is especially evident in the -400 mesh (38 μ m) fraction.

The analysis, of the raw feed coal after 10 passes through a jaw crusher, is tabulated and presented in Appendix II, as the Screen Analysis. In this table, the screen sizes are listed in columns 1 and 2, the percentage by weight in column 3, and the ash and sulfur percentages in columns 4 and 5. The remaining columns contain calculated cumulative results from which the weight percentage, and the ash and sulfur percentages may be read for the total product passing or retained on any of the screens used in the test. Thus, if it is desired to know the analysis of the total product on 16M x 20M (1.18mm x 850 μ m), opposite 20M (850 μ m) in column 2, could be read the % weight of 50.51 in column 6, the % ash of 27.87 in column 7, and % sulfur of 4.01 in column 8. In a similar manner, the results for the product passing 16M x 20M (1.18mm x 850 μ m) would be read opposite 20M (850 μ m), column 1 in columns 9, 10, and 11 (refer to Appendix IV for sample calculations).

These results are best compared by plotting them with size

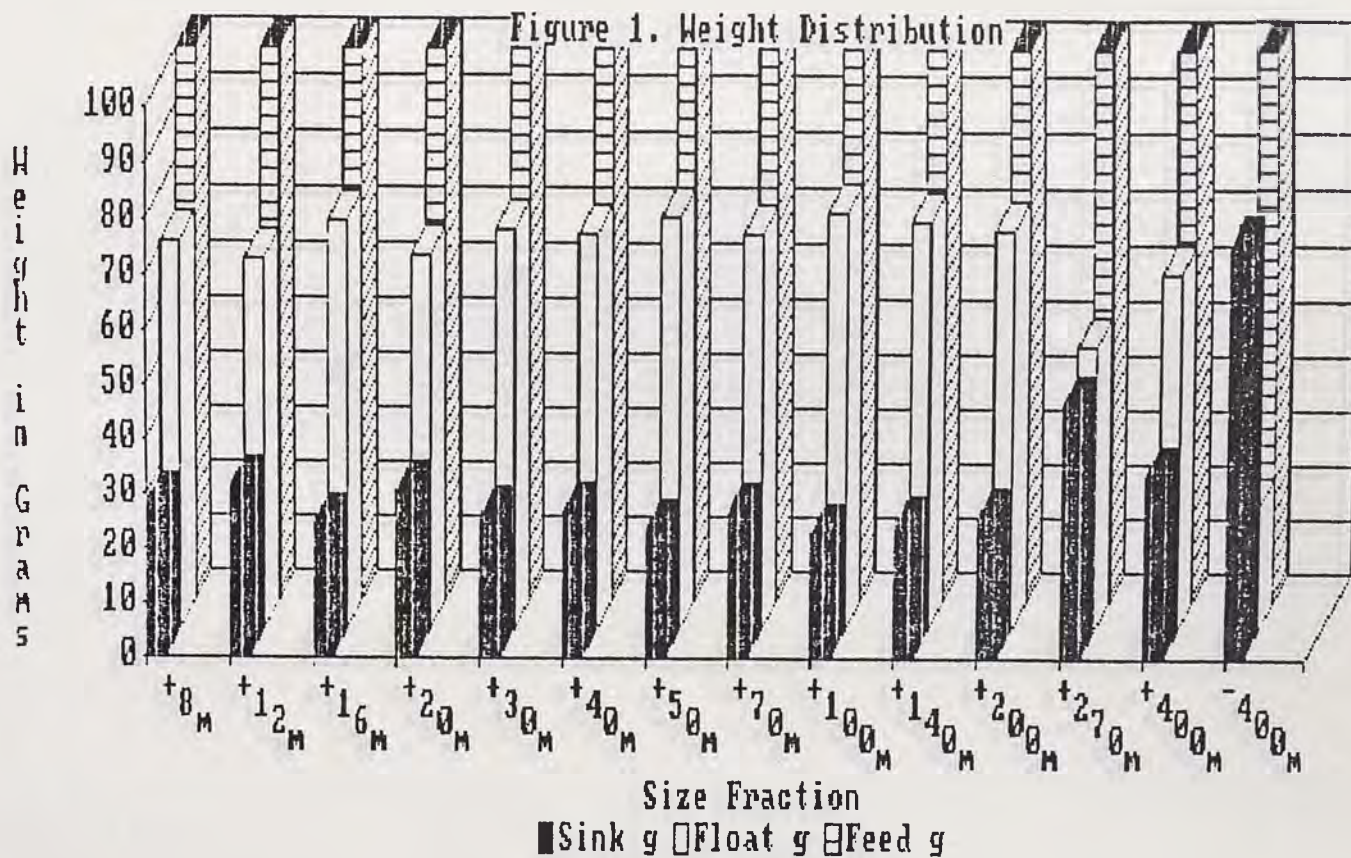


Figure 1. Comparison of weight distribution between float (product coal), and sink (reject or refuse) based on 100 grams of raw coal (feed).

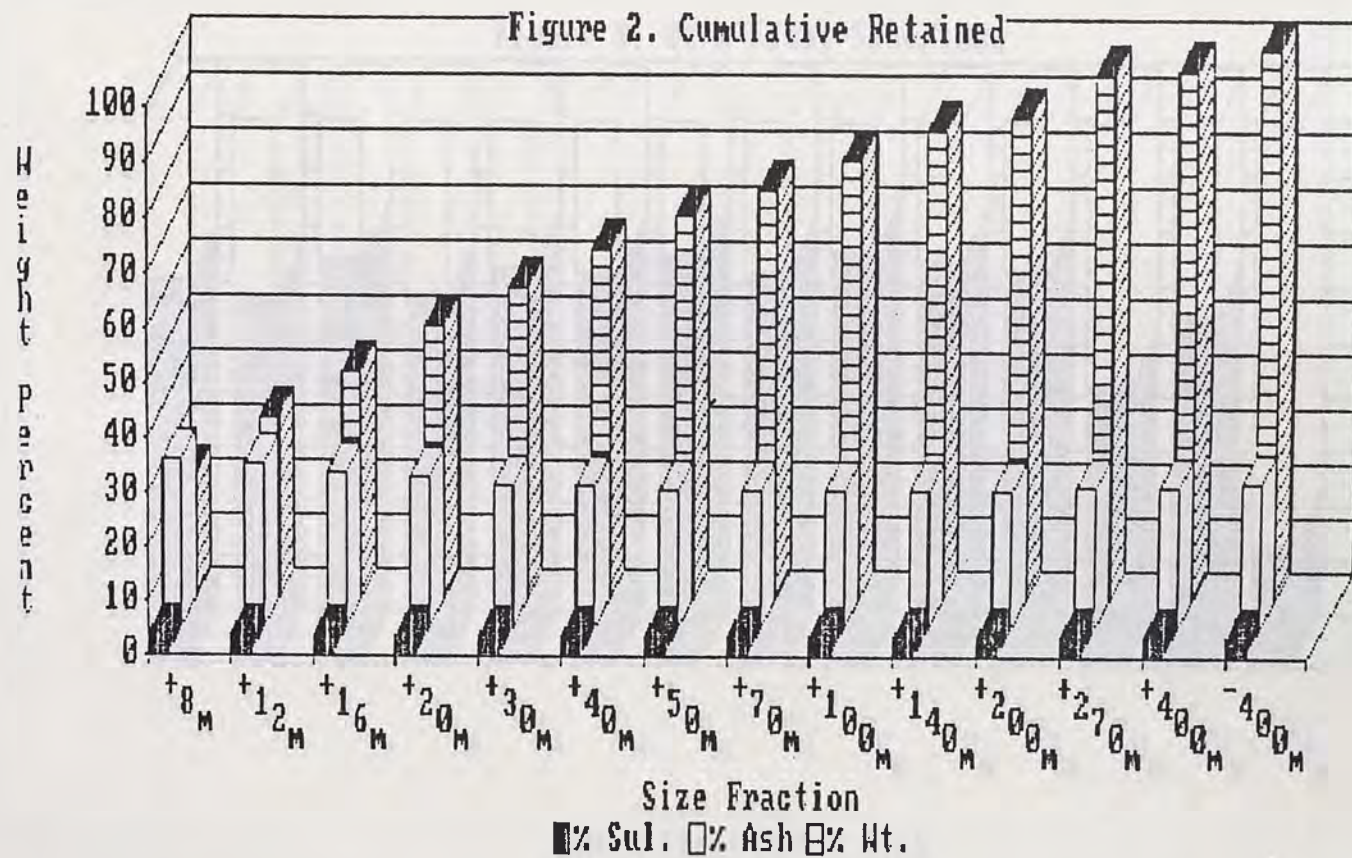


Figure 2. Comparison of cumulative retained for % ash, % total sulfur, and % weight.

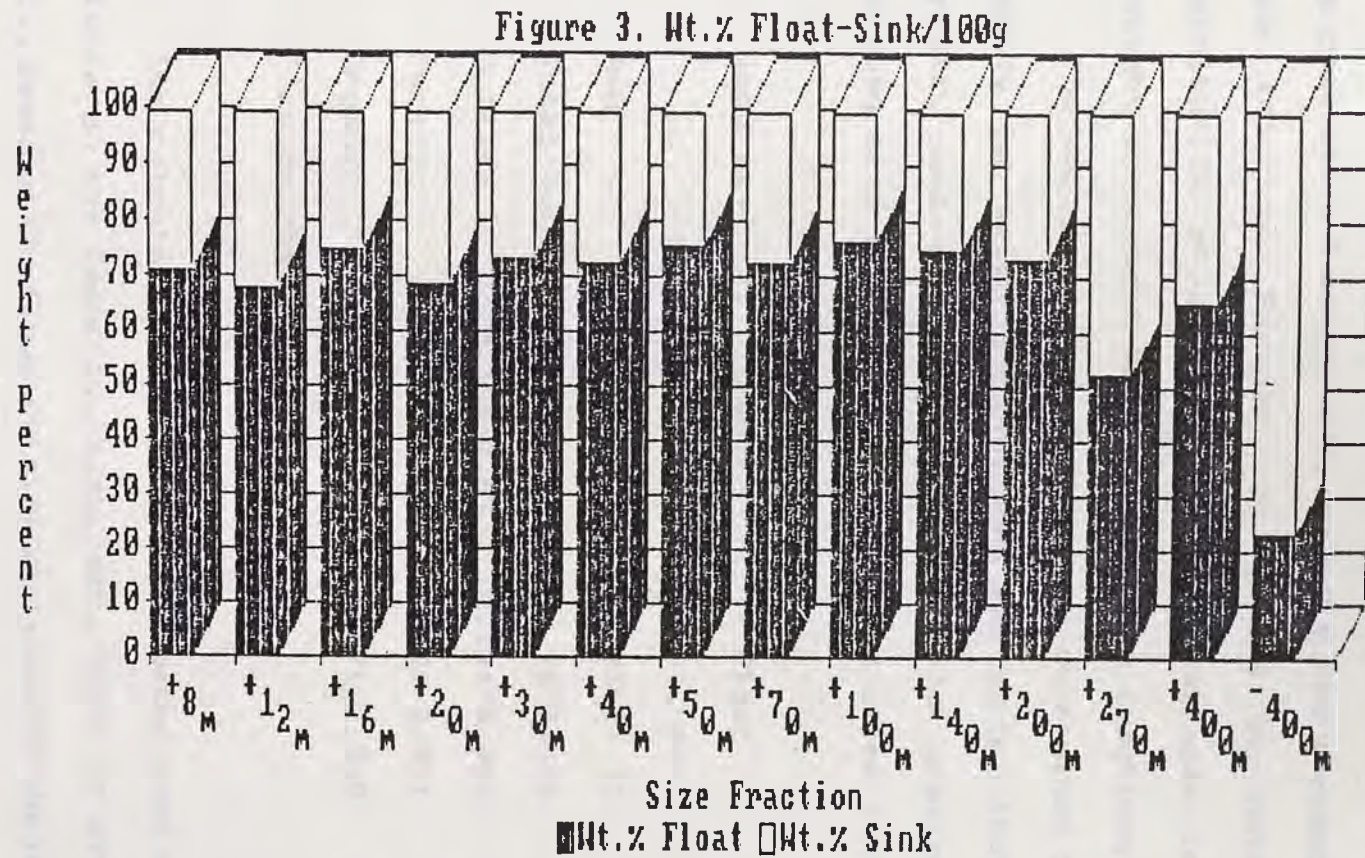


Figure 3. Stacked bar chart comparing wt. % float to wt. % sink using 100 grams raw coal as a basis.

fraction as ordinate and the weight percent as abscissa. For example, the cumulative retained (columns 6, 7, and 8) is plotted in Figure 2. The comparative 3-dimensional bar chart expresses the cumulative % weight with ash and sulfur percentages for each size fraction. Further, it should be noted that the distribution of ash and sulfur percentages is essentially constant throughout the fourteen size fractions.

The following range of values determined by analytical methods for weight-percent ash and sulfur (total, sulfate, pyritic, and organic) throughout the previously defined fourteen size fractions are tabulated below (in wt. %):

Impurity	Feed*		Float		Sink	
	min.	max.	min.	max.	min.	max.
Ash	23.80	40.44	07.95	21.15	46.40	77.65
Total sulfur	03.50	04.44	02.90	03.64	03.90	06.37
Sulfate	0.366	0.693	0.194	0.444	0.758	0.944
Pyritic	1.340	2.124	0.624	0.921	1.719	5.043
Organic	1.534	2.072	1.871	2.540	0.153	1.411

* calculated

The calculated feed values are determined by a material balance on the impurity, i.e., ash, total or pyritic sulfur, etc., based on the float and sink analysis for the impurity. The sum of impurities present in the float and sink fractions must equal the impurity present in the feed coal fraction.

Figures 4 through 8 report the following analyses: ash, total sulfur, sulfate sulfur, pyritic sulfur, and organic sulfur, respectively. The figures are comparative bar charts reporting the actual distribution of impurities of the feed coal between the float and sink fractions. The results are plotted with size fraction as ordinate and weight percent as abscissa. The weight percents are tabulated in Appendix II.

Thus, on Figure 4 it is seen that the float contains less ash than the feed and significantly less ash than the sink. The ash content of the float, feed, or sink fractions is fairly constant until the 140M x 200M (106 x 75 μ m) fraction. At this point the sink ash is decreasing while the float and feed ash is increasing. Since the ash content of the feed coal is increasing, one can say that the ash is concentrating in these fine size fractions. Furthermore, the fractions 200M x 270M (75 x 53 μ m) through 400M x 0 (38 μ m x 0) indicate that separation between float and sink coal is not as efficient as in the previous fractions, i.e., 6M x 8M (3.35 x 2.36mm) through 100M x 140M (150 x 106 μ m). The sink fractions contain the majority of ash distributed throughout the fourteen size fractions in a range of 7.95 to 21.15 wt. % compared to a range of 7.95 to 21.15 wt. % for the float. The feed ash ranges from 23.80 to 40.44 wt. %.

Figure 5 is the total sulfur analysis. This figure is termed "Total Sulfur Analysis" and is meant to show graphically the distribution of total sulfur as it occurs in each size fraction among feed coal and float-sink fractions. The term

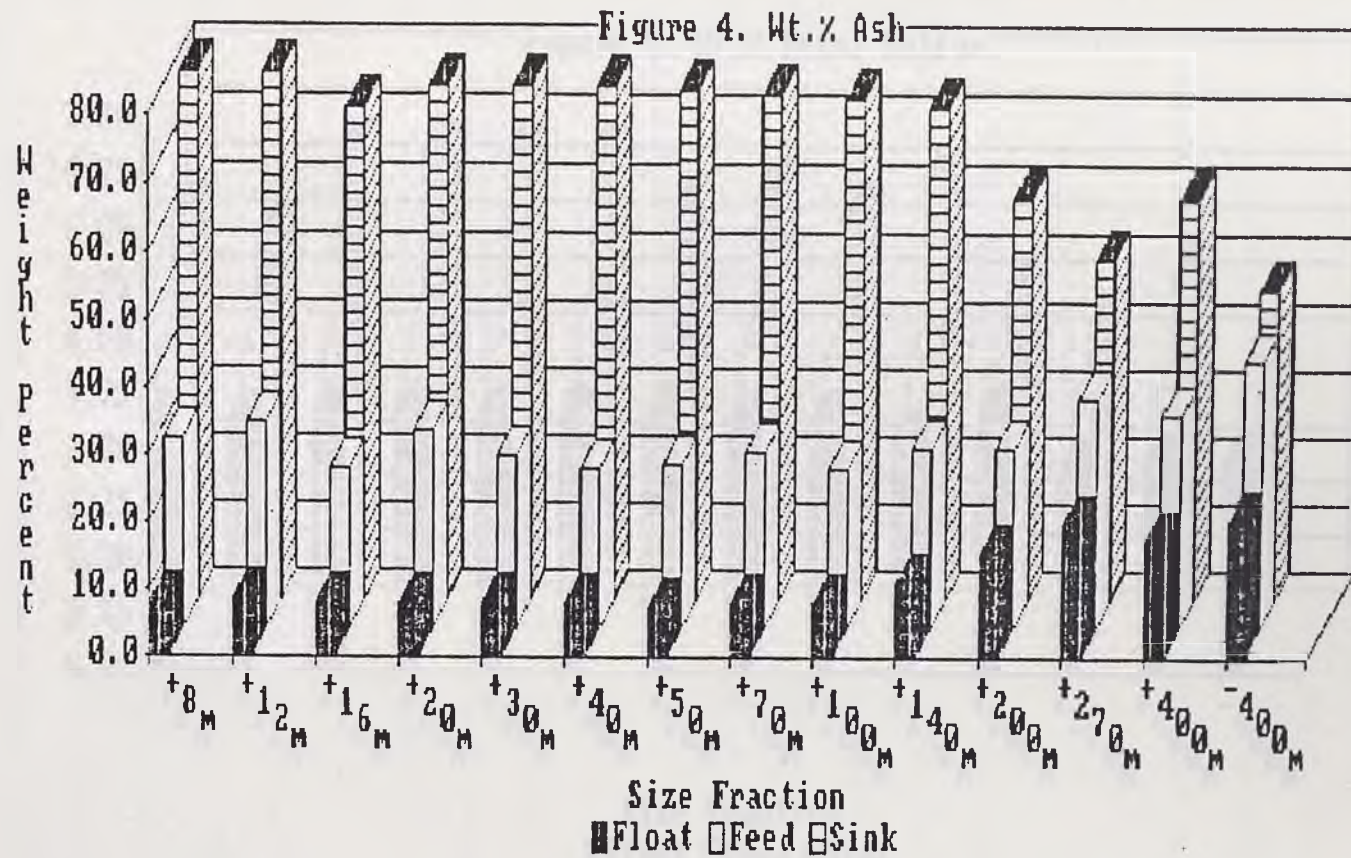


Figure 4. Comparison of assayed values for wt. % ash in feed, float, and sink fractions.

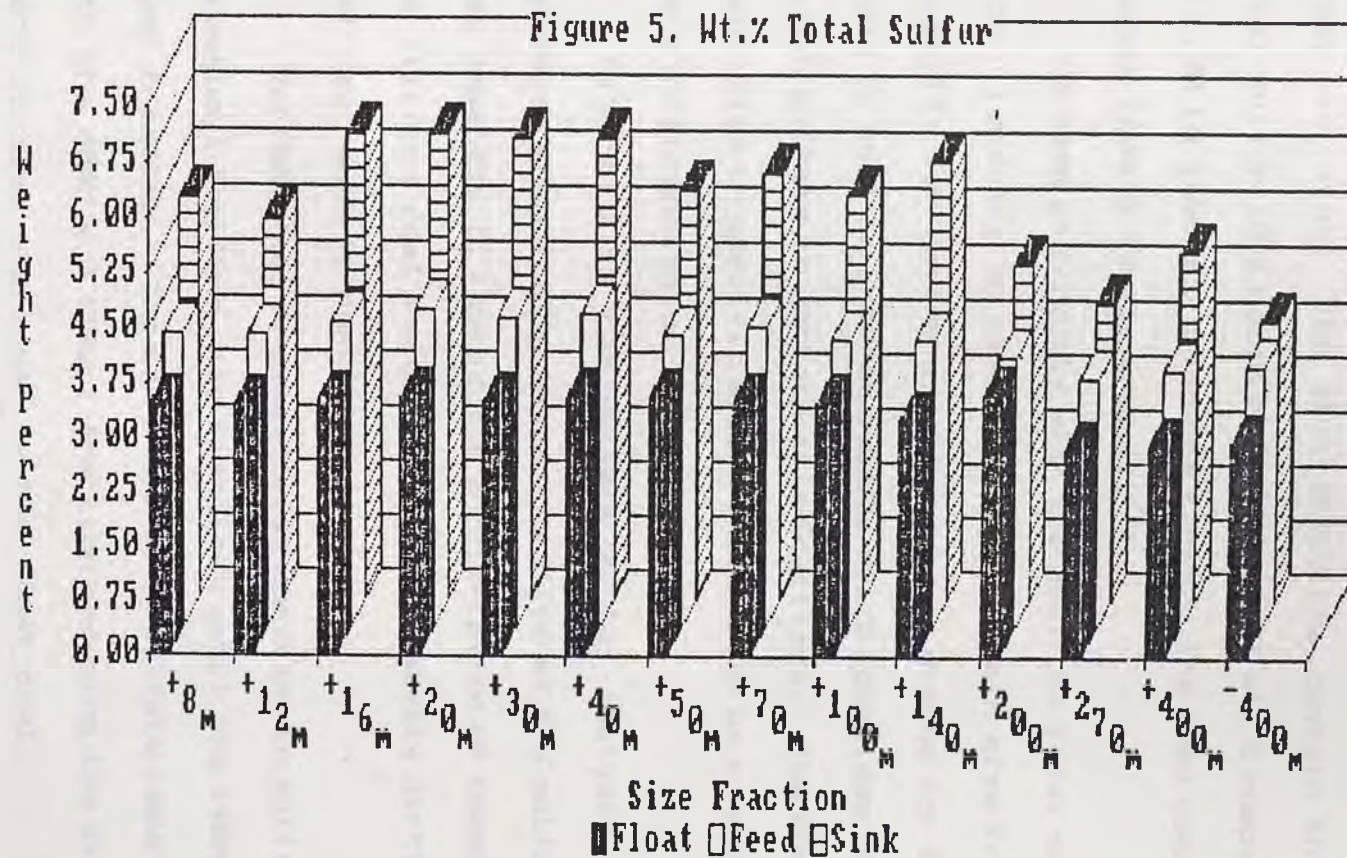


Figure 5. Comparison of assayed values for wt. % total sulfur in feed, float, and sink fractions.

"total sulfur" includes sulfate, pyritic, and organic sulfur.

For instance, it can be observed in Figure 5 that there is a moderately constant distribution of total sulfur between the float and sink. The sink fractions contain the majority of total sulfur in a range of 3.90 to 6.37 wt. % compared to a range of 2.90 to 3.64 wt. % for the float. The feed coal total sulfur ranges from 3.50 to 4.44 wt. %.

As seen previously with the ash, the total sulfur does not show a tendency to concentrate in certain size fractions. The necessity for having a series of bar graphs for the impurities found in coal is understandable since sometimes impurities tend to concentrate in certain size fractions. The concentration of impurities in specific size fractions can be a major influence in the beneficiation of fine coal.

Figure 6 is the sulfate sulfur analysis. This figure expresses graphically a concentration of the sulfate impurities (see Appendix I, Table 1 for a description of these impurities). The Illinois coal is becoming progressively dirtier in sulfate with decreasing particle size.

The feed coal is essentially constant in sulfate impurities throughout the fine size fractions until the 140M x 200M (106 x 75 μ m) fraction. Then an increase in sulfate impurities is noted with the 400M x 0 (38 μ m) fraction becoming the dirtiest. This trend is also expressed by the float coal.

The sink fractions contain the majority of sulfate sulfur in a range of 0.758 to 0.944 wt. % compared to a range of 0.194 to

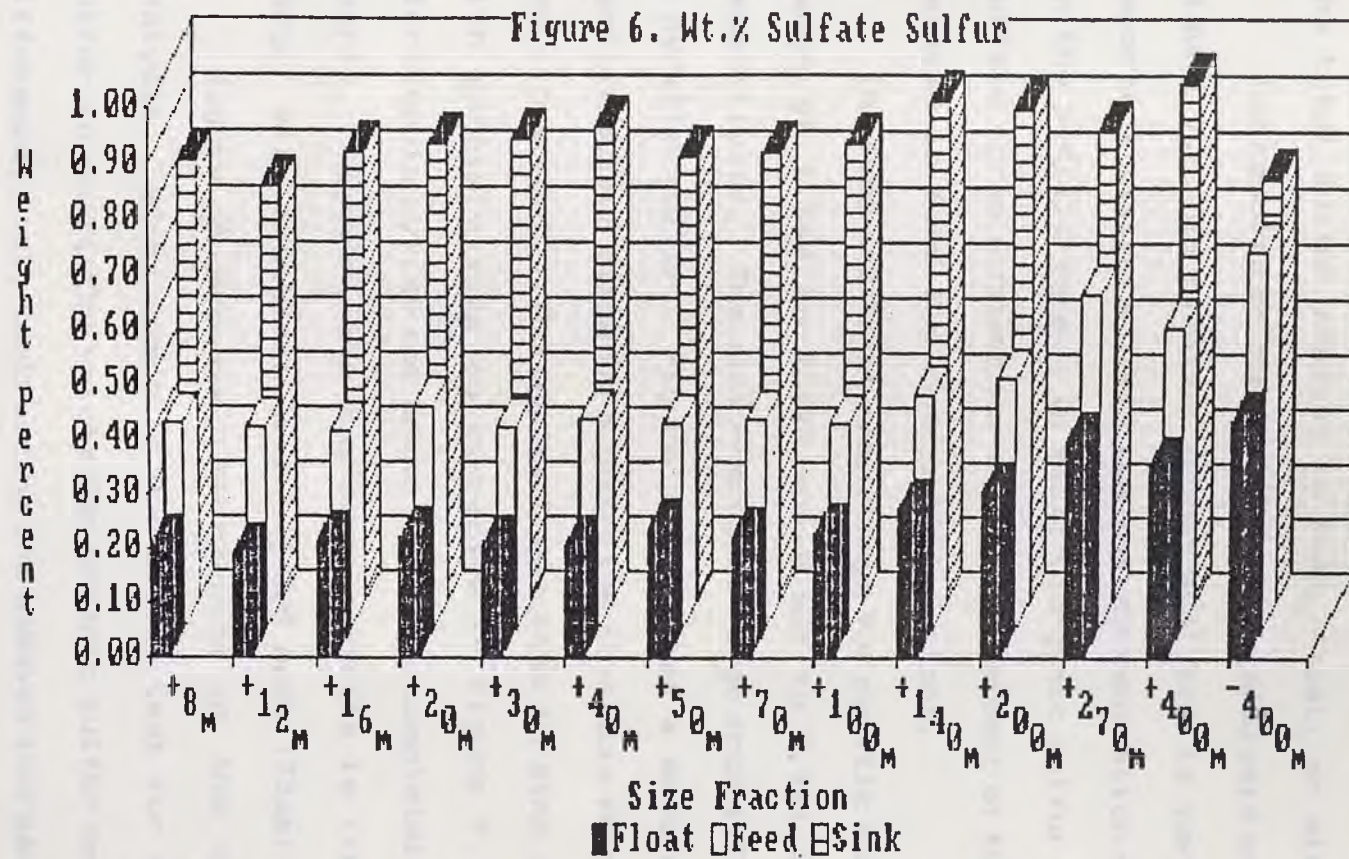


Figure 6. Comparison of assayed values for wt. % sulfate sulfur in feed, float, and sink fractions.

0.444 wt. % for the float fractions. The feed coal sulfate sulfur ranges from 0.366 to 0.693 wt. %. However, it must be noted that sulfate impurities amount to less than one percent of the total sulfur assayed in feed, float, or sink fractions.

The results of the pyritic sulfur analysis are expressed in Figure 7. The pyritic sulfur analysis is perhaps the most important analysis due to the high concentration of sulfur found in the pyritic form. In fact, inorganic sulfur in pyritic and sulfate forms account for over fifty percent of the total sulfur assayed in feed and sink coal fractions.

The feed and float coal assays for pyritic sulfur are in the range of 1.340 to 2.124 and 0.624 to 0.921 weight percent respectively. The sink fractions range from 1.719 to 5.043 wt. % pyritic sulfur. Figure 7 expresses a moderately constant pyritic sulfur value throughout the fine size fractions for feed and float coal. The apparent decrease in sink pyritic sulfur with particle size as expressed in Figure 7 is due to the microscopic pyrite not being liberated completely from the coal matrix. However, the macroscopic pyrite is liberated in the larger size fractions, i.e., ≥ 200 mesh ($75\mu\text{m}$).

Figure 8 express the results of the organic sulfur analysis. Since there is not a true test for actual organic sulfur in coal, the values for organic sulfur were obtained by difference, i.e., subtracting the assayed inorganic sulfur from assayed total sulfur.

The results are what one would expect. The majority of

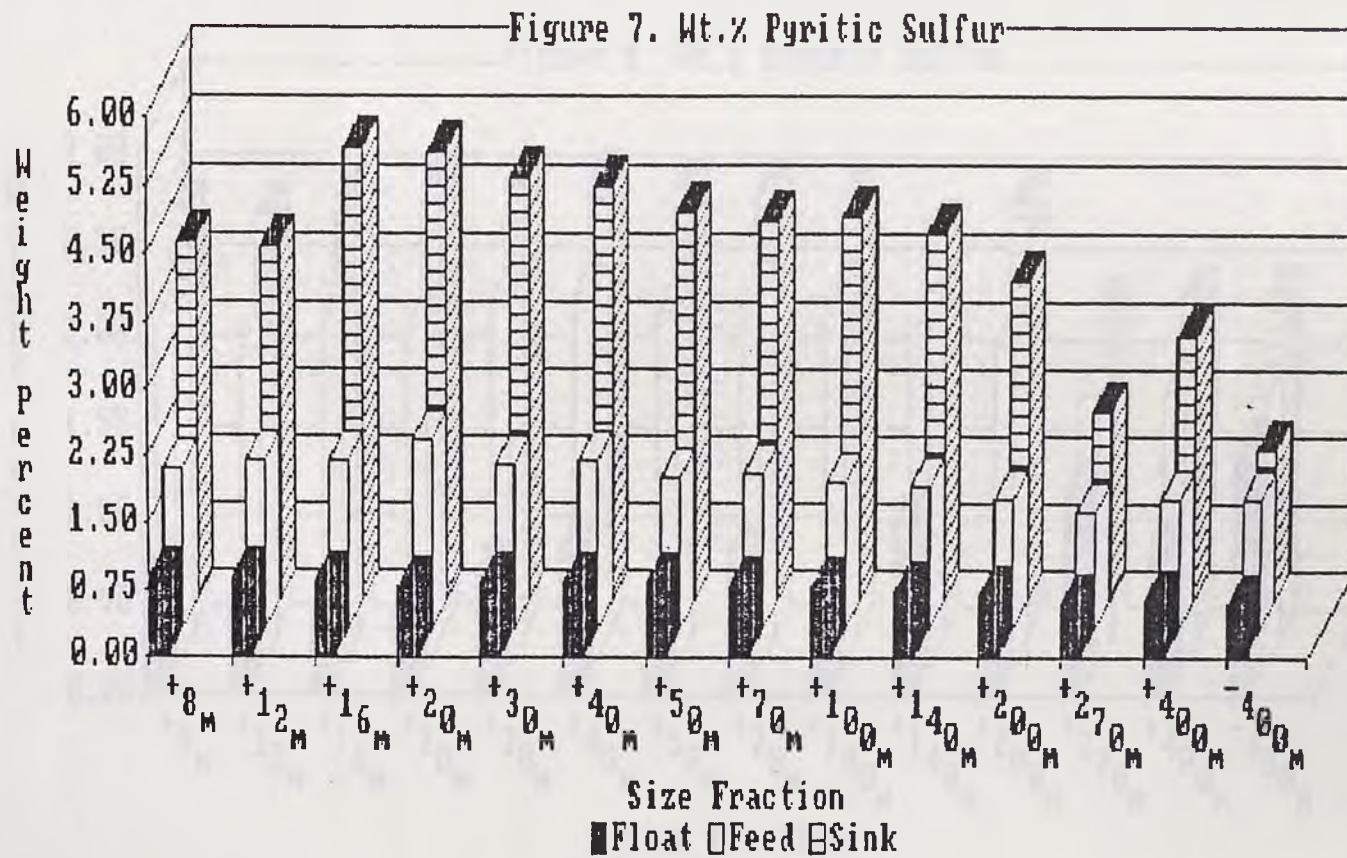


Figure 7. Comparison of assayed values for wt. % pyritic sulfur in feed, float, and sink fractions.

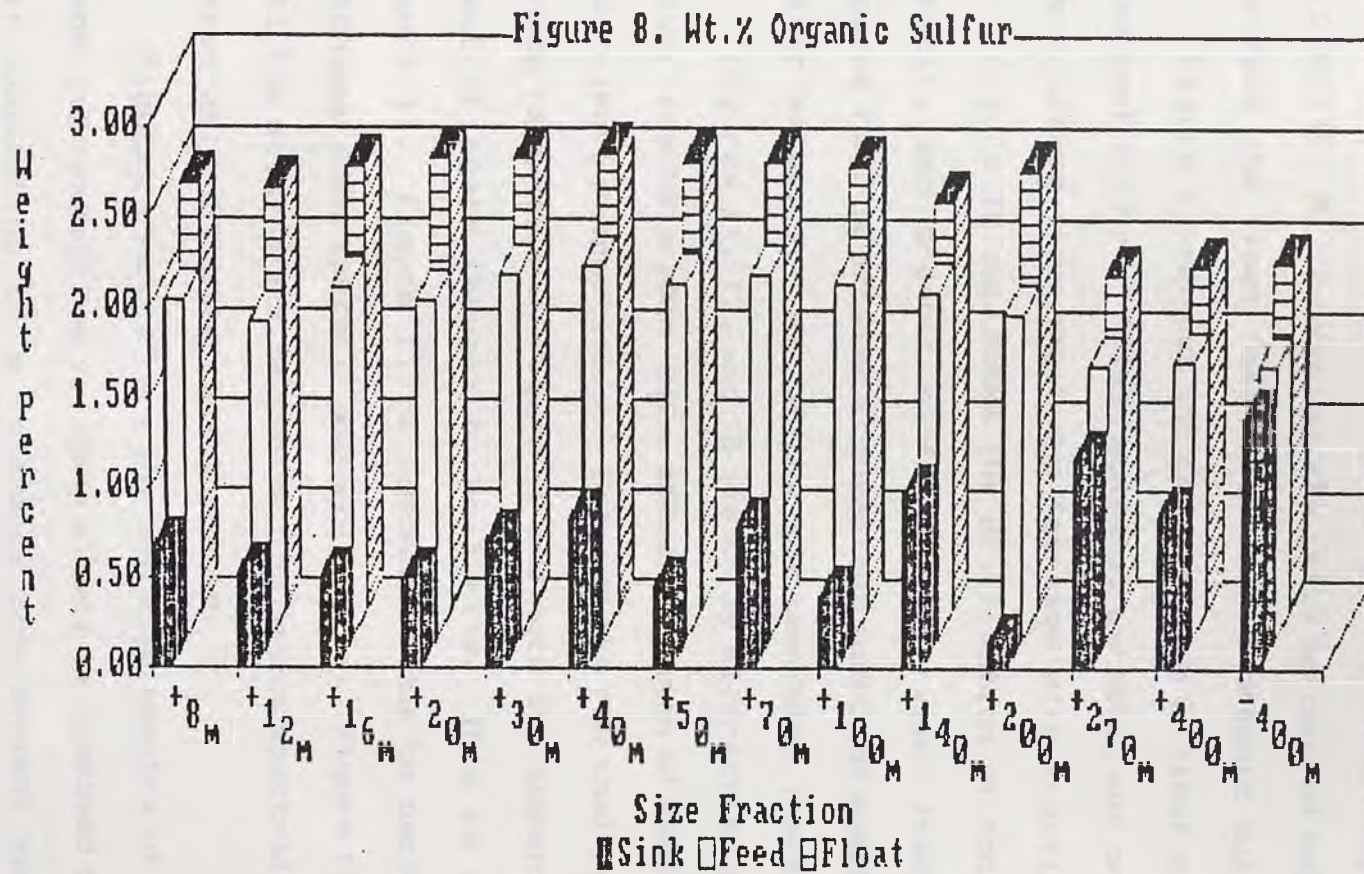


Figure 8. Comparison of calculated values for wt. % organic sulfur in feed, float, and sink fractions.

organic sulfur is found in the float (ranging from 1.871 to 2.540 wt. %) and a minimum in the sink (ranging from 0.153 to 1.411 wt. %). The organic sulfur in feed coal fractions ranges from 1.534 to 2.072 wt. %. Approximately fifty percent of total sulfur in the feed and float coal fractions is organic sulfur.

Figure 9 compares the distribution of float coal sulfur to feed coal sulfur. Total, sulfate, pyritic, and organic sulfur are compared throughout the fourteen size fractions.

Figure 10 expresses the distribution of total, sulfate, pyritic, and organic sulfur in the reject fractions. Both Figures 9 and 10 provide a convenient source of easily determined sulfur values for any of the fourteen size fractions.

Figures 11, 12, and 13 are X-ray diffraction patterns of a pulverized 70M x 100M (212 x 150 μ m) fraction of feed coal, float, and reject, respectively. Compare the raw coal pattern to the pyrite reference in Figure 11 and note the absence of several spectral peaks thought to be pyrite. This is difficult in Figure 11. Figure 12 is impossible due to background noise which masks the spectral peaks of pyrite. Figure 13 illustrates well the elimination of pyrite influenced spectral peaks in the nitric acid digested reject sample.

Figures 14 and 15 are the infrared spectra of Freon-11 and Freon-113 respectively. Each figure is composed of a spectral pair consisting of a reference and solvent spectrum. The reference spectrum is the as received Freon, and the solvent spectrum is of Freon having had intimate association with the

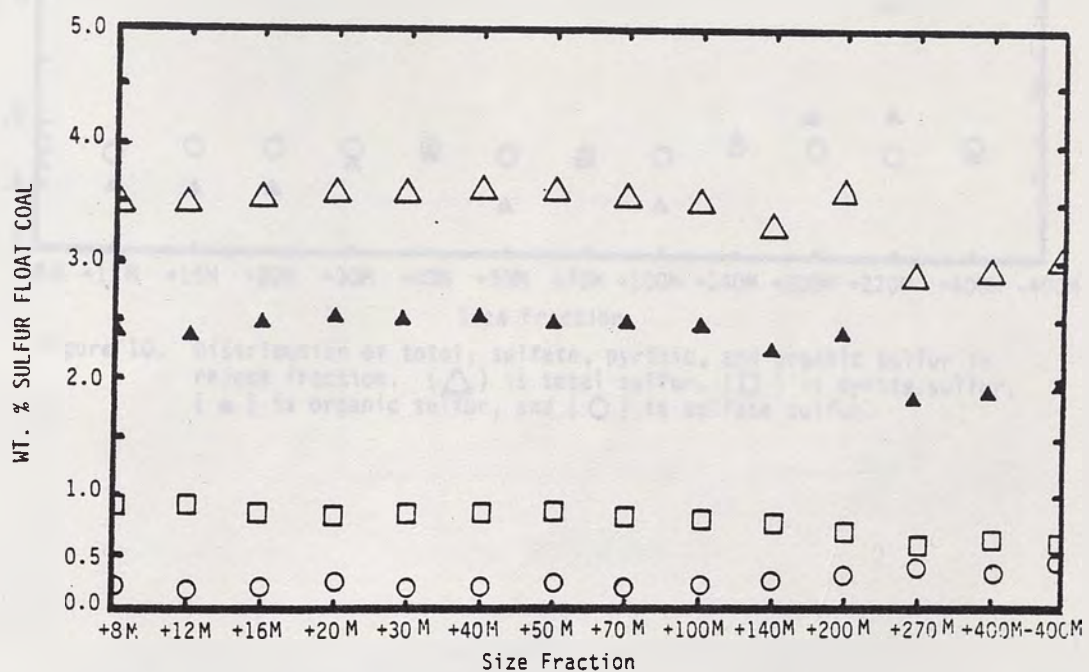
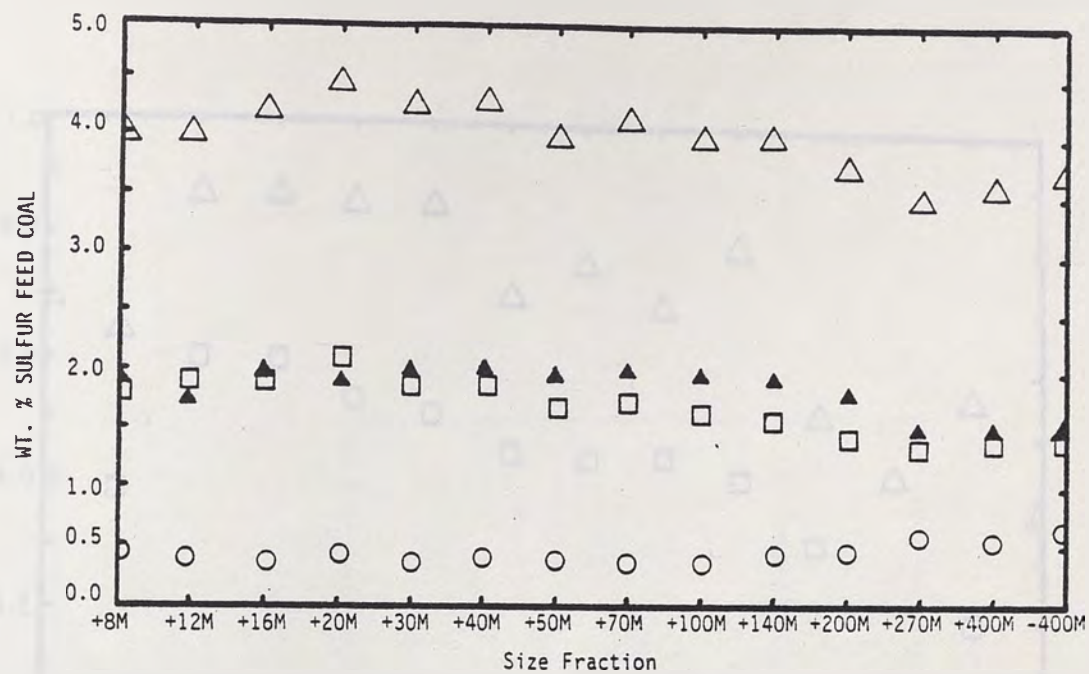


Figure 9. Comparison of total, sulfate, pyritic, and organic sulfur in feed and float coal fractions. (△) is total sulfur, (□) is pyrite sulfur, (▲) is organic sulfur, and (○) is sulfate sulfur.

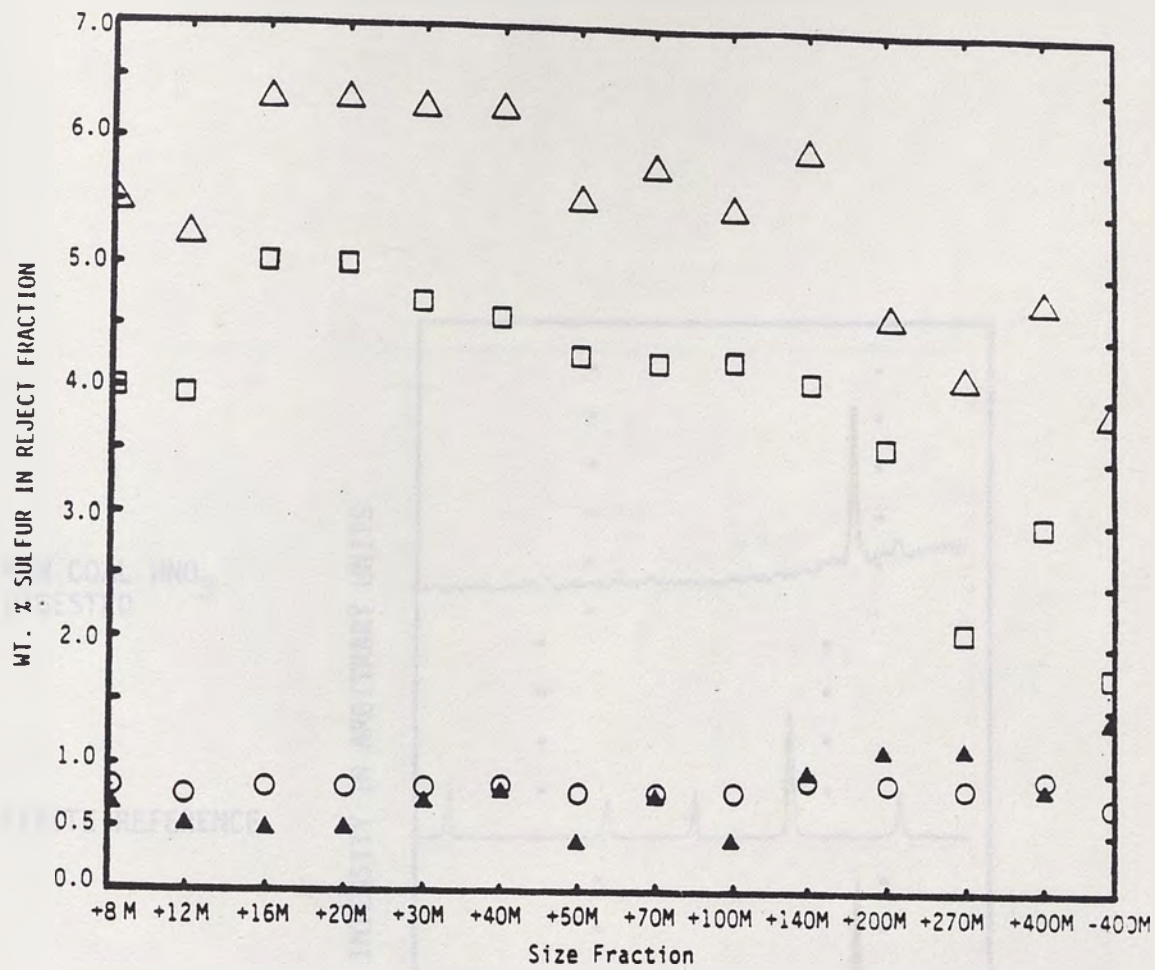


Figure 10. Distribution of total, sulfate, pyritic, and organic sulfur in reject fraction. (Δ) is total sulfur, (\square) is pyrite sulfur, (\blacktriangle) is organic sulfur, and (\circ) is sulfate sulfur.

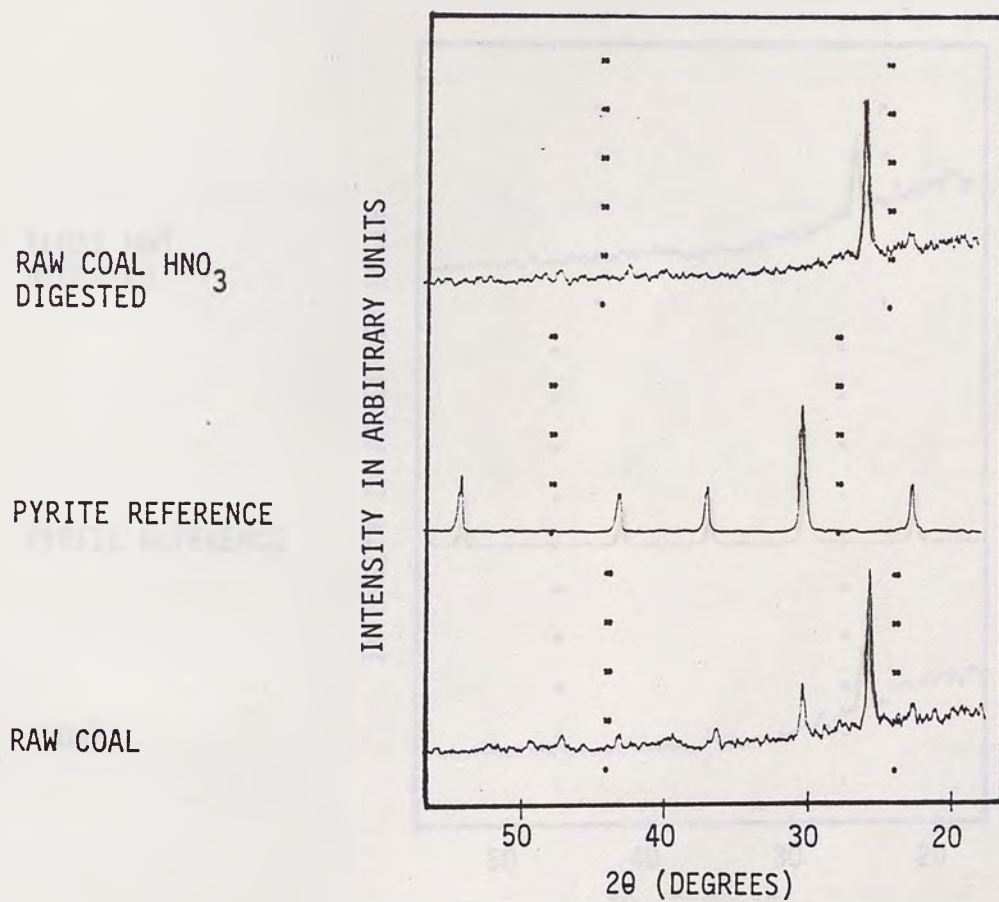


Figure 11. X-ray diffraction patterns of 70M x 100M fraction feed coal and feed coal HNO₃ digested compared to a pyrite reference.

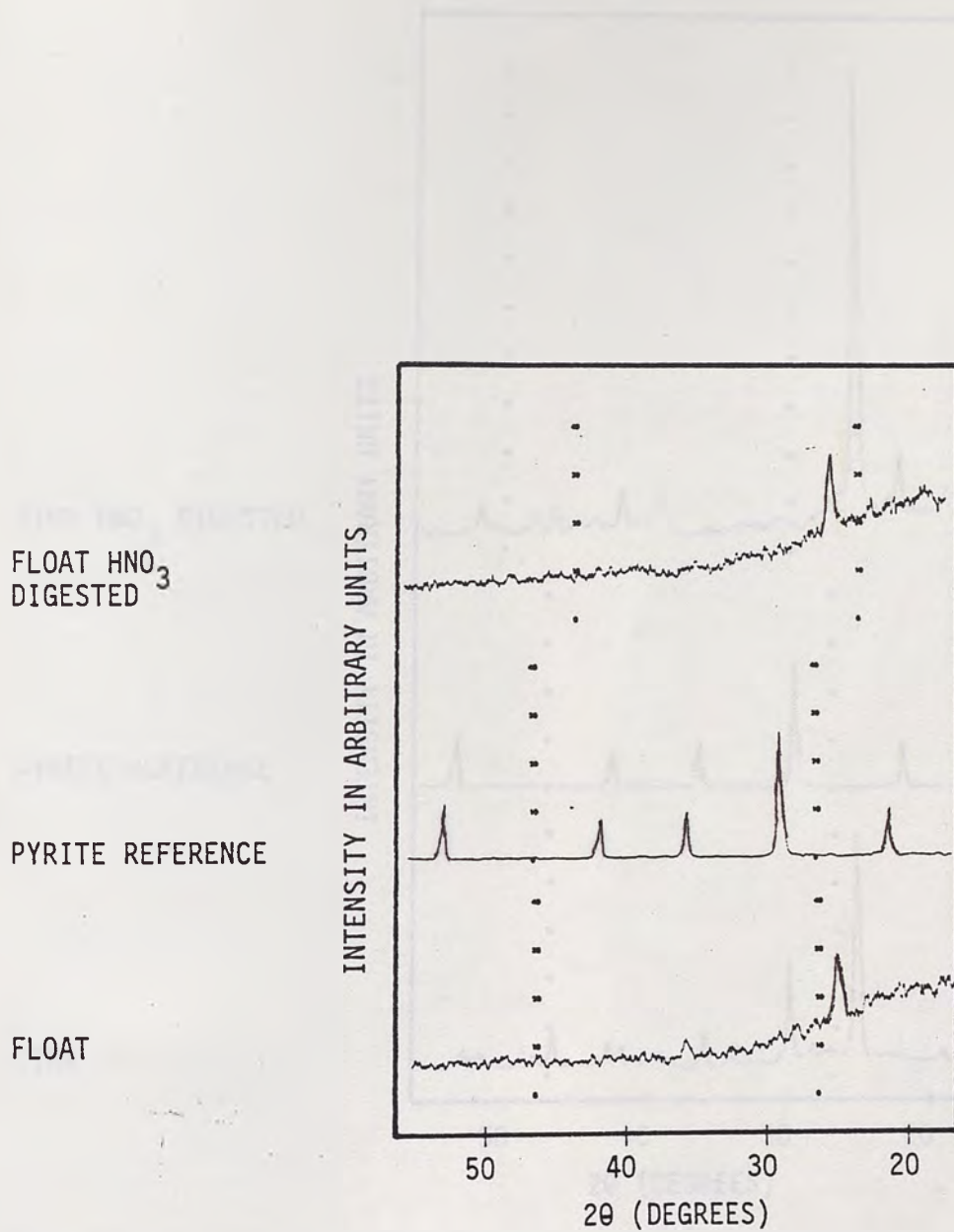


Figure 12. X-ray diffraction patterns of 70M x 100M fraction float coal and float coal HNO₃ digested compared to a pyrite reference.

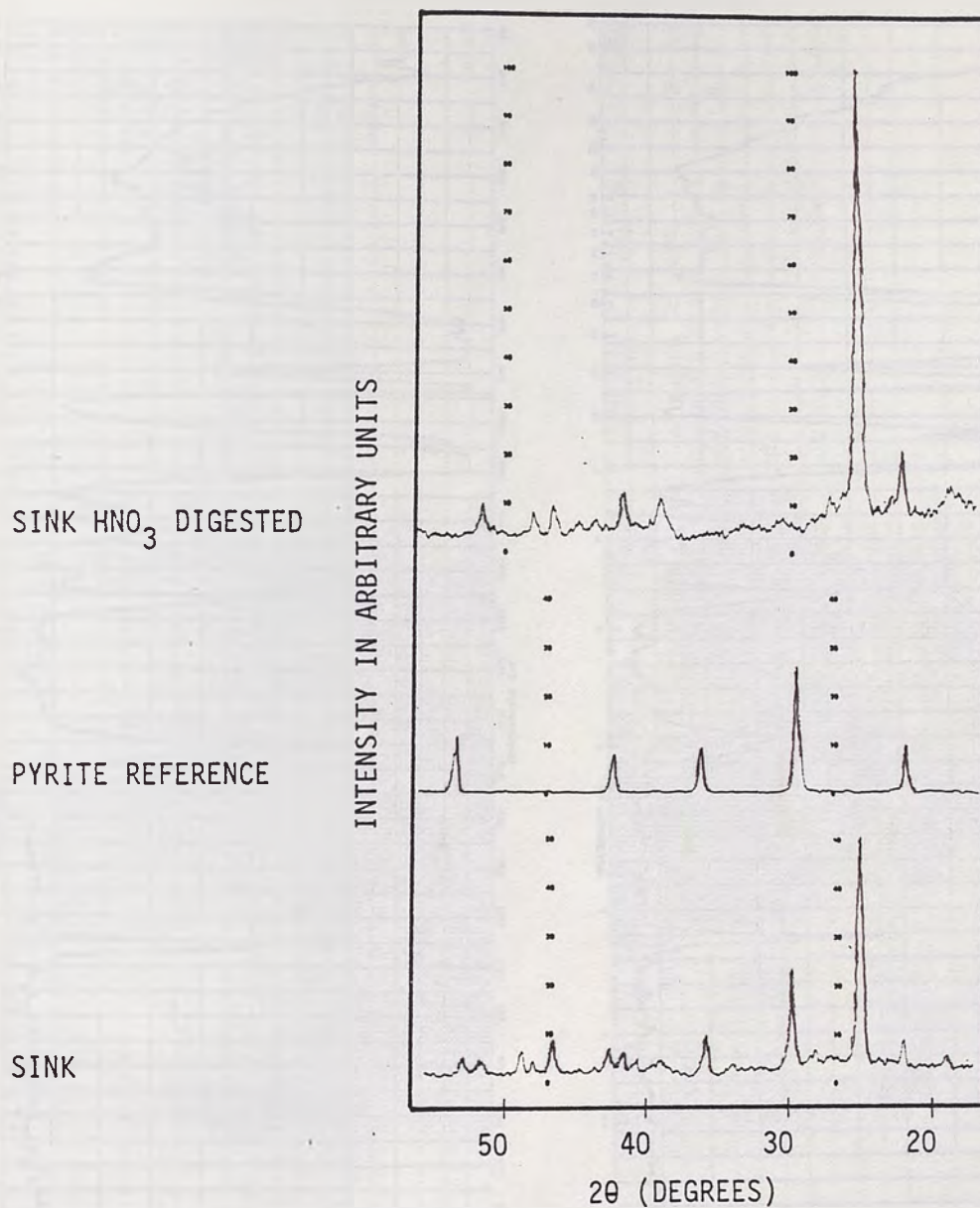


Figure 13. X-ray diffraction patterns of 70M x 100M reject fraction and reject fraction HNO₃ digested compared to a pyrite reference.

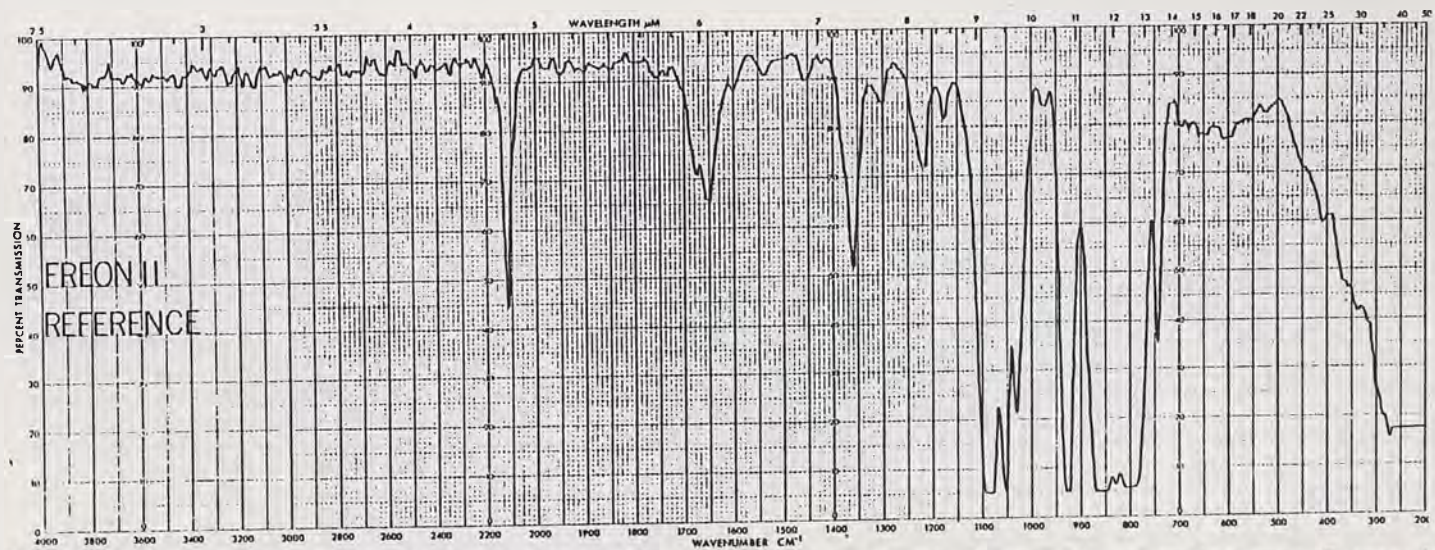
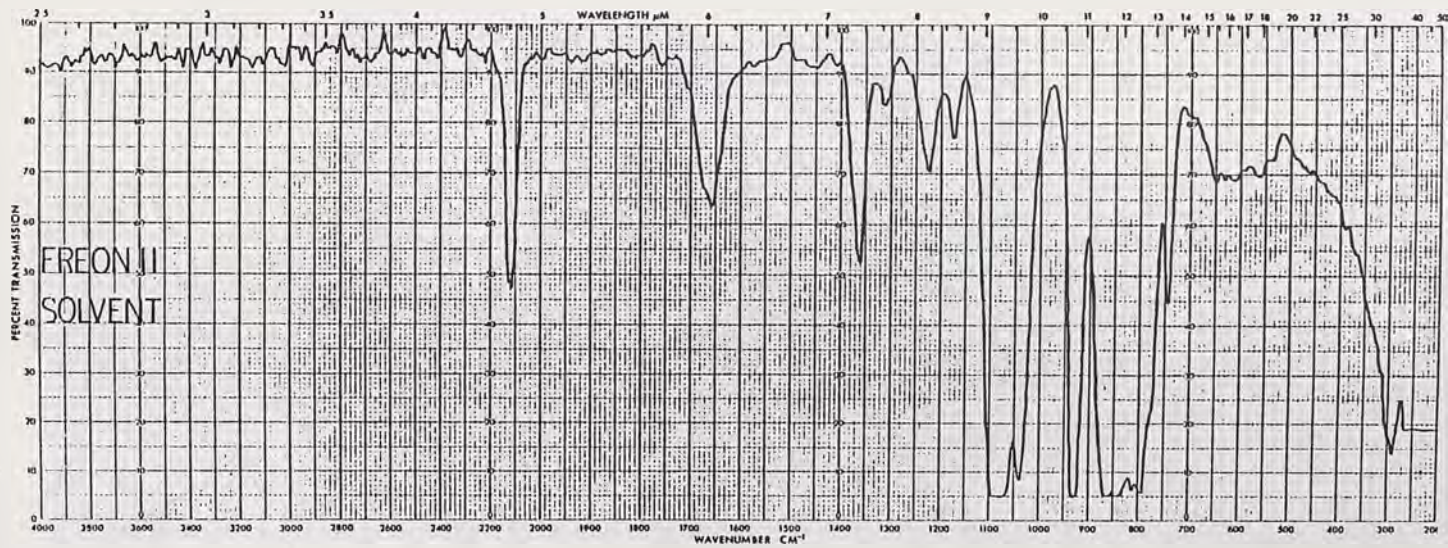


Figure 14. Infrared spectra of Freon-11 comparing solvent Freon-11 to reference Freon-11.

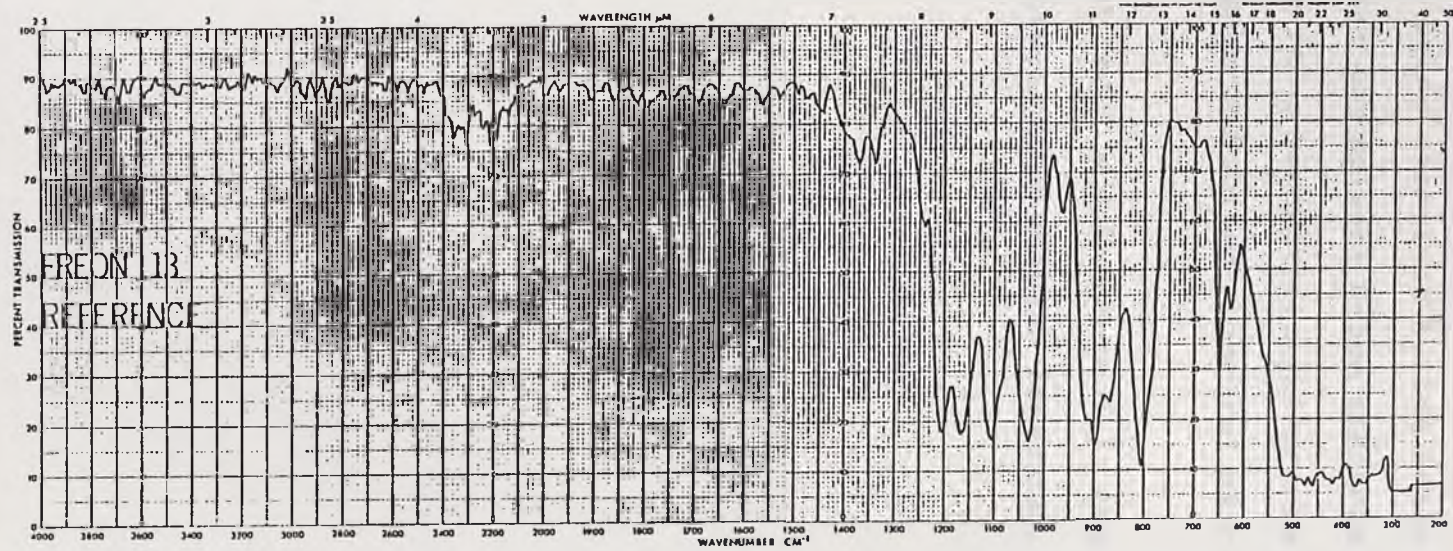
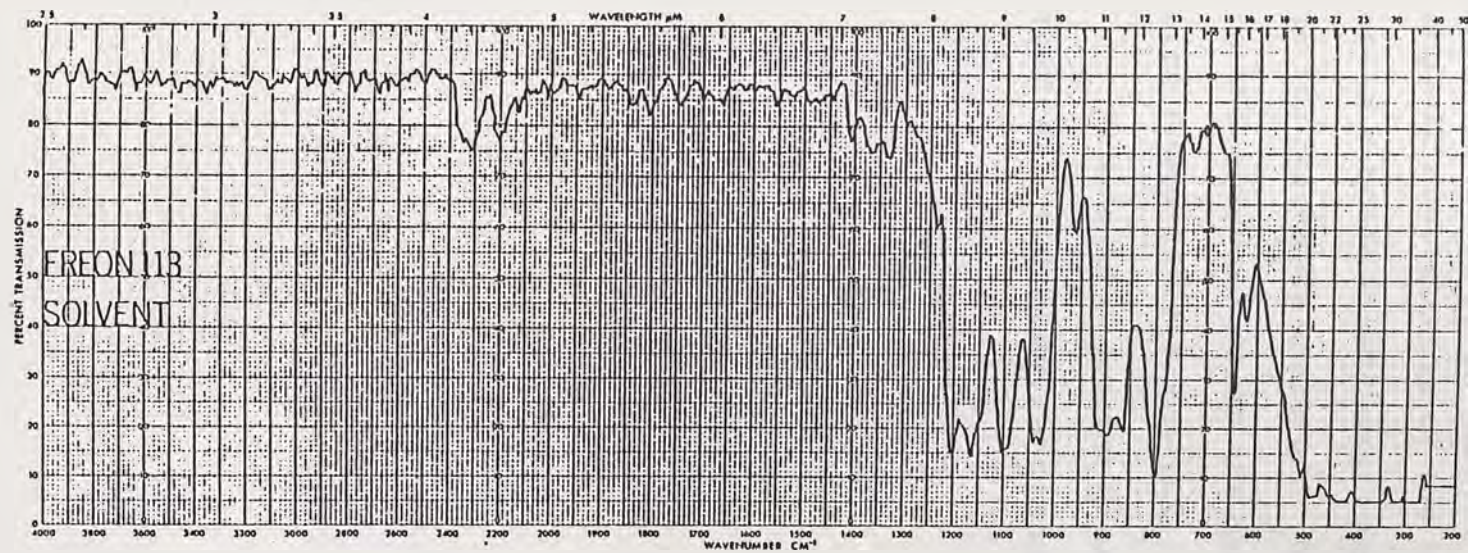


Figure 15. Infrared spectra of Freon-113 comparing solvent Freon-113 to reference Freon-113.

Illinois #6 coal. If Freon-11 or Freon-113 was acting as a solvent towards the coal, then the organic sulfur and/or other organic constituents of the coal being dissolved by the Freon would express itself when comparing the solvent Freon spectrum to its reference spectrum. There is no perceptible difference in the spectra between the reference and solvent spectral pair with Freon-11 or Freon-113.

Figure 16 shows the grams of total sulfur present in the feed coal, float coal, and reject based on 100 grams of feed coal.

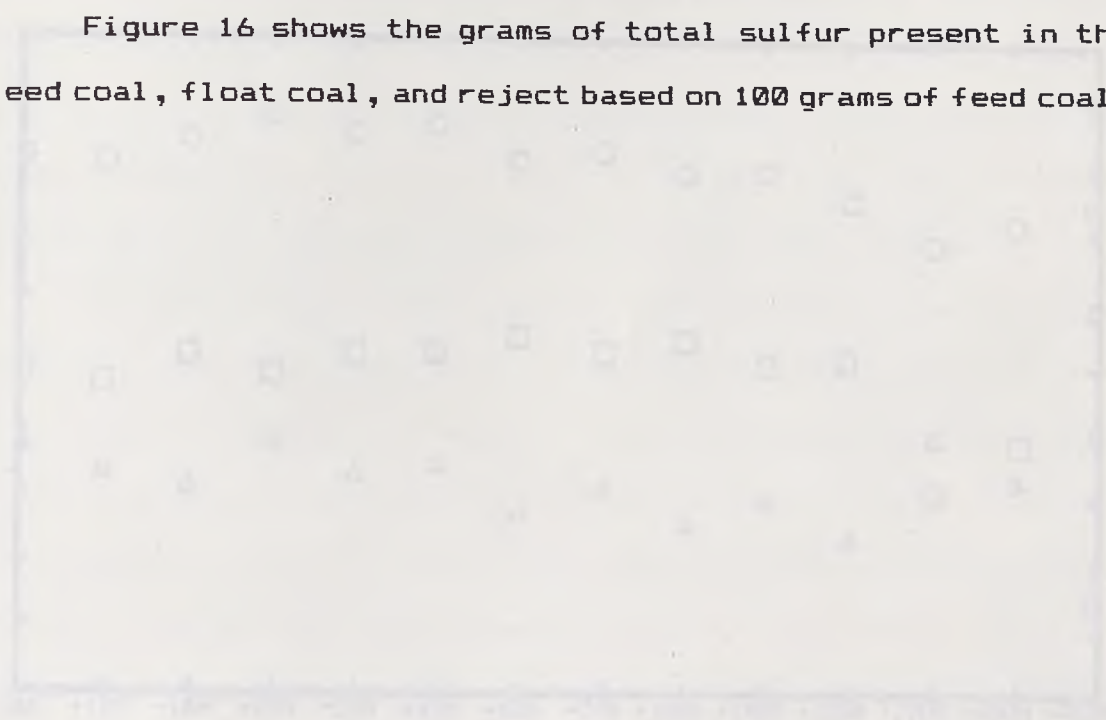


Figure 16. Grams of total sulfur per 100 grams of feed coal. (O) is feed coal, (□) is float coal, and (Δ) is reject.

DISCUSSION

The data on sulfur in the flow-sheet testing are reported by means of Tables and Figures, the figures being graphical interpretations of the results shown in the tables. In general it can be said that the figures are drawn for the purpose of illustrating the generalization of the results.

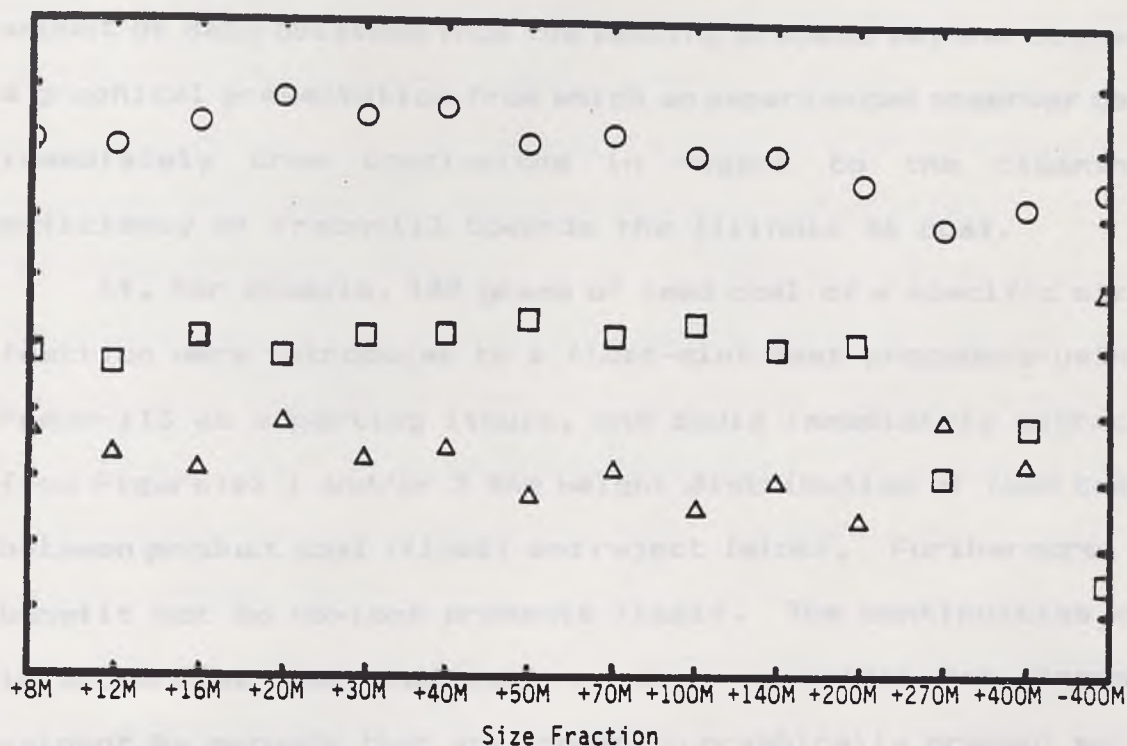


Figure 16. Grams of total sulfur per 100 grams of feed coal. (○) is feed coal, (□) is float coal, and (△) is reject.

DISCUSSION

The data developed from the float-sink testing are reported by means of tables and figures, the figures being graphical interpretation of the results shown by the tables (refer to Appendix II for data tables). The figures are drawn for two reasons, i.e., to facilitate the interpretation of the immense amount of data obtained from the testing procedures, and to give a graphical presentation from which an experienced observer can immediately draw conclusions in regard to the cleaning efficiency of Freon-113 towards the Illinois #6 coal.

If, for example, 100 grams of feed coal of a specific size fraction were introduced to a float-sink test procedure using Freon-113 as a parting liquid, one could immediately extract from Figure(s) 1 and/or 3 the weight distribution of feed coal between product coal (float) and reject (sink). Furthermore, a benefit not so obvious presents itself. The continuities or irregularities that sometimes exist in numerical data become evident by methods that are chosen to graphically present such experimental results.

Taking Figure 1 as an example, one can immediately discover a continuity in the distribution of feed coal between float and sink from +8M to +200M (in coal preparation nomenclature +8M means retained by an 8 mesh testing sieve and -8M means passing an 8 mesh testing sieve). A discontinuity manifests itself at the +270M (53 μ m) fraction through -400M (38 μ m). This irregularity

is believed to be the cut-off point (-270M) of static separation. The separation of feed coal into float and sink fractions will not be achieved with the same efficiency as previous size fractions illustrate when using Freon-113 as the parting liquid.

The significance of Figure 2 is that if ash and/or sulfur was cumulatively increasing in any size fraction, the bars depicting the ash and sulfur would increase in the same manner as weight percent. It is that obvious ash and sulfur are not expressing cumulative behavior.

The behavior of ash throughout the 14 size fractions is expressed by Figure 4. This ash behavior exemplifies the cut-off point of static separation using a Freon parting liquid. The weight-percent ash in the sink fraction is eighty percent and greater from the +8M (2.36mm) through +140M (106 μ m) fractions inclusive. Then a significant reduction in percent ash reporting to the sink fraction is noted with the remaining fine size fractions.

The cut-off point for static separation to occur between the float and sink with the efficiency of previously larger size fractions is the 140M x 200M (106 x 75 μ m) fraction. The float and sink fractions just could not be realized without the use of a centrifuge to hasten separation of the coal slurry with the 200M x 270M (75 x 53 μ m), 270M x 400M (53 x 38 μ m), and 400M x 0 (38 μ m x 0) size fractions.

However, as stated previously, most utilities crush coal to <200 mesh (75 μ m) prior to combustion. Thus, the ash appearing

in the sink fraction decreases, while the feed coal and float coal (product) shows an increase in ash; this behavior is not favorable to a coal cleaning procedure for these very fine size fractions. Consequently, while there is a potential for ash removal, it has not been realized in these fine size fractions.

As indicated previously, total sulfur consists of sulfate sulfur, pyritic sulfur, and organic sulfur. Total sulfur, sulfate sulfur, pyritic sulfur, and organic sulfur are expressed graphically in Figures 5 through 8 respectively. The weight-percent values reported for total sulfur, sulfate sulfur, and pyritic sulfur have the following distribution. As expected, the sink fractions have the highest weight percent values in sulfur, ranging from less than 1 percent sulfate sulfur, 5 percent pyritic sulfur, and 6 percent total sulfur. The float fractions express a minimum in sulfur, ranging from about 3 percent total sulfur, less than 0.4 percent sulfate sulfur, and less than 1 percent pyritic sulfur. The feed coal fractions report sulfur values somewhere between those of the sink and float sulfur, ranging from less than 4.5 percent for total sulfur, less than 0.7 percent for sulfate sulfur, and less than 2.5 percent for pyritic sulfur.

The obvious reduction in inorganic sulfur does not have any great significance since the organic sulfur remaining is intimately associated with the float fraction as can be seen in Figure 9. The organic sulfur accounts for the majority of the sulfur remaining in the float fractions. The organic sulfur

ranges from 1.9 to 2.5 %.

Figures 8, 9, and 10 show explicitly that the float fractions contain the majority of organic sulfur. Since this accounts for approximately fifty percent of the total sulfur, any sulfur reduction for the Illinois #6 coal must include the organic sulfur.

The efficiency of separation, E_s , is tabulated in Appendix II. The efficiency of separation was calculated for ash, total, sulfate, pyritic, and organic sulfur. It should be noted that the trend in efficiency values minimize in the finest size fractions. In fact, the $400\text{M} \times 0$ ($38\mu\text{m} \times 0$) fraction has the lowest efficiency value calculated for any of the various impurities, i.e., ash or sulfur etc.. As indicated before, static separation of the feed coal to float and sink fractions was impossible in the very fine size fractions ($\leq 200\text{M} \times 270\text{M}$ ($75 \times 53\mu\text{m}$)).

The tabulation below compares performance data for ash from tests in several coal preparation plants using Hancock's efficiency expression (Peng, et al., 1979):

Cleaning unit	Feed size	Coal seam	Specific gravity	Feed coal ash	E_s (%)
Sand cone	4" x 1/4"	Freeport	1.54	31.6	66.4
D. M. V.*	5" x 1/4"	Sewell	1.35	30.2	56.6
Conc. tbl.	3M x 200M	Jonesville	1.97	31.1	46.6
Jig	4" x 200M	Kochler	1.47	30.0	45.5
Hydrocyclone	8M x 200M	Illinois #6	1.68	23.8	34.4
D. M.* cyclone	5/8" x 30M	Illinois #6	1.42	14.1	64.5
Sep. funnel	6M x 8M	Illinois #6	1.56	28.5	70.9
Sep. funnel	100M x 140M	Illinois #6	1.56	27.1	57.8
Centrifuge tube	400M x 0	Illinois #6	1.56	40.4	19.2

* = Dense-medium vessel, and dense-medium cyclone

As would be expected, the values of E_s for ash are generally in the sixties but become increasingly lower as particle size decreases. In comparison to the above performance data, our E_s results for ash are generally superior (see Table 10, Appendix II) to other methods employed.

Very low values were obtained for E_s of total sulfur. This can be expected since essentially all organic sulfur and microscopic pyrite was retained in the float fraction. Sulfate sulfur averaged in the low thirties and high twenties while pyritic sulfur averaged generally in the high thirties for E_s values. The moderately low E_s values for pyritic sulfur may be due to the distribution of highly dissipated microscopic pyrite within the coal matrix.

Initially X-ray diffraction was used to determine if

microscopic pyrite was digested by nitric acid. However, the results were deemed inadequate (see Figures 11, 12, and 13) since the pyrite appeared to be digested but further analysis by electron microscopy revealed that extremely fine pyritic sulfur on the order of 1 to 10 micrometers was encapsulated by coal. This creates a two-fold problem.

The determination of pyritic sulfur in coal by analytical methods is dependent on dissolution of the pyrite by nitric acid digestion. Testing the coal residue after nitric acid digestion through electron probe microanalysis (hand pulverizing to <<< 400 mesh (38 μ m) was required) revealed substantial pyrite.

The immediate consequence of this fact lead to a material balance on the pyritic sulfur with acceptable error parameters of less than four percent. This will only occur if the feed or float coal is first pulverized to <<< 400 mesh (38 μ m) prior to an analytical procedure requiring nitric acid digestion. Testing procedures proposed in the technical literature of the last fifty years indicate passing 60 mesh (250 μ m) for the coal sample is adequate for these analytical procedures involving a nitric acid digestion. These wet chemical procedures simply will not yield accurate pyritic sulfur determinations if microscopic pyrite is present substantially as in the Illinois #6 coal.

Furthermore, the microscopic pyrite would be extremely difficult to remove from the coal. In order to remove the microscopic pyrite from the coal, the coal particle size would

have to be on the order of the microscopic pyrite before physical separation could occur between the liberated pyrite and coal. This cannot be economically justified through current comminution technology.

The results of the investigation, the following conclusions may be drawn:

1. Pyrite was found to be associated with microscopic pyrite particles. The size of the pyrite particles in the coal samples varies with the microscopic pyrite on the order of 1 to 10 micrometers in diameter.

2. Liberation of the microscopic and macroscopic pyrite particles before liberation of the coal particles can be achieved only if the particle size of the coal is on the order of the microscopic pyrite.

3. The size of the particles of pyrite in the coal varies with the size of the coal particles. The liberation of pyrite from the coal is dependent on the size of the coal particles. The liberation of pyrite from the coal is dependent on the size of the coal particles. The liberation of pyrite from the coal is dependent on the size of the coal particles.

SUMMARY and CONCLUSIONS

The purpose of any cleaning or preparation of coal is to improve the quality by lowering the ash and sulfur content. The quality of the Illinois #6 coal was improved by reduction of ash and total sulfur. The reduction of total sulfur consisted of reducing inorganic sulfur, i.e., sulfate sulfur and pyritic sulfur. There was no reduction in organic sulfur. From the results of the investigation, the following conclusions may be made:

1. Pyritic sulfur is found as macroscopic and microscopic pyrite (framboids). Much of the pyrite is present in the microscopic range, and the microscopic pyrite is on the order of 1 to 10 micrometers in diameter.

2. Liberation of the macroscopic and microscopic pyrite must occur before substantial reduction in pyritic sulfur can be achieved, i.e., the size composition of the coal must be on order of the microscopic pyrite.

3. The ash shows a tendency to concentrate in the finest size fractions. Total sulfur and pyritic sulfur do not show a tendency to concentrate in any size fraction. The feed coal is becoming progressively dirtier in sulfate sulfur with a decrease in particle size. Organic sulfur is concentrated in the float

fraction.

4. If microscopic pyrite is present substantially as in the Illinois #6 coal, current testing procedures are inadequate for the analytical procedures involving nitric acid digestion of the pyrite. Standard leaching procedures clearly failed to dissolve most of the microscopic pyrite. These procedures stipulate that the coal be pulverized to pass 60 mesh prior to chemical digestion of pyrite with nitric acid. This results in the incomplete dissolution of the pyrite which is encapsulated in the hydrophobic organic matrix of the coal. A very important necessary improvement is to pulverize the coal sample to much less than 400 mesh prior to analytical procedures involving pyrite and nitric acid. If this improvement is not realized significant errors will exist in the pyritic sulfur determinations and any attempts at a pyrite material balance will be useless.

5. No organic constituents of the coal were dissolved by Freon-11 or Freon-113.

RECOMMENDATIONS FOR FUTURE RESEARCH

A method of chemical comminution for coal using a number of chemical compounds including various alcohols, amines, acetone, galcial acetic acid and solutions of hydroxides should be investigated. Ammonia appears to be promising for coal. Moreover, when Illinois #6 coal is soaked in liquid anhydrous ammonia at atmospheric pressure, fragmentation occurs. However, if the bonds between the organic and inorganic components of coal could be selectively broken, much of the ash forming mineral matter including pyrite could be liberated without expensive physical size reduction. Then microscopic inorganic sulfur removal could be addressed.

REFERENCES

- Annual Book of ASTM Standards, Part 19, ASTM D271, Sulfur by the Eschka Method, 1973, pp. 15-18; ASTM D2492, Standard Method of Test for Forms of Sulfur in Coal, 1973, pp. 380-384.
- Chapman, W.R. and Jones, R.D.C., 1954. The Removal of Sulfur From Coal. In: F.G. Miller, 1964, Reduction of Sulfur in Minus 23 Mesh Bituminous Coal, Trans. SME/AIME, 229:7.
- Edward, A.N., Jones, J.M. and Newcombe, W., 1964. The Extraction by Nitric Acid of Pyrites From Vitraains and Coal Samples. In: Fuel, 1964, 43:55-62.
- Greer, R.T., 1979. Organic and Inorganic Sulfur in Coal. In: Scanning Electron Microscopy, 1:477-486.
- Hancock, R.T., 1979. Calculating Efficiencies From Washery Figures, Colliery Engineering, pp. 140-142, 156, 191-193, 237-239.
- Humphreys, K.K., Quinlan, R.M. and Venkatesan, S., 1979. The Economics of Coal Preparation. In: J.W. Leonard (Editor), 1979, Coal Preparation. Port City Press, New York, N.Y., pp. 5-3, 5-4.
- Keller, D.V., Jr., Smith, C.D. and Bunch, E.F., 1977. Demonstration Plant Test Results of the Otisca Process Heavy Liquid Benefication of Coal. SME/AIME Preprint 77-F-82, Atlanta, GA., 16 pp.
- Leonard, J.W. (Editor), 1979. Coal Preparation. Port City Press, Inc., Baltimore, Ma., pp. 1-3, 5-3.
- McCartney, J.T., O'Donnell, H.J. and Ergun, S., 1969. Pyrite Size Distribution and Coal Pyrite Particles Association in Steam Coals. U.S. Bureau of Mines R.I. 7231, 18 pp.
- McClung, J.D. and Geer, M.R., 1979. Properties of Coal and Coal Impurities. In: J.W. Leonard (Editor), Coal Preparation, Port City Press, New York, N.Y., pp. 1-3, 1-53, 1-69, 1-71.
- Miller, F.G., 1964. Reduction of Sulfur in Minus 28 Mesh Bituminous Coal. Trans. SME/AIME, 229:7-15.
- Morrison, G.F., 1981. Chemical Desulphurisation of Coal. I.E.A. Coal Research, London, 11 pp.

- Nelson, J.B., 1953. The Assessment of the Mineral Species Associated With Coal. In: J.W. Leonard (Editor), 1979, Coal Preparation. Port City Press, New York, N.Y., pp. 1-56, 1-57.
- Office of the Director of Coal Research, 1967. Methods of Analyzing and Testing Coal and Coke. U.S. Bureau of Mines R.I. 638, 12 pp.
- Peng, F.G. and others, 1979. Evaluation and Prediction of Optimum Cleaning Results. In: J.W. Leonard (Editor), 1979, Coal Preparation. Port City Press, New York, N.Y., pp. 18-48, 18-49.
- Roberts, E.J. and others, 1970. Solids Concentration. Chemical Engineering, pp. 52-68.
- Schulz, N.F., 1970. Separation Efficiency. Trans. AIME, 247:81-87.
- Taggart, A.F., 1945, Handbook of Mineral Dressing. John Wiley, New York, N.Y., pp. 19.150-19.154.
- Thiessen, R., 1920. Occurrence and Origin of Finely Disseminated Sulfur Compounds in Coal. Trans. AIME, 63:912-926.
- Wright, A.C. (Editor), 1985. Collecting Fines for New Markets. In: Coal Age, pp. 57-64.

APPENDIX I

TABLE 1. Minerals Associated with Bituminous Coal

GROUP (FORMULA)	SPECIES	SPECIFIC GRAVITY	
SHALE (K, Na, H ₃ O, Ca) ₂ (Al, Mg, Fe, Ti) ₄ (Al, Si) ₈ O ₂₀ (OH, F) ₄	Muscovite	2.76-2.88	
	Hydromuscovite	
	Illite	
	Bravaisite	4.66	
KAOLIN Al ₂ (Si ₂ O ₅)(OH) ₄	Montmorillonite	2.50	
	Kaolinite	2.60	
	Livesite	
	Metahalloysite	
SULFIDE FeS ₂	Pyrite	5.02	
	Marcasite	4.89	
CARBONATE (Ca, Mg, Fe, Mn)CO ₃	Ankerite	2.95-3.00	
	Ankeritic calcite	2.71	
	Ankeritic dolomite	2.85	
	Ankeritic chalybite	3.96	
CHLORIDE (K, Na)Cl	Sylvine	1.99	
	Halite	2.16	
ACCESSORY MINERALS SiO ₂	Quartz	2.65	
	Feldspar	2.55-2.76	
	(K, Na) ₂ O·Al ₂ O ₃ ·6SiO ₂	Garnet	3.50-4.30
	3CaO·Al ₂ O ₃ ·3SiO ₂	Hornblende	3.00-3.40
	CaO·3FeO·4SiO ₂	Gypsum	2.32
CaSO ₄ ·2H ₂ O			

Table 1 (continued). Minerals Associated with Bituminous Coal

GROUP	SPECIES	SPECIFIC GRAVITY
ACCESSORY MINERALS	Apatite	3.15-3.20
$9\text{CaO} \cdot 3\text{P}_2\text{O}_5 \cdot \text{CaF}_2$	Zircon	4.68
ZrSiO_4	Epidote	3.35-3.45
$4\text{CaO} \cdot 3\text{Al}_2\text{O}_3 \cdot 6\text{SiO}_2 \cdot \text{H}_2\text{O}$	Biotite	2.80-3.20
$\text{K}_2\text{O} \cdot \text{MgO} \cdot \text{Al}_2\text{O}_3 \cdot 3\text{SiO}_2 \cdot \text{H}_2\text{O}$	Augite	3.20-3.40
$\text{CaO} \cdot \text{MgO} \cdot \text{SiO}_2$	Prochlorite	2.60-3.30
$2\text{FeO} \cdot 2\text{MgO} \cdot \text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$	Diaspore	3.35-3.45
$\text{Al}_2\text{O}_3 \cdot \text{H}_2\text{O}$	Lepidocrocite	4.09
$\text{Fe}_2\text{O}_3 \cdot \text{H}_2\text{O}$	Magnetite	3.00-3.20
Fe_3O_4	Kyanite	3.55-3.60
$\text{Al}_2\text{O}_3 \cdot \text{SiO}_2$	Staurolite	3.65-3.75
$2\text{FeO} \cdot 5\text{Al}_2\text{O}_3 \cdot 4\text{SiO}_2 \cdot \text{H}_2\text{O}$	Topaz	3.40-3.60
$(\text{AlF})_2\text{SiO}_4$	Tourmaline	3.00-3.25
$\text{H}_9\text{Al}_3(\text{BOH})_2\text{Si}_4\text{O}_{19}$	Hematite	5.26
Fe_2O_3	Penninite	2.60-3.30
$5\text{MgO} \cdot \text{Al}_2\text{O}_3 \cdot 3\text{SiO}_2 \cdot 2\text{H}_2\text{O}$		

TABLE 2. Partial Summary of Heavy Liquid Separators
(Keller, 1980)

COMPOUND	BOILING POINT °C	SPECIFIC GRAVITY	TOXICITY TLV
Dichloromethane	40	1.325	toxic
1,1,1-trichloroethane	75	1.338	irritant
1,1,2-trichloroethane	112	1.435	mutagenic
Trichloroethylene	87	1.476	mutagenic
Trichlorofluoromethane (Freon-11)	24	1.500	1000
1,2-dibromotrichloro ethane	195	1.560	toxic
1,1,2-trichlorotrifluoro ethane (Freon-113)	46	1.564	1000
1,1,1-trichlorotrifluoro ethane	46	1.579	1000
1,1,2,2-tetrachloro ethane	147	1.586	severe
Carbon tetrachloride	76	1.594	toxic
1,1,1,2-tetrachloro ethane	138	1.598	severe
Tetrachloroethylene	121	1.623	mutagenic
1,2-difluorotetrachloro ethane (Freon-112)	121	1.634	1000
Pentachloroethane	161	1.680	severe
Bromochloromethane	68	1.991	corrosive
1,2-dibromotetrafluoro ethane (Freon-114B2)	48	2.175	1000
1,2-dibromoethane	131	2.180	mutagenic
1,2-dibromo-2-chloro fluoroethane	93	2.230	1000
Dibromomethane	97	2.477	1000
Chlorodibromomethane	119	2.451	irritant
Magnetite/H ₂ O	100	variable	non
Zinc chloride/H ₂ O	100	variable	non

APPENDIX II

DATA TABLES

TABLE 3.

AS RECEIVED CRUSHED TO 6M x 0

SCREEN ANALYSIS

1	2	3	4	5	6	7	8	9	10	11
SIZE FRACTION					CUMULATIVE RESULTS					
Pass- ing	Retained on	% Wt.	DRY BASIS % Ash	% Sul.	Retained on Screen			Passing Screen		
					% Wt.	% Ash	% Sul.	% Wt.	% Ash	% Sul.
6M x 8M		22.91	31.10	3.85	22.91	31.10	3.85	100.00	26.94	3.98
8M x 12M		10.37	26.65	3.93	33.28	29.71	3.88	77.09	25.71	4.02
12M x 16M		8.84	23.95	4.35	42.12	28.50	3.97	66.72	25.40	4.03
16M x 20M		8.39	23.45	4.21	50.51	27.66	4.01	57.88	25.62	3.98
20M x 30M		6.78	18.65	3.91	57.28	26.60	4.00	49.49	25.99	3.94
30M x 40M		6.96	21.75	4.29	64.25	26.07	4.03	42.71	27.16	3.94
40M x 50M		5.77	22.00	3.85	70.01	25.74	4.02	35.75	28.21	3.88
50M x 70M		5.32	23.35	4.20	75.33	25.57	4.03	29.98	29.40	1.63
70M x 100M		4.94	24.40	3.99	80.27	25.50	4.03	24.66	30.71	3.81
100M x 140M		5.80	26.75	4.01	86.07	25.58	4.03	19.72	32.29	3.77
140M x 200M		1.98	26.55	4.16	88.06	25.60	4.03	13.92	34.60	3.67
200M x 270M		7.53	34.55	3.59	95.58	26.31	4.00	11.94	35.93	3.59
270M x 400M		1.09	32.10	3.67	96.67	26.37	3.99	4.41	38.29	3.58
400M x 0		3.33	40.30	3.55	100.00	26.84	3.98	3.33	40.30	3.55

Table 4.
FLOAT AND SINK ANALYSIS

SIZE FRACTION	PRODUCT COAL Wt. %	REJECT Wt. %
6M x 8M	71.13	28.87
8M x 12M	68.01	31.99
12M x 16M	75.20	24.80
16M x 20M	69.00	31.00
20M x 30M	73.70	26.30
30M x 40M	72.39	27.61
40M x 50M	75.69	24.31
50M x 70M	72.93	27.07
70M x 100M	76.29	23.71
100M x 140M	74.70	25.30
140M x 200M	73.72	26.28
200M x 270M	52.44	47.56
270M x 400M	65.86	34.14
400M x 0	23.63	76.37

Table 5.
ASH ANALYSIS

SIZE FRACTION	Wt. % ASH			% ASH REPORTING	
	*FEED	FLOAT	SINK	TO FLOAT	TO SINK
6M x 8M	28.50	8.55	77.65	21.34	78.66
8M x 12M	30.91	8.95	77.60	19.69	80.31
12M x 16M	24.31	8.30	72.80	25.68	74.32
16M x 20M	29.53	8.55	76.20	19.98	80.02
20M x 30M	26.22	8.50	75.85	28.90	76.10
30M x 40M	24.29	8.15	75.75	24.29	75.71
40M x 50M	24.40	7.95	75.60	24.66	75.34
50M x 70M	26.39	8.55	74.45	23.62	76.38
70M x 100M	23.80	8.20	74.00	26.29	73.71
100M x 140M	27.10	11.70	72.55	32.30	67.70
140M x 200M	27.40	16.00	59.40	43.04	56.96
200M x 270M	34.67	20.00	50.85	30.24	69.76
270M x 400M	32.12	17.75	59.85	36.40	63.60
400M x 0	40.44	21.15	46.40	12.36	87.64

*Calculated

Table 6.
TOTAL SULFUR ANALYSIS

SIZE FRACTION	Wt. % *FEED	% TOTAL SULFUR		% TOTAL SULFUR REPORTING	
		FLOAT	SINK	TO FLOAT	TO SINK
6M x 8M	4.06	3.47	5.51	60.81	39.19
8M x 12M	4.04	3.47	5.24	58.47	41.53
12M x 16M	4.23	3.53	6.37	62.70	37.30
16M x 20M	4.44	3.58	6.35	55.65	44.35
20M x 30M	4.29	3.57	6.29	61.39	38.61
30M x 40M	4.35	3.60	6.33	59.85	40.15
40M x 50M	4.08	3.59	5.62	66.54	33.46
50M x 70M	4.18	3.55	5.86	62.00	38.00
70M x 100M	4.01	3.53	5.54	67.22	32.78
100M x 140M	4.01	3.33	6.02	62.02	37.98
140M x 200M	3.77	3.64	4.64	67.66	32.34
200M x 270M	3.50	2.90	4.15	43.51	56.49
270M x 400M	3.59	2.96	4.81	54.28	45.72
400M x 0	3.69	3.03	3.90	19.38	80.62

*Calculated

Table 7.

SULFATE SULFUR ANALYSIS

SIZE FRACTION	Wt. % SULFATE *FEED	SULFUR FLOAT	SULFUR SINK	% SULFATE TO FLOAT	SULFUR REPORTING TO SINK
6M x 8M	0.379	0.208	0.801	39.02	60.98
8M x 12M	0.374	0.194	0.758	35.24	64.76
12M x 16M	0.366	0.217	0.816	44.65	55.35
16M x 20M	0.415	0.227	0.835	37.69	62.31
20M x 30M	0.376	0.209	0.844	40.96	59.04
30M x 40M	0.390	0.210	0.862	38.97	61.03
40M x 50M	0.383	0.246	0.810	48.59	51.41
50M x 70M	0.389	0.230	0.816	43.16	56.84
70M x 100M	0.379	0.238	0.831	47.96	52.04
100M x 140M	0.433	0.279	0.910	46.88	53.12
140M x 200M	0.468	0.315	0.896	49.66	50.34
200M x 270M	0.619	0.405	0.854	34.33	65.67
270M x 400M	0.560	0.361	0.944	42.46	57.54
400M x 0	0.693	0.444	0.770	15.14	84.86

* Calculated

Table 8.
PYRITIC SULFUR ANALYSIS

SIZE FRACTION	Wt. % PYRITIC SULFUR *FEED	Wt. % PYRITIC SULFUR FLOAT	Wt. % PYRITIC SULFUR SINK	% PYRITIC SULFUR REPORTING TO FLOAT	% PYRITIC SULFUR REPORTING TO SINK
6M x 8M	1.795	0.891	4.021	35.31	64.69
8M x 12M	1.893	0.921	3.958	33.10	66.90
12M x 16M	1.895	0.857	5.043	34.01	65.99
16M x 20M	2.124	0.830	5.005	26.96	73.04
20M x 30M	1.870	0.852	4.724	33.57	66.43
30M x 40M	1.892	0.850	4.624	32.52	67.48
40M x 50M	1.703	0.856	4.339	38.05	61.95
50M x 70M	1.758	0.835	4.243	34.64	65.36
70M x 100M	1.647	0.826	4.288	38.27	61.73
100M x 140M	1.627	0.784	4.118	35.97	64.03
140M x 200M	1.469	0.713	3.591	35.78	64.22
200M x 270M	1.340	0.624	2.129	24.42	75.58
270M x 400M	1.472	0.685	2.992	30.64	69.36
400M x 0	1.468	0.655	1.719	10.54	89.46

*Calculated

Table 9.

ORGANIC SULFUR ANALYSIS

SIZE FRACTION	Wt. % ORGANIC SULFUR *FEED	% ORGANIC SULFUR FLOAT	% ORGANIC SULFUR SINK	% ORGANIC SULFUR TO FLOAT	% ORGANIC SULFUR REPORTING TO SINK
6M x 8M	1.855	2.371	0.688	89.46	10.54
8M x 12M	1.769	2.355	0.524	90.53	9.47
12M x 16M	1.974	2.465	0.511	93.58	6.42
16M x 20M	1.899	2.523	0.510	91.67	8.33
20M x 30M	2.039	2.509	0.722	90.69	9.31
30M x 40M	2.072	2.540	0.844	88.75	11.25
40M x 50M	1.998	2.489	0.471	94.27	5.73
50M x 70M	2.029	2.485	0.801	89.31	10.69
70M x 100M	1.984	2.470	0.420	94.98	5.02
100M x 140M	1.952	2.277	0.992	87.14	12.86
140M x 200M	1.833	2.432	0.153	97.81	2.19
200M x 270M	1.536	1.871	1.167	63.85	36.15
270M x 400M	1.555	1.910	0.871	80.90	19.10
400M x 0	1.534	1.931	1.411	29.74	70.26

*Calculated

Table 10.

SEPARATION EFFICIENCIES

SIZE FRACTION	ASH E_s	SULFUR			
		TOTAL E_s	SULFATE E_s	PYRITIC E_s	ORGANIC E_s
6M x 8M	70.89	8.35	32.50	36.48	-25.15
8M x 12M	68.01	9.02	35.30	35.23	-22.46
12M x 16M	65.08	14.02	32.08	41.93	-16.36
16M x 20M	64.72	12.07	31.33	42.64	-09.51
20M x 30M	56.82	7.92	31.97	40.52	-25.40
30M x 40M	63.83	12.56	33.20	40.02	-14.60
40M x 50M	64.87	6.12	24.09	38.45	-26.31
50M x 70M	64.12	11.61	26.48	38.99	-14.55
70M x 100M	66.20	9.26	30.20	39.36	-24.47
100M x 140M	57.81	13.20	28.54	38.01	-12.23
140M x 200M	40.95	7.14	25.65	38.74	-14.01
200M x 270M	34.00	8.93	18.36	26.63	-11.42
270M x 400M	43.40	12.38	23.68	35.62	-14.45
400M x 0	19.23	6.11	10.34	14.33	-01.62

APPENDIX III

Discussion of testing procedures and modifications required in prescribed procedures to obtain acceptable precision:

Ash Analysis (ASTM designation D 3174-82)

Summary of Method

Ash is the residue derived from the mineral matter during complete incineration of the coal. It differs in chemical composition and is usually less than the mineral matter originally present in the coal. During incineration various weight changes take place, such as loss of water in the constitution of silicates, loss of carbon dioxide from the carbonates, oxidation of iron pyrites to iron oxide, and fixation of oxides of sulfur by such bases as calcium and magnesium. As the conditions of incineration determine the extent to which the weight changes take place, this determination is empirical.

Ash is determined by weighing the residue remaining after burning the coal under rigidly controlled conditions of sample weight, temperature, time, and equipment specifications.

The amount of ash normally is determined by burning a 1 g sample of coal in a muffle furnace to temperatures of 700-750°C. The most important cause of variation in the amount of determined ash is the effect sulfur has on the analysis. If the amount of

pyrite and carbonate minerals in coal is low, sulfur retention is not critical and ashing may be done rapidly. On the other hand, for coals with considerable amounts of pyrite and calcite, the normal practice is to burn the coal at low temperature to decompose the pyrite before the decomposition point of the carbonate minerals is reached. Therefore, less sulfur is left in the coal to react with the bases formed at high temperatures.

The coal analysis report, which was received with this particular coal for major elements in ash (one of five reports), of the Illinois #6 bed is:

SiO ₂	50.14%
Al ₂ O ₃	16.17
Fe ₂ O ₃	14.65
TiO ₂	00.98
CaO.....	07.56
MgO.....	01.62
Na ₂ O.....	01.01
K ₂ O.....	01.34
Sulfites.....	05.58

The sulfur forms (as received) are:

Sulfate.....	00.02%
Pyritic.....	03.04
Organic.....	01.92

The combustion products existing (CaO, MgO, Fe₂O₃, etc.) in the above ash analysis indicate high carbonate and pyrite. The high carbonate and pyrite present required the following temperature regulation to obtain the acceptable ASTM precision for ash in the analysis sample [triplicate results with samples differing by only $\leq 0.3\%$]:

An eight hour incineration was required, i.e., initial four hours...no more than 500°C; the remaining four hours...no more than 750°C.

Organic sulfur in coal is usually determined simply as the difference between total sulfur and the sum of pyritic and sulfate sulfur. Much of the organic sulfur may actually be undissolved pyritic sulfur which does not appear in the pyritic sulfur determination. Standard procedures stipulate that passing 60 mesh coal (<250 μm diameter) be used in the tests.

Pyrite (many times the major source of sulfur in coal) can be distributed on a scale finer than 50 μm and is not leached out using standard analytical procedures (since the organic components of coal are less soluble in HNO₃ used in these tests, much of the pyrite remains undissolved).

Although limits of repeatability for the ASTM methods insure reasonably high precision, a question of accuracy exists concerning the complete dissolution of pyrite by extraction with

nitric acid. Nitric acid only penetrates approximately 5 μm into a coal specimen (Edwards et al., 1964).

Furthermore, the solid residue remaining after the HNO_3 digestion was examined by scanning electron microscopy. In all cases, both Fe and S were detected in significant amounts. This problem can be alleviated if an improvement is made, i.e., all samples are to be pulverized to $\lll 400$ mesh (38 μm) prior to HNO_3 digestion.

Total Sulfur Analysis (ASTM designation D 3177-75)

Summary of Method

The Eschka Method: A weighed sample of coal (pulverized with mortar and pestle to $\lll 400$ mesh (38 μm)) and Eschka mixture (2 parts of light calcined magnesium oxide (MgO) and 1 part of anhydrous sodium carbonate (Na_2CO_3)) are ignited together, and the sulfur is precipitated from the resulting solution as barium sulfate (BaSO_4). The precipitate is filtered, ashed, and weighed.

The acceptable ASTM precision for total sulfur in the analysis sample is triplicate results with samples differing by no more than 0.10% for coal containing 2% sulfur or more.

Determination of Sulfur Forms in Coal (Bulletin 638 Bureau of Mines)

Summary of Method

Sulfate sulfur is determined by extracting a weighed sample of coal (pulverized with mortar and pestle to <<< 400 mesh (38 μm)) with dilute hydrochloric acid followed by precipitation with barium chloride (BaCl_2) and weighing as barium sulfate (BaSO_4). Sulfate sulfur is soluble in dilute hydrochloric acid; pyritic and organic forms of sulfur are insoluble.

The pyritic sulfur is extracted from the hydrochloric acid residue (from the sulfate sulfur determination) with dilute nitric acid. The pyritic sulfur is then determined from the nitric acid soluble sulfur.

The organic sulfur is the difference between the total sulfur and the sum of the pyrite and sulfate sulfur.

The acceptable ASTM precision for sulfate and pyritic sulfur in the analysis sample is triplicate results with samples differing by no more than 0.02% for sulfate sulfur and 0.10% for pyritic sulfur, for coal containing 2% or more.

Calculations for sulfur S, 2.40, volatile coal retained is described by the following:

APPENDIX IV

Sample calculations for the values used in Table 3, page 56 are illustrated by the following tabulations. However, only the initial three and last size fractions will be used to illustrate sample calculations:

Size Fraction (mesh)	Wt. Coal (g) retained on screen	Wt. Coal 2671 (g) basis dry
6M x 8M	612	$(612 / 2671)100 = 22.91$
8M x 12M	277	$(277 / 2671)100 = 10.37$
12M x 16M	236	$(236 / 2671)100 = 08.84$
.....		
.....		
.....		
400M x 0	89	$(89 / 2671)100 = 03.33$
Total	2761	100.00

Weight retained is the actual weight of coal retained by a size fraction, i.e., for example the 8M x 12M fraction retained 277 grams of coal. The value 2671 is the total weight of coal in grams accumulated by the fourteen size fractions. Columns 4 and 5 of Table 3 are actual assay values obtained for weight-percent ash and sulfur found in each size fraction.

Calculations for column 6, % Wt. cumulative coal retained is described by the following:

Size Fraction (mesh)	Wt. Coal (g) retained on screen	Wt. Coal (g) cumulative retained	% Wt. Coal cumulative retained
6M x 8M	612	612	$(612 / 2671)100 = 22.91$
8M x 12M	277	$(277 + 612) = 889$	$(889 / 2671)100 = 33.28$
12M x 16M	236	$(236 + 889) = 1125$	$(1125 / 2671)100 = 42.12$
.....			
.....			
.....			
400M x 0	89	$(89 + 2582) = 2671$	$(2671 / 2671)100 = 100.0$
Total	2671	2671	100.0

The sequence of calculations for column 7, % Wt. cumulative ash retained is described by the following:

Size Fraction (mesh)	% Wt. Ash dry basis assay value	Wt. Ash (g) retained	Wt. Ash (g) cumulative retained
6M x 8M	31.10	$(612)(0.311) = 190.33$	190.33
8M x 12M	26.65	$(277)(.2665) = 73.82$	164.15
12M x 16M	23.95	$(236)(.2395) = 56.52$	320.64
.....			
.....			
.....			
400M x 0	40.30	$(89)(0.403) = 35.87$	716.82

Size Fraction (mesh)	Wt. Coal (g) comulative retained	Wt. Ash (g) comulative retained	% Ash comulative retained
6M x 8M	612	190.33	$(190.33 / 612) 100 = 31.10$
8M x 12M	889	264.15	$(264.15 / 889) 100 = 29.71$
12M x 16M	1125	320.64	$(320.64 / 1125) 100 = 28.15$
.....
.....
.....
<u>400 x 0</u>	<u>2671</u>	<u>716.81</u>	<u>$(716.81 / 2671) 100 = 28.50$</u>

The values for % Wt. sulfur cumulative retained may be obtained by following the above procedures illustrated for ash.

Calculations for column 9, % Wt. cumulative coal passing is described by the following:

Size Fraction (mesh)	Wt. Coal (g) retained on screen	Wt. Coal (g) cumulative passing	% Wt. Coal cumulative passing
6M x 8M	612	2671	$(2671 / 2671) 100 = 100.00$
8M x 12M	277	2059	$(2059 / 2671) 100 = 77.09$
2M x 16M	236	1782	$(1782 / 2671) 100 = 66.72$
.....
.....
.....
<u>400M x 0</u>	<u>89</u>	<u>89</u>	<u>$(89 / 2671) 100 = 3.33$</u>

Calculation for column 11, % Wt. cumulative sulfur passing is described by the following:

Size Fraction (mesh)	Wt. Sul. (g) retained on screen	Wt. Sul. (g) cumulative passing	% Sulfur cumulative passing
6M x 8M	23.56	106.23	$(106.23 / 2671)100 = 3.98$
8M x 12M	10.89	82.67	$(82.67 / 2059)100 = 4.02$
12M x 16M	10.27	71.78	$(71.78 / 1782)100 = 4.03$
.....			
.....			
.....			
400M x 0	3.16	3.16	$(3.16 / 89)100 = 3.55$
Total	106.23		

The values for % wt. ash cumulative passing may be obtained by following the above procedure illustrated for sulfur.

The actual experimental results from the float-sink tests are tabulated below. These values are the basis for weight-percent product coal and reject found in Table 4, page 57.

Size Fraction	Float (grams)	Sink (grams)	Total Wt. (grams)
6M x 8M	17.000	6.900	23.900
8M x 12M	16.257	7.647	23.904
12M x 16M	17.855	5.887	23.742
16M x 20M	14.296	6.424	20.720
20M x 30M	14.570	5.200	19.770
30M x 40M	14.248	5.435	19.683
40M x 50M	14.891	4.784	19.675
50M x 70M	17.564	6.521	24.085
70M x 100M	14.628	4.545	19.173
100M x 140M	17.830	6.040	23.870
140M x 200M	29.489	10.511	40.000
200M x 270M	10.982	9.962	20.944
270M x 400M	10.516	5.450	15.966
400M x 0	8.445	27.300	35.745

The weight-percent product coal (float) is calculated by dividing the grams of float coal collected by the combined weight in grams of float and sink collected per size fraction. Thus using the 12M x 16M fraction as an example:

$$\begin{aligned} \text{Wt. \% Float} &= [17.855 / (17.855 + 5.887)] 100 \\ &= 75.20 \end{aligned}$$

Tables 5 through 9 show calculated values for the feed coal

ash, total sulfur, sulfate sulfur, pyritic sulfur, and organic sulfur, respectively. It was assumed the assays from the float and sink fractions were more representative than those of the feed coal. It was believed that the feed coal was a more heterogeneous mixture, while the processed coal (float and sink fractions) was more homogeneous and thus would yield more accurate assay values. The following calculations are based on assay values taking the 40M x 50M as an illustrative example:

	Wt. (grams)	Wt.% Ash	Wt.% Total Sulfur	Wt.% Sulfate Sulfur	Wt.% Pyrite	Wt.% Organic
Feed	19.675	22.00	3.85	0.348	1.722	1.780
Float	14.891	7.95	3.59	0.246	0.856	2.489
Sink	4.784	75.60	5.62	0.810	4.339	0.471

Then for feed coal, the following calculations show the actual weight in grams of impurity present:

Ash	(0.22) (19.675) = 4.329
Total Sulfur	(0.0385) (19.675) = 0.80345
Sulfate Sulfur	(0.00348) (19.675) = 0.068
Pyritic Sulfur	(0.01722) (19.675) = 0.339
Organic Sulfur	(0.01780) (19.675) = 0.350

Similar calculations may be made for the impurities present in the float and sink samples based on the above assay determinations.

The sum of the ash in the float and sink must equal that in the feed coal. Although ash was discussed as an example, total sulfur or organic sulfur etc., may be calculated in the same manner.

The values for float and sink ash in grams using the above procedure are:

Fraction	Total Sulfur	Inorganic Sulfur	Organic Sulfur
2500 + 200	1.18383	0.47	0.71
200 + 100	3.61670	0.34	3.27
100 + 100	4.80053	0.81	3.99
100 + 200	5.16	1.20	3.96

Since from above, the weight of ash in the feed coal was determined by:

2500 + 200	1.18	0.71	0.47
200 + 100	3.62	3.27	0.35
100 + 100	4.329	3.99	0.34
100 + 200	4.329	3.76	0.57

Then a calculated value for wt. % ash may be obtained by:

1000 + 2000	19.675	12.46	7.21
2000 + 2700	4.329	3.99	0.34
2700 + 4000	24.40	1.90	2.50
4000 + 0	2.95	2.17	0.78

The error between the assayed feed coal ash and the calculated value for ash from the float and sink assay ash is:

$$\% \text{ Error} = [(0.24399 - 0.220) / (0.24399)] 100$$

= 9.83

The following table shows this error for the calculated feed coal values of Tables 5 through 9 inclusive:

Size Fraction (mesh)	-----Error-----				
	Ash	Total Sulfur	Sulfate Sulfur	Pyritic Sulfur	Organic Sulfur
6M x 8M	9.13	5.15	3.43	0.17	11.94
8M x 12M	11.55	2.63	1.34	6.08	0.79
12M x 16M	1.46	2.74	16.12	0.53	3.34
16M x 20M	20.59	5.16	1.20	11.16	20.33
20M x 30M	28.86	8.75	4.79	3.64	14.17
30M x 40M	10.45	1.47	2.56	6.45	3.19
40M x 50M	<u>9.83</u>	5.72	9.17	1.40	10.91
50M x 70M	11.53	0.59	12.25	0.24	3.24
70M x 100M	2.46	0.41	10.30	5.53	7.76
100M x 140M	1.28	0.02	3.04	1.87	0.72
140M x 200M	3.12	9.37	10.24	4.90	12.46
200M x 270M	0.36	2.72	1.29	6.49	1.04
270M x 400M	0.07	2.14	2.27	2.81	1.61
400M x 0	0.33	3.91	3.17	1.60	6.44