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SIZE AND MACERAL ASSOCIATION OF SULFIDE GRAINS
IN ILLINOIS COALS AND THEIR WASHED PRODUCTS

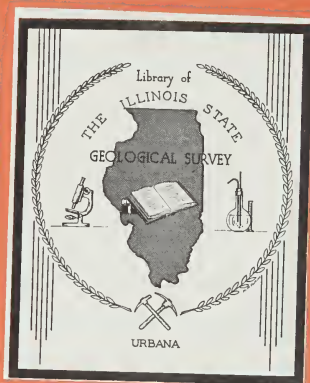
Richard D. Harvey
Philip J. DeMaris

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Final Report to the Coal Research Board
Illinois Department of Energy and Natural Resources
through the Center for Research on Sulfur in Coal
Contract 1-5-90068

Illinois Department of Energy and Natural Resources
STATE GEOLOGICAL SURVEY DIVISION



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**Richard D. Harvey
Philip J. DeMaris**


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ABSTRACT

Density-based processes are commonly used in coal preparation. The amount of pyrite and other sulfides removed from mined coal varies considerably from one deposit to another. Some variation is due to the design and operation of the preparation plant, but some is due to the physical properties of the coal material. The influence of the physical properties (primarily the size and maceral association of the pyrite grains) has not been sufficiently evaluated. The objectives of this project were to develop a microscopic procedure for assessing these properties, and to compare the results with float-sink tests for a few samples.

Microscopic measures were made on polished specimens of samples crushed to less than 840 micrometers (μm) in size. The apparent diameter of pyrite grains was measured along a line superposed on the the grain so as to bisect the grain (Martin's Statistical Diameter). Precision tests indicated that at least 1000 grains selected at random must be measured to obtain a reproducible mean diameter characteristic of the sample. The maceral-mineral association of each measured grain was classified as one of seven different types (modified microlithotypes).

The procedure was used to study three feed samples and six to seven float-sink fractions from each. The mean diameter of all pyrite grains of the specimens ranges from 6 to 30 μm . Pyrite grains in low density fractions are almost entirely associated with maceral rich particles, which average 6 to 10 μm in diameter. The largest enclosed grains are associated with other pyrite grains within pyritic coal particles.

The characteristic found most useful for evaluating the float-sink behavior of coal was the percentage of the grain diameters within the various maceral associations. Pyrite grains judged easy to remove are free grains and grains enclosed in carbominerite or pyritic coal particles; pyrite grains judged hard to remove are those enclosed in vitrite, inertite, liptite, and bi- and trimacerites. The ratio of the summed diameters within these two groups of maceral associations gives a value we have defined as the pyrite cleanability index (PCI). PCI correlates very closely with pyritic sulfur content, measured chemically, and may provide a useful means to evaluate the cleanability of feed coals and compare the efficiency of various cleaning methods.

INTRODUCTION

Issues

Density-based cleaning processes efficiently remove a high percentage of the pyrites and other sulfide impurities for some coals, but for others these processes are rather inefficient. These differences are illustrated by the washability curves of pyritic sulfur in two coals (Fig. 1). A coal that can be cleaned efficiently will follow a path similar to curve 1a in the figure for which the pyritic sulfur content of the float coal will drop rapidly with only a small reduction of recovery from the 100 percent level. A coal that can not be cleaned efficiently will follow a path similar to curve 1b. These differences have been observed for many years, but the factors responsible for them have not been thoroughly investigated.

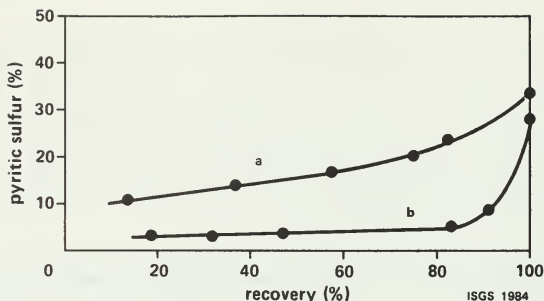


Figure 1 Washability curves of pyritic sulfur for two types of coals: curve a, for a coal that can be cleaned efficiently, drops rapidly between 90 and 100 percent recovery; curve b, for a coal that cannot be cleaned efficiently, drops slowly in this recovery range.

We postulate that coals with large and abundant sulfide grains more or less free of macerals would readily sink in washing processes and yield a rapidly decreasing pyritic sulfur-recovery curve (fig. 1a) and that coals with small and abundant sulfide grains enclosed within macerals would tend to float and yield a curve such as 1b. Because these physical characteristics are best determined by microscopic methods, a microscopic procedure to characterize the size and maceral association of sulfide grains in coals needed to be developed and applied to a series of float-sink tests. The results of such a study might provide a quantitative measure of the cleaning potential for coals tested and thereby assist in the design of improved washing processes.

Previous investigations

The problem of sulfur impurities in Illinois coals is far from a new issue. Analyses of sulfur in coals were included in reports dated as early as 1894, as well as in the first report on Illinois coal published by the University of Illinois Experimental Station (Breckenridge, Parr, and Dirks, 1906). By 1919 the subject warranted a special study on the methods to determine the various forms of sulfur in coal (Powell and Parr, 1919). Shortly thereafter, the U.S. Bureau of Mines initiated an investigation of the distribution of sulfur in coal seams and was the first to report the inhomogeneity of pyrites in Illinois coals (Yaney and Fraser, 1921). More recently the topic was reviewed and additional geologic and analytical data were presented by Gluskoter and Simon (1968). Chemical investigations of the partitioning of pyrites and other ash forming minerals in cleaned and refuse fractions of coals led to many improvements in coal preparation in recent years (Saltsman, 1970; Deurbrouck, 1972; Miller, 1977; and Liu, 1982).

Microscopic and chemical characterizations of five high-sulfur coals, made in conjunction with selective pulverization carried out by Glenn and Harris (1961) showed that pyrites tended to concentrate in the -200 mesh fractions of crushed coals. They measured the chord lengths across grains encountered along a regular series of traverses over a polished pellet of -1/2 inch coal mixed with carnauba wax as a binder, and reported the average volume and

percentage of grains occurring in seven different size ranges. Their tests of a sample from Illinois indicated that pyrite grains with diameters of $\sim 20 \mu\text{m}$ represented only 1.6 percent of the total weight of pyrite, but 90.3 percent of the pyrite grains (by number) in the sample. Glenn and Harris (1961) further observed abundant pyrite grains along bedding planes within the coal particles and suggested that a new design for grinding machines might increase the liberation of pyrite.

In a report on the petrographic and chemical forms of sulfur in samples from the Pittsburgh seam, Gray, Schapiro, and Coe (1963) found that the particles of coal of intermediate density contained the greatest abundance of unliberated pyrite. They also observed that, on a dry and ash-free basis, the organic sulfur correlated linearly with increases in the liptinite maceral content in all size and density fractions of the coal. Bomberger and Deul (1964) used a motorized stage to translate a polished epoxy pellet of crushed coal under a microscope equipped to continuously measure reflectance. The apparatus scanned the pellet and recorded the chord length of exposed grains of high reflectance.

The method used by McCartney, O'Donnell, and Ergun (1969) was similar to that of Glenn and Harris (1961). Samples of -14 mesh coal, pelletized with a plastic binder and polished, were examined at 375 magnification along 80 traverses. They recorded the diameter of all pyrite grains encountered, approximately 3,000. They assessed the precision of their method in terms of the coefficient of variation, which for their tests ranged between 3.5 to 7 percent. This was deemed satisfactory in view of the inhomogeneity of pyrite distribution in coals. McCartney, O'Donnell, and Ergun (1969) reported the mean size of pyrite grains in 61 coals, including eight from Illinois. The mean size in the Illinois samples ranged from 27 to 90 μm , while the mean in all other samples ranged from 20 to 400 μm .

Kneller and Maxwell (1983) studied the size, shape, and distribution of microscopic pyrite in some Ohio coals. They distinguished eight different types of pyrite: framboidal, euhedral, fracture filling, layered, dendritic, cell filling, bleb, and submicron. Their observations on polished specimens of crushed samples, mounted in epoxy, revealed a marked enrichment of pyrite within the 40- to 32- μm size range in most of their samples. Cecil, Stanton, and Dulong (1981) examined the location, type, and size of pyrites in the lower Freeport Coal of West Virginia. They reported data on grain size using standard coal pellets and normalized their microscopic data on each sample by use of its pyritic sulfur content as determined by chemical methods.

Lebiedzki and Dutcher (1977) pioneered in the application of computer controlled image analysis of coal for size measurements of pyrites. Robinson and Stark (1983) studied the size of pyrite grains in Illinois coals using an automated image analysis system based on the scanning electron microscope. Polished surfaces of pellets were scanned under an electron beam and the system automatically identified pyrite by its very bright back scattered electron image, relative to other coal constituents. They expressed their data in terms of the surface area of pyrite grains per unit volume of coal. Of the six potential sources of error investigated by Robinson and Starks (1984), only the error due to the inhomogeneity of pyrite grains from area to area on the polished pellet was judged to be a significant source of variation.

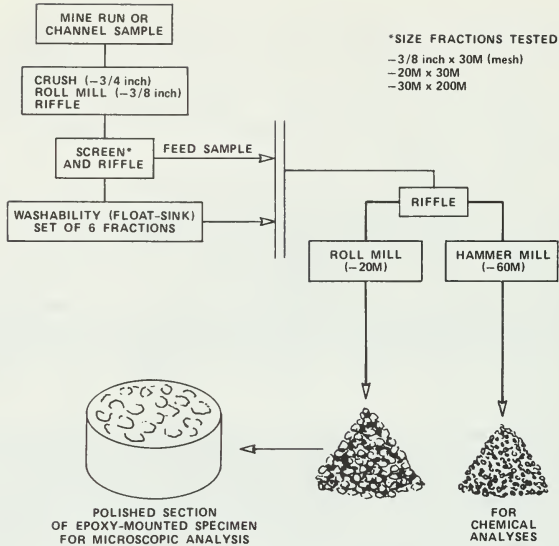


Figure 2 Sample preparation.

Table 1 Project samples.

Sample Location in Illinois	Seam	Sample type	Particle size*	Sample number(s)
Southern	Herrin (No. 6) Coal	Run of mine 6 float-sink fractions of C22169	-2 inch 20M x 30M	C22169 C22232 to C22237
Southern	Springfield (No. 5) Coal	channel 6 float-sink fractions of C22173	-2 inch 20M x 30M	C22173 C22238 to C22243
Central	Springfield (No. 5) Coal	channel [†] coarse fraction of C22419 7 float-sink fractions	-2 inch 3/8" x 30M 3/8" x 30M	C22419 C22458 C22422 to C22428
		fine fraction of C22419 7 float-sink fractions	30M x 200M 30M x 200M	C22457 C22429 to C22435
		Run of wash plant	-1 inch	C22420

*M: mesh; 20M = 841 μ m, 30M = 600 μ m, 200M = 75 μ m[†]Composite of block samples taken from each layer of the seam.

Frankie and Hower (1984) analysed samples from the Herrin, Springfield, and other seams, most in Kentucky, for the size, type, and microlithotype distribution of their pyrite constituents.

Objectives

The objectives of this project are to: (1) develop a method for determining the size distribution of sulfide grains (pyrites*) and their maceral associations in coal; (2) characterize the diameter of sulfide grains and their maceral association in selected samples of Illinois coals; and (3) seek useful correlations between these properties and the pyritic contents of various density (washability) fractions of the coals.

Acknowledgements

Several staff members of the Illinois State Geological Survey contributed to this work. R. H. Shiley collected two of the samples studied; L. Camp and D. Lowry prepared the samples; L. Kohlenberger supervised the chemical analyses; L. Khan and J. Buckentin supervised the float-sink testing; J. Kaczanowski designed and constructed needed electrical components. The project was supported by the Illinois Coal Research Board through the Center for Research on Sulfur in Coal. Dr. Paul D. Robinson, Geology Department, Southern Illinois University, reviewed the manuscript.

SAMPLES AND METHODS OF PREPARATION

Samples were collected from three mines: one, a mine run (tippie) sample from the Herrin (No. 6) Coal seam in southern Illinois, and two channel samples from the Springfield (No. 5) Coal (table 1).

Samples were prepared for analyses as shown in fig. 2. Representative splits of the samples were crushed to 840 μm or less (-20 mesh), mounted in epoxy, and polished to expose cross-sections through several thousand particles of coal for microscopic study. Two epoxy mounted specimens were prepared and studied for most samples. These procedures followed those described in standard D2797 of American Society for Testing and Materials (1983). Other splits were prepared and subjected to sequential density separations by float-sink (washability) methods, following procedures described by Helfinstine et al. (1974). Other splits of the samples, including float-sink fractions, were crushed to 250 μm or less (-60 mesh) and chemically analyzed for their moisture, volatile matter, fixed carbon, ash, varieties of sulfur, and total sulfur content.

MICROSCOPIC METHODS

General remarks

Pyrite grains exhibit a wide diversity of size and shape in coals. The task of measuring their size is a complex one; nonetheless, if consistent microscopic procedures are followed, the measurements can be used to determine differences between samples that will be meaningful for coal preparation. In addition, microscopy permits the simultaneous determination of the grain's association with other mineral and maceral constituents in the coal.

*The term pyrites refers to grains of iron sulfide, of which pyrite is the most abundant and marcasite is a trace component in Illinois coals.

There are some inherent difficulties in the microscopic measure of size. Careful procedures are required during sample preparation to ensure that a representative sample is examined under the microscope. In this connection, plucking of grains while polishing is a potential problem, as is enrichment of heavy minerals on the bottom of the epoxy mounted specimen. Therefore we prepared, mounted, ground and polished each sample in a consistent manner, using a minimum amount of epoxy to prevent segregation and a minimum amount of polishing to reduce plucking. These procedures can therefore be used to measure size characteristics that enable comparisons to be made between samples.

Another inherent difficulty concerns the fact that random cross-sections of grains rarely pass through the maximum diameter of three-dimensional grains. As our primary use of grain-size data is to make comparisons between samples, this difficulty is of secondary importance. We assume this sectional effect is essentially the same for one sample as another. Given this assumption, and random sectioning, a sample containing large grains should yield a mean size that is proportionately larger than another sample with smaller grains.

Automated scanning method tried

Attempts were first made to adapt a motorized scanning stage that was available in the laboratory. With this stage the specimen could be scanned along a traverse while a photometer recorded the reflectance at regularly spaced points (3- μ m diameter) along the traverse. Sulfide grains, being very highly reflective, would appear on the record as high peaks. The width of the peak would be proportional to the diameter of the grain. The electronic signals to the motorized stage were modified with this goal in mind to make the scan velocity as constant as possible and to interface the control unit with our microcomputer. Computer programs were written to store and to evaluate the reflectance record. Several test scans were run on a specimen; however, the scanning velocity could not be maintained perfectly constant, and too many traverses were required on each specimen to obtain data on enough grains. The motorized stage was not designed for scanning back and forth and so the analyst had to manually set up each traverse across the specimen. This procedure also yielded rather inaccurate diameter values insofar as the traverses passed along randomly oriented chords through the grains, rarely along the apparent diameter. The resulting record did not provide a consistent measure of the grains. Because of these disadvantages we stopped further work on this method.

Method and procedures adopted

Grain diameter measurement. The ribbon method was chosen as a means to randomly select grains for manual measurement (Van Der Plas, 1962). The specimen pellet was manually traversed across the stage of the microscope with a mechanical device, left to right, and each sulfide grain or cluster of contacting agglomerated grains observed to occur with a 240- μ m wide ribbon centered on the line of traverse was measured. Some 10 such ribbons, spaced 2- to 4-mm apart were traversed so as to cover about 80 percent of the pellet surface. Measurements were made in sets of 500 grains for each sample. Details of the microscopical procedure are given in section I of fig. 3.

Microscopic Procedures For
Pyrite Characterization in Particulate Coal Samples

I. Grain Size Determination

1. A polished sample pellet is scanned along a 240 μm wide ribbon, left to right. This width corresponds to the length of the micrometer graticule on the pellet.
2. The diameter recorded is the length of the graticule line superposed on the grain when the grain is positioned so that the line bisects it. With the line of the micrometer graticule set vertically in the microscope field of view, record the diameter in whole units of micrometer divisions of each grain that occurs along the scan. Do not count grains of less than half a division. Measure only the first 5 grains encountered in a single particle of coal.
3. An irregular shaped grain (or cluster of contacting grains) is positioned so that approximately equal mass of the grain occurs on both sides of the line of measure. Clusters or dendritic forms are measured as a single mass excluding intervening pores of coaly matter. One exception is permitted for very large rectangular grains larger than the graticule (240 μm). The length and width of such a grain are measured, and the diameter (in divisions) of a circle of equal area is computed and recorded. Return to the original scanning track after making these measurements.
4. Approximately 10 parallel scans are made across the pellet, spaced at least 2 mm apart so as to cover more than 80 percent of the pellet surface. At least 1000 grains must be measured.

II. Grain Association

The association of pyrite grains is classified simultaneously with the size measurement according to the following sequence of questions:

1. Is the grain free or enclosed? If it is free of macerals or the particle contains less than 5 percent maceral matter, record it as "F" and go to the next grain. Otherwise it is one of the following types of enclosed grains:
2. Is the enclosed pyrite grain in a particle that exceeds 20 percent in total pyrite by surface area? If yes, record "P" for pyritic coal and go to next grain; otherwise go to question 3.
3. Is the enclosed pyrite grain in a particle that contains from 20 to 60 percent mineral matter, or has from 5 to 20 percent pyrite? If yes, record "C" for carbominerite and go to the next grain; otherwise go to question 4.
4. Is the enclosed pyrite grain in a particle that is either 95 percent or more vitrinite, or liptinite, or inertinite and less than 20 percent mineral matter or less than 5 percent pyrite? If yes, record "V" of "L" or "I" respectively and go to the next grain; otherwise record "T" for trimacerite or bimacerite. The particle should then contain more than 5% of a mixture of vitrinite and/or liptinite or inertinite and less than 20% mineral matter (or less than 5% pyrite).

Figure 3 Microscopic procedures.

The grain size characteristic measured was the Martin's statistical diameter (Martin, 1928), the length of a line that bisected the grain seen under the microscope (fig. 4). The mean value of Martin's diameter of sand grains was found by Heywood (1947) to give substantially the same value as that obtained from more tedious measurements of the projected area of the grains. For the few especially large, rectangular grains (greater than 240 μm across), which occurred in many samples as cleat-fill pyrite, we measured their length and width and recorded the diameter of a circle of equal area. Because a few coal particles contain numerous grains, we measured only the first five grains encountered in a single particle to ensure that a large and representative number of particles was examined. Grains or clusters of contacting grains (framboids) occurring as polycrystalline and porous masses were measured as if they were a single grain. Likewise, dendritic groupings of crystals were treated as a single mass; the intervening coaly matrix (see fig. 8) was visually excluded.

The diameter recorded was that of the grain measured to the nearest division on the gratricular line, superposed on the image of the grain. Given the magnification used (500x), each micrometer division was 2.41 μm on the surface of the grain. This factor was used to convert the recorded values to true length in micrometer (μm) units. Grains less than half of a division in size (<1.2 μm) were not counted; this is justified because the weight-effect of such ultrafine grains of pyrite on washability of the coal is considered negligible.

Maceral associations. To document the associations of the sulfide grains, a scheme to classify the associations was developed (table 2). This classification was adapted from the microlithotype classification of coal described by Mackowsky in Stach et al. (1982). Sulfide grains occurring free of macerals were recorded as a free association, and grains enclosed within or attached to macerals were recorded as one of six possible microlithotype associations (table 2). In contrast to the usual microlithotype procedure, which uses a 20-point graticule to quantitatively determine the maceral and mineral percentages within a 50- μm square area, we visually estimated the percentages within the entire particle of coal containing the grain. The sequential manner in which we determined the associates of each grain measured is described in figure 3.

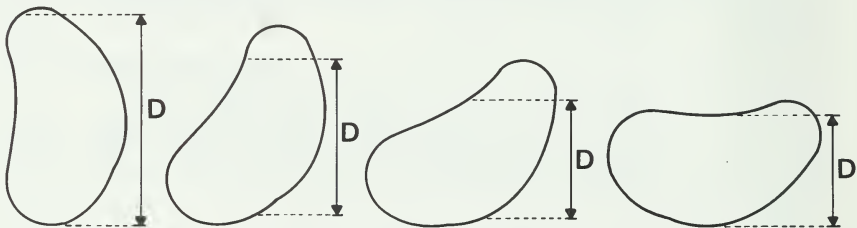


Figure 4 Martin's Statistical Diameter (M) measured on the same grain in different orientations (after Heywood, 1946).

Computer programs. Three computer programs were written to process the data (see appendix). The programs were written in Microsoft Basic to run on a CP/M 2.2 operating system, with 64K bytes of memory. Our microcomputer is one of the earliest made, a MITS model Altair 8800b equipped with an IMSAI dual disk drives.

The program to enter and store the data, PSTORE, prompts the microscopist for the sample number, date, number of grains measured and the magnification factor. The number of micrometer divisions corresponding to the Martin's diameter of each measured grain is entered and stored along with one of the codes corresponding to its maceral association. PSTORE allows changes to be made for erroneously entered data.

The program PCHAR recalls data from a file created by PSTORE, converts the diameter in division (graticule) units to micrometers, computes statistical values, and prints the results in a report entitled, "Pyrite Characterization." Subtotals, percentages, means and standard deviations are computed for all pyrites measured and for each of the various types of pyrites. In addition, the program computes statistical values expressed as grain number, grain diameter, grain area (assuming circular grains), and grain volume (assuming spherical grains). These values, along with the type of maceral association, are printed on the report (fig. 9).

The program SIZEFREQ uses the raw data from PSTORE and converts the grain's size in division units to ϕ units (Krumbein, 1934), where ϕ is the $-\log_2 D$ and D is the grain diameter in mm. Each of these values is grouped into one of several ϕ class intervals, converted to percentage units, and listed in a table. These results can be readily plotted as a histogram or a cumulative distribution curve of grain sizes from which the median diameter can be interpreted (fig. 10).

Table 2 Classification of pyrite grain associations.

Association	Maceral-mineral composition of coal particle
1. Free grains	<5% macerals attached
2. Enclosed grains	
2a. Vitritite	>95% vitritite macerals encloses pyrites
2b. Liptite	>95% liptite macerals encloses pyrites
2c. Inertinite	>95% of inertinite macerals encloses pyrites
2d. Trimacerite	>95% mixtures of macerals. This category includes bimacerites.
2e. Carbominerite	macerals plus: 5 to 20% pyrite, or 20 to 60% clay, quartz or calcite
2f. Pyritic coal	>20% pyrite

RESULTS

Pyritic sulfur in washed coals

The weight yield for the various float-sink fractions of the mine run and channel samples and their chemical analyses are given in table 3, and washability curves derived from these data on typical samples are shown in figure 5. The points along the curves were computed as described by Helfinstine et al. (1974). The raw data for pyritic sulfur in each float-sink fraction, starting with the lightest fraction, is multiplied by the yield of that fraction and cumulative percentages of the products are plotted against the cumulative yield. Figure 5a represents the -20 x 30 mesh fractions of two samples. Note that the low-recovery end of the curve for the Herrin sample

Table 3 Weight yield and selected chemical and maceral analyses of float-sink tests.*

Sample No.	Feed and density	Yield	H-T ASH	Sulfate Sulfur	Pyritic Sulfur	Organic Sulfur	Total Sulfur	Vitrinite	Liptinite	Inertinite
HERRIN (NO. 6) COAL - SOUTHERN IL										
C22169	mine run	--	14.5	0.140	1.53	1.01	2.69	86.1	6.1	7.9
232	1.25F	9.85	3.1	0.031	0.34	0.97	1.34	89.1	3.9	7.0
233	1.30FS	28.79	3.2	0.32	0.40	0.93	1.36	86.4	4.2	9.4
234	1.35FS	20.41	4.6	0.042	0.55	0.91	1.51	87.4	4.6	8.0
235	1.40FS	12.46	8.1	0.063	0.78	0.83	1.67	85.2	4.9	9.9
236	1.60FS	19.07	15.3	0.081	1.03	0.72	1.84	74.1	5.8	20.1
237	1.60S	9.41	67.7	0.367	8.09	0.11	8.57	68.4	2.5	29.1
SPRINGFIELD (NO. 5) COAL - SOUTHERN IL										
C22173	channel	--	10.8	0.342	2.02	2.17	4.53	82.9	2.9	14.2
238	1.25F	12.28	3.0	0.021	0.27	2.23	2.52	84.9	5.5	9.4
239	1.30FS	15.35	5.2	0.042	0.60	2.19	2.83	84.8	4.4	10.8
240	1.35FS	7.57	6.7	0.053	0.81	2.15	3.02	83.1	4.2	12.7
241	1.40FS	47.48	8.7	0.111	1.14	2.06	3.31	80.3	6.6	13.0
242	1.60FS	11.16	15.6	0.256	3.40	1.95	5.60	75.0	4.3	20.7
243	1.60S	6.16	46.0	0.388	12.84	0.69	13.92	55.5	3.0	41.5
SPRINGFIELD (NO. 5) COAL - CENTRAL IL										
C22419	channel	--	11.8	0.058	1.63	2.15	3.83	83.9	6.1	10.1
458	3/8 x 30M	89.00	12.9	0.329	1.43	2.12	3.88	--	--	--
422	1.25F	3.56	3.3	0.055	0.33	1.82	2.20	91.8	5.5	2.7
423	1.31FS	31.43	5.3	0.078	0.45	1.92	2.45	87.2	8.8	4.1
424	1.35FS	32.36	8.5	0.143	0.72	2.34	3.20	83.4	10.0	6.6
425	1.40FS	26.46	13.3	0.354	1.55	2.11	4.01	83.7	9.5	6.8
426	1.60FS	4.12	23.6	0.515	3.42	1.85	5.78	67.6	13.6	18.8
427	1.80FS	0.84	36.7	0.442	4.62	1.15	6.21	55.3	9.7	35.0
428	1.80S	1.23	55.0	0.578	14.17	0.54	15.28	25.4	4.6	70.0
457	30 x 200M	11.00	12.1	0.318	0.86	1.99	3.17	--	--	--
429	1.25F	5.86	4.4	0.161	0.30	2.12	2.58	76.5	8.5	15.0
430	1.31FS	7.99	4.7	0.142	0.35	2.09	2.58	73.4	10.7	16.0
431	1.35FS	15.88	4.7	0.132	0.38	2.11	2.63	73.8	13.8	12.4
432	1.40FS	39.20	5.7	0.148	0.52	2.06	2.73	75.1	16.1	8.9
433	1.60FS	21.65	14.4	0.291	1.51	1.93	3.73	65.1	17.4	17.4
434	1.80FS	3.79	36.2	0.543	4.46	1.16	6.16	29.6	10.4	60.0
435	1.80S	5.63	63.5	0.747	10.33	0.19	11.27	23.5	5.6	70.8
459	-200M	1.00	18.3	0.465	1.81	1.67	3.94	--	--	--
420	wash plant	--	12.4	0.237	0.99	2.05	2.76	--	--	--

*Abbreviations: F=float, S=sink, FS=float sample of the sink from the next lighter fraction, M=mesh size. size.
 +The Vitrinite, Liptinite and Inertinite results are based on point counts of 468 to 1055 macerals per sample and reported on a volume percent, mineral-free basis; all other results are on a weight percent, dry basis.

nearly levels off while the curve continues to drop for the Springfield sample. These curves are different for the two samples. Nonetheless, both of these samples lost considerably more pyritic sulfur above 80 percent recovery than did both the fine and coarse fractions of the other Springfield sample (fig. 5b).

The float-sink curves for the coarse and fine fractions of sample C22419 (fig. 5b) reflect the difficulty in cleaning this coal. Our sample was selected in the mine so as to exclude clay dike material (locally a mining problem), thereby enabling the sulfur cleaning problem to be studied more directly. The whole sample (C22419) was crushed and split into a fine (30 X 200 mesh) and a coarse (3/8 inch X 30 mesh) fraction: 89 percent was coarse and 11 percent was fine. Differences in the curves for the two subsamples are largely explained by this particle size differentiation. The cleat-fill and other megascopic sulfides were preferentially freed and concentrated in the fine fraction, which had a higher content of pyritic sulfur (1.39%) and a steeper slope for the washability curve between 80 and 100 percent recovery. Both the coarse and fine fractions retained some of the coarser sulfide materials and had a similar amount of finely dispersed pyrite within coal particles. As a result, the two curves converge toward the low-recovery side.

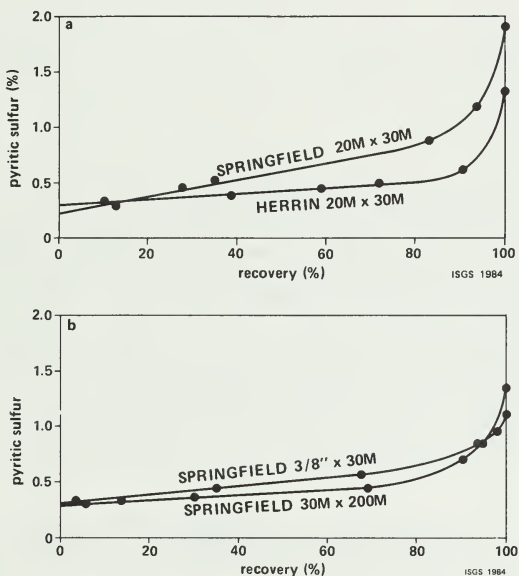


Figure 5 Washability curves for pyritic sulfur in: (a) channel samples of 20x30 mesh: C22173 (Springfield Coal), C22169 (Herrin Coal); (b) coarse (C22458) and fine (C22457) fractions of channel C22419 (Springfield Coal).

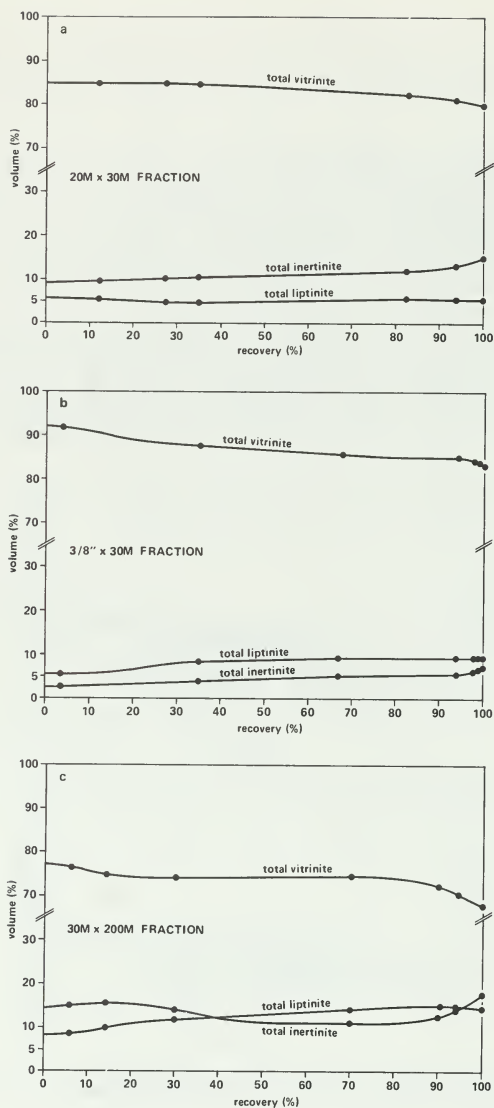


Figure 6 Washability curves for the three group macerals: (a) channel C22173; (b) coarse fraction (C22458) of channel C22419; (c) fine fraction (C22457) of channel C22419.

Macerals in washed coals

The general trends of maceral distributions in the float-sink tests (table 3) are as follows:

- Vitrinite is progressively enriched in the lighter fractions and conversely is depleted in the heavier fractions.
- Liptinite tends to be slightly less abundant in both the light and heavy fractions and enriched in the 1.40 to 1.60 density range.
- Inertinite is progressively depleted in the lighter fractions and enriched in the heavier fraction.

Departures from these trends are due primarily to differences in the density of individual macerals, but also to typical maceral associations, and to the degree of mineralization of the inertinite components. Even though liptinite has the lowest density the particles that are mostly pure vitrinite concentrate in the lightest fractions, because liptinite is most often associated with finely disseminated clays. Inertinite (primarily fusinite and semifusinite) occurs in particles having a broad density range because of their susceptibility to mineralization during a late stage of coalification. Unmineralized fusinite and semifusinite have densities slightly higher than that of vitrinite, and many of these macerals occur as smaller inclusions in the medium-to-light density fractions. The coarse and fine fractions of C22419 behaved somewhat differently. The differences are shown by the inertinite content which in the lighter fraction(s) of the coarse sample was more enriched (15 to 16 %) while the fine sample was most depleted (table 3).

The washability curves for the macerals (fig. 6) were determined by the same mathematical procedures that were used for the pyritic sulfur; however, the maceral data are in units of volume percent rather than weight percent. The curves illustrate many of the same trends as discussed previously. The washability behavior of the macerals in the 30 X 20 mesh fraction of the southern Springfield channel shows rather smooth curves (fig. 6a). On the other hand, the behavior of macerals in the coarse (fig. 6b) and fine (fig. 6c) fractions of sample C22419 is different. Inertinite-rich particles were preferentially concentrated in the fine fraction (30 X 200 mesh). These types of particles were preferentially released during crushing because fusinite and semifusinite types of inertinite occur in the coal aligned mostly parallel to bedding. This causes preferential breakage along their boundaries, and such particles tended to concentrate in the medium to light float-sink fractions. On the other hand, much of the fusinite and semifusinite was associated with minerals and such particles were therefore concentrated in the heaviest fractions.

The liptinite macerals were preferentially concentrated in the finer fraction of C22419, apparently because they occur more abundantly along bedding planes where breakage of particles occurs. Their preferred alignment along bedding planes probably contributed to breakage along their boundaries, which resulted in their enrichment in the fines. While liptinites have the lowest density of the three maceral groups, they rarely dominate coal particles by weight or volume. Liptinites macerals are generally the most evenly distributed maceral group, but still concentrate in the middle of the recovery range (fig. 6), corresponding to the 1.40-1.60 density range.

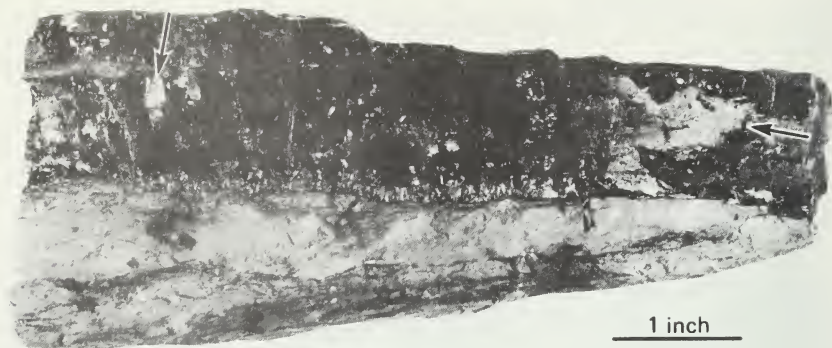


Figure 7 Examples of the two most common types of megascopic occurrences of pyrite in coal: (1) cleat-fill shown as light gray patches (arrows), (2) part of a lenticular nodule in the lower part of the photograph.

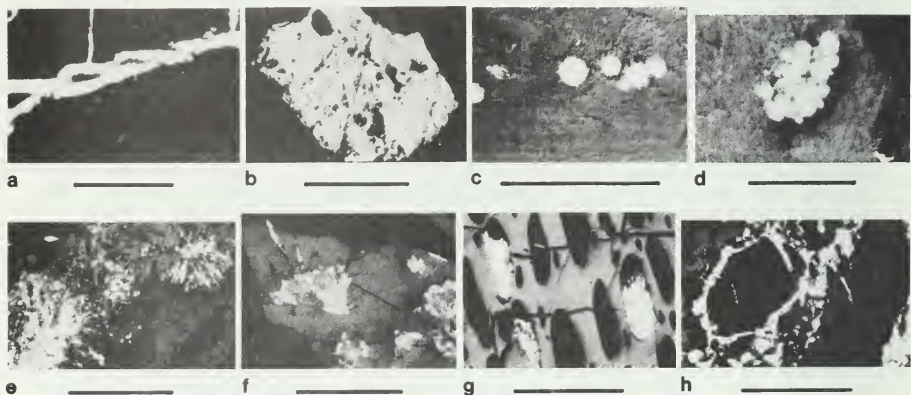


Figure 8 Examples of microscopic occurrences of pyrites (shown here as white grains) enclosed in vitrinite (except b and g): (a) coarse grains along a veinlet; (b) a very large but porous grain free of macerals; (c) frambooids; (d) clump of frambooids; (e) radiating crystallites (dendritic pattern); (f) irregular masses; (g) grains filling some pores in fusinite; (h) irregular patch work of grains. Bar scale is $10\ \mu\text{m}$ for each photograph.

Grain-size results

Occurrences of pyrite in the samples. As is well-known, pyrites are widely distributed in coal seams and they occur in a variety of forms. Forms visible with the unaided eye (macroscopic) occur as: (1) cleat-fill veinlets, commonly less than 1/2-mm wide and 20 to 30-mm long, oriented normal to the bedding of the seam; (2) lenticular pyrite, which occurs as lenses of various sizes and shapes oriented parallel to the coal's bedding; (3) nodules or "sulfur balls," roughly lenticular, measuring up to a foot or more across; and (4) disseminated crystals and irregular aggregates of various sizes and shapes. The cleat and lenticular types are common in Illinois coals (fig. 7).

Several microscopic forms of pyrites are readily distinguished. The common types in Illinois coals are: (1) a microscopic variety of cleat-fill; (2) irregular masses or blebs; (3) framboidal pyrite--single spheres of microcrystalline pyrite, commonly 2 to 20 μ m in diameter; (4) agglomerates or clusters of framboids; (5) very small crystals; (6) dendritic groupings of crystals; (7) cell fillings; and (8) replacements of plant material (fig. 8).

Because the mine run and channel samples tested were crushed, riffled, and processed to provide a representative sample for microscopic study, the megascopic pyrite types described above appeared microscopically as broken pieces, most commonly as free grains. The diameter of isolated grains and framboids (fig. 8b and c) and other elliptical masses was measured quite consistently. The adopted procedure provided excellent results for these types of grains. However, the measurement of some of the more irregularly shaped forms (clusters of framboids, dendritic groups of crystals, plant replacement forms) (fig. 8d, e, f, and h) demanded more subjective decisions by the microscopist as to where to position the "grain" for diameter measurement and in some cases as to the exact diameter to record. The procedures adopted (fig. 3) had to be meticulously followed. Despite some variation in repeat measurements of the irregular forms, their contribution to inaccuracies of the mean statistical diameter is relatively small because only a few of these forms were present in the samples tested.

Precision of mean diameter determination. Repeat measurements of 500 grains were made at various times on two pellets of some samples (table 4). Although the mean diameter of 500 grains in both free and enclosed classes varied somewhat from that of the next set of 500 grains, the mean diameter based on 1000 measurements was repeated within 0.5 μ m when a third and fourth set of 500 grains was added to the total. To date, only three samples have been tested in this manner. Additional samples need to be evaluated, but these results show that 1000 grains in each sample should be measured to obtain an acceptable value for the mean diameter. The mean value should be reported only to the nearest micrometer.

The greatest variation in the results stems from differences in distribution of grains within the pellet examined. This is in agreement with the results of Robinson and Starks (1984). To counter this problem, more than 80 percent of the pellet surface should be scanned and two pellets should be tested.

Somewhat related to precision of diameter measurement is the precision of pyrite abundance measurement. It has long been recognized that the overall precision of maceral (and mineral) analysis based on 500 to 1000 point counts

is ± 2 volume percent. However, our point count analysis for pyrite, converted to pyritic sulfur on a weight basis, does not closely agree with the chemical analysis for many samples. Our counts increased with increasing chemical values, as they should, but we consistently observed more pyrite than the chemical analysis indicated was present. The correlation was poorest for samples with high pyrite content. Any pellet preparation irregularities, especially settling of large pyrite grains, would show up dramatically in sink fractions. We tentatively conclude that there was some settlement of pyrite to the bottom surface of the specimens during epoxy mounting and that this caused the poor correlation.

Size and association of sulfide grains in project samples.

The mean diameter of sulfide grains in the three mine run and channel samples range from 8 to 11 μm (table 5). The mean diameter of the free grains is smaller (6 to 7 μm), while that of the enclosed grains is larger (13 to 17 μm). In these samples, 13 to 17 percent of the grains are free of macerals and other minerals. The large grains are associated with other pyrites that are commonly enclosed in particles of pyritic coal. The mean diameter is generally smaller for grains enclosed within maceral-mineral rich particles than it is for those in carbominerite and pyritic coal particles (table 5). Pyrites in density fractions lighter than 1.4 g/cc are predominately associated with trimacerite (and bimacerite) particles, and these grains average 6 to 10 μm in diameter. In the mine run and channel samples, pyrites are largest in the pyritic coal association. In density fractions, the mean grain diameter in both free and enclosed associations is largest in the most dense fraction.

Table 4 Precision of mean diameter determination.

sample	# grains	Cum.* #grains	Mean diameter (\bar{d} in μm)					
			all grains		free grains		enclosed grains	
			\bar{d}	Cum \bar{d}	\bar{d}	Cum \bar{d}	\bar{d}	Cum \bar{d}
C22173	500	500	6.6	6.6	5.4	5.4	6.8	6.8
	500	1000	10.9	8.8	8.0	6.9	12.0	9.3
	490	1490	6.8	8.1	4.1	5.5	9.2	9.3
	500	1990	8.9	8.3	4.9	5.4	10.4	9.6
	500	2490	9.6	8.6	4.3	5.1	12.3	10.1
C22235	500	500	4.7	4.7	3.0	3.0	5.4	5.4
	500	1000	8.6	6.7	3.5	3.2	10.3	7.9
	500	1500	8.2	7.2	3.0	3.2	9.4	8.4
C22419	500	500	8.6	8.6	7.0	7.0	9.1	9.1
	500	1000	12.4	10.5	6.7	6.9	13.9	11.5
	500	1500	12.2	11.1	6.7	6.8	13.6	12.2

*Cumulative values.

Table 5 Size of 500 or more sulfide grains and their maceral associations. ^b

Sample No.	Sample Type	All grains d	Free grains d %	Enclosed grains d %	Enclosed grains by association								
					Vitrite d %	Inertite d %	Trimacerite d %	Carbominerite d %	Pyritic Coal d %				
HERRIN (NO. 6) COAL (Southern Illinois)													
C22169	mine run [†]	8*	7 15	9 85	7 12	12 2	7 47	10 11	24 14				
C22232	1.25F	6	3 13	6 87	6 22	4 <1	6 62 ^a	4 <1	21 2				
C22233	1.30FS	8	3 5	8 95	9 28	12 2	8 59	15 3	30 2				
C22234	1.35FS	8	3 11	10 89	9 16	7 1	9 56	14 13	22 3				
C22235	1.40FS	7*	3 11	8 89	6 12	10 1	8 59	10 9	21 8				
C22236	1.60FS	10	3 5	11 95	9 10	8 5	9 49	16 16	30 16				
C22237	1.60S	16	8 11	18 89	- 0	11 <1	8 4	7 19	38 65				
SPRINGFIELD (NO. 5) COAL (Southern Illinois)													
C22173	channel [†]	9*	6 17	10 83	9 7	10 <1	7 37	13 17	25 22				
C22238	1.25F	6	4 13	7 87	6 23	3 <1	6 42	16 15	23 7				
C22239	1.30FS	6	3 13	7 87	6 15	- 0	7 62	14 10	- 0				
C22240	1.35FS	8*	4 8	9 92	9 11	9 1	8 61	17 16	44 3				
C22241	1.40FS	6	4 18	7 82	6 14	12 1	7 52	12 13	36 2				
C22242	1.60FS	8	4 12	9 88	6 4	7 1	7 34	11 41	24 8				
C22243	1.60S	13	7 15	16 85	10 1	- 0	10 12	17 69	26 4				
SPRINGFIELD (NO. 5) COAL (Central Illinois)													
C22419	channel	11*	7 13	12 87	9 8	15 1	9 46	16 10	32 23				
C22458	3/8"x30m	11*	9 13	12 87	10 9	8 2	9 41	12 9	25 26				
C22422	1.25F	8*	5 13	9 87	8 20	4 1	9 53	12 10	19 3				
C22423	1.31FS	8*	5 10	9 90	8 12	10 <1	9 64 ^a	13 11	19 3				
C22424	1.35FS	10*	4 4	10 96	11 12	5 <1	19 65 ^a	14 12	23 7				
C22425	1.40FS	12*	6 8	13 92	10 7	7 <1	10 45	18 23	30 18				
C22426	1.60FS	14*	8 14	16 86	13 14	20 1	11 34	17 22	34 26				
C22427	1.80FS	17*	11 13	18 87	8 1	12 1	11 22	14 23	39 40				
C22428	1.80S	21*	20 34	22 66	19 <1	15 6	13 6	12 5	28 50				
C22457	-30x200m	9	6 9	10 91	7 11	10 2	8 42 ^a	12 21	30 14				
C22429	1.25F	6	3 20	7 80	7 36	6 4	7 35 ^a	7 2	16 4				
C22430	1.31FS	8	4 8	9 92	8 35	8 2	8 44	9 2	23 9				
C22431	1.35FS	9	4 9	10 91	8 25	7 2	9 48	12 3	24 14				
C22432	1.40FS	10	4 7	11 93	10 26	9 2	10 51	14 4	34 10				
C22433	1.60FS	17	14 6	17 94	13 10	13 4	11 34	25 17	35 30				
C24434	1.80FS	25	21 7	25 93	20 2	15 7	12 9	20 14	35 62				
C24435	1.80S	30	34 28	29 72	16 1	14 3	13 2	14 4	36 61				
C22459	-200m	13	13 44	13 56	8 6	8 2	7 9	7 4	23 35				
C22420	washed	12	14 16	12 84	11 16	8 1	10 40	12 11	36 16				

[†]The float-sink samples in this set consisted of the 20x30m fraction of this sample.

*1000 or more grains measured

^aIncludes <1% of grains enclosed in liptite

^bThe mean diameter (d) is given in micrometers (um) and the percentage (%) is based on the sum of the diameters of all grains measured.

PYRITE CHARACTERIZATION

Date:
 Sample Number: M22419.P
 Magnification factor: 2.40

Comments:

MERGED PSTORE FILES C22419.P, D22419.P AND E22419.P.

PJD 2/27/84

	PYRITE			PYRITE CLEANABILITY INDEX
	FREE	ENCLOSED	TOTAL	$\frac{P_f + P_p + P_c}{P_v + P_i + P_l + P_t}$
GRAINS	323	1177	1500	COUNT
%	21.53	78.47	100.00	0.57
DIAMTR	2201	14400	16601	DIAMETER
%	13.26	86.74	100.00	0.85
mean	6.81	12.23	11.07	
s.d.	8.36	10.63	10.42	
AREA	29431	242629	272061	AREA
%	10.82	89.18	100.00	1.88
mean	91.12	206.14	181.37	
s.d.	637.92	470.88	513.42	
VOLUME	1114000	5564120	6678120	VOLUME
%	16.68	83.32	100.00	4.91
mean	3448.93	4727.37	4452.08	
s.d.	50390.10	20634.60	29660.60	

MACERAL-MINERAL ASSOCIATION OF ENCLOSED PYRITE

	V	I	L	T	C	P
GRAINS	140	7	0	807	106	117
%	9.33	0.47	0.00	53.80	7.07	7.80
DIAMTR	1274	106	0	7596	1680	3744
%	7.68	0.64	0.00	45.76	10.12	22.55
mean	9.10	15.09	0.00	9.41	15.85	32.00
s.d.	6.72	6.31	0.00	6.00	9.77	16.70
AREA	14040	1438	0	78925	28776	119452
%	5.16	0.53	0.00	29.01	10.58	43.91
mean	100.28	205.41	0.00	97.80	271.47	1020.96
s.d.	180.93	151.54	0.00	141.40	346.00	1091.52
VOLUME	188897	18255	0	923728	528004	3905240
%	2.83	0.27	0.00	13.83	7.91	58.48
mean	1349.26	2607.84	0.00	1144.64	4981.17	33378.10
s.d.	4520.32	2545.81	0.00	2914.32	10577.70	56569.70

V = Vitrite
 I = Inertite
 L = Liptite
 T = Trimacerite and Bimacerite
 C = Carbominerite
 P = 20% or more Pyrite

Figure 9 Example of output from PCHAR program.

These and other results (table 5) are compilations of the diameter values from the report of pyrite characterization on each sample, an output report from the PCHAR program (fig. 9). Note that results are expressed in terms of numbers of grains, diameter, area, and volume, the latter two computed from the diameter measurements.

Pyrite size distribution

The size distribution of pyrite grains was determined with the computer program SIZEFREQ. As this is a logarithmic scale, the categories at the small end of the distribution have only one possible measured size per category, while those in the middle of the range (16 μm to 126 μm) have several possible measured sizes. Because of the low number of counts, no subcategories were distinguished for grains above 125 μm , although this could have been useful for some of the sink fractions.

The range of grain size in the mine run and channel samples (feed samples) is very large (fig. 10), and is widest in the 1.6 or 1.8 sink fractions (fig. 11). In the feed samples, 0.5 percent or less of the grains were over 64 μm in diameter. However, these large grains contribute a much greater proportion to the total weight of pyrite. The median grain size is defined as the size intercept off the cumulative curve at the 50 percent level. The size distribution of pyrites in the feed and float-sink samples may be compared on the basis of their cumulative size curves (fig. 11). The lightest float fraction consistently has the lowest proportion of the larger grains, expected, and the sink fraction has the largest proportion of the larger sulfide grains. The trend shown by the four sets of curves is quite consistent; there is a progressive increase in median value with increasing density (fig. 11). The curves for float-sink fractions of a feed coal tend to parallel each other in the middle range of recoveries. The heavier fractions are generally depleted in the finest pyrite grains, as exhibited most pronouncedly by the two portions of C22419 (fig. 11c and d).

The size distribution of pyrite grains reported here can be compared with results on Illinois Basin coals reported by others. McCartney, O'Donnell, and Ergun (1969) analyzed eight samples of the Herrin and Springfield Coals and reported mean pyrite sizes from 29 to 90 μm . Our values are significantly smaller and we suggest the difference is due to differences in the magnification used in the two studies: they used a magnification of 375x whereas we used 500x. It is likely that we distinguished individual grains among aggregates of grains at 500x. It should be noted that unpublished data generated by scanning electron microscope methods confirm that abundant crystallites much less than 2 μm in diameter occur in coal. Such ultra small crystallites could not be accurately measured even at the 500x we used. It is clear that the absolute mean size of grains is smaller as the magnification is higher. Nonetheless, the combined weight of the submicrometer grains appears to be insignificant in terms of weight percent as reported by standard chemical analyses.

Frankie and Hower (1984) used about the same magnification as reported here, and they recorded similar size ranges for pyrite as reported here. They classified grains according to various types of morphologic occurrences (i.e., euhedral, framboidal, dendritic) as well as various microlithotype associations. Their association scheme is similar to the present study, and to the extent that comparisons can be made, our results are quite similar.

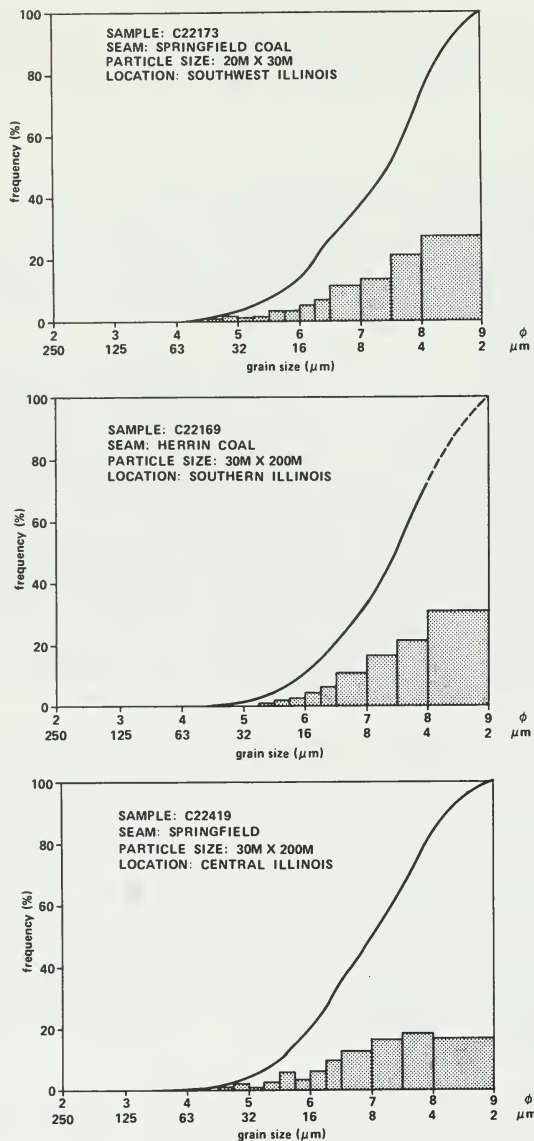


Figure 10 Distribution of pyrite diameters and the cumulative distribution curve for the three feed samples.

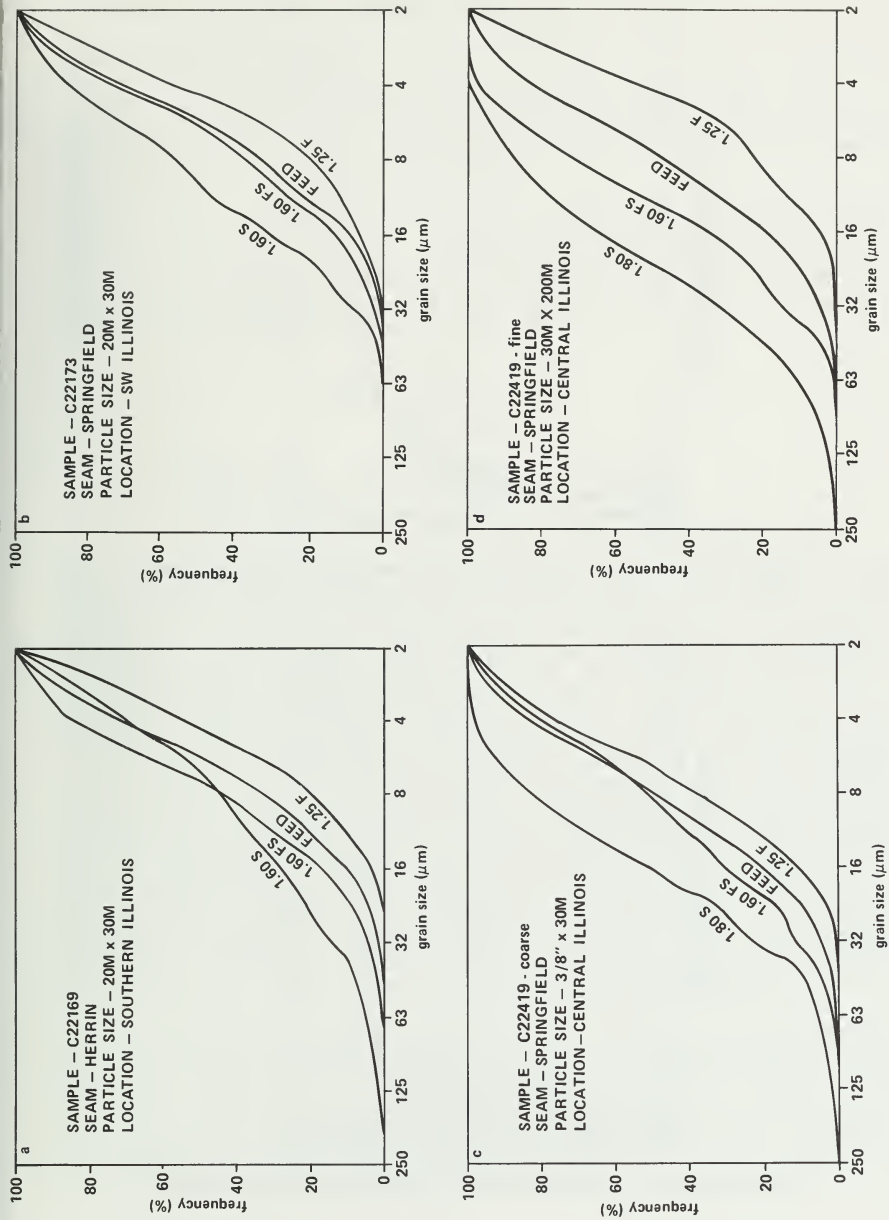


Figure 11 Comparison of the size distribution in various density fractions in feed and crushed samples.

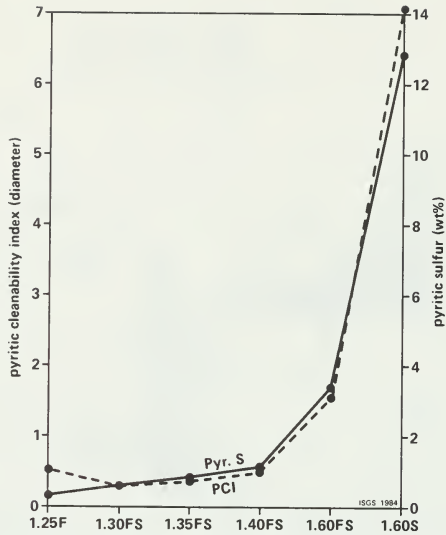


Figure 12 PCI and pyritic sulfur content for float-sink set C22238-C22243 (feed samples C22173).

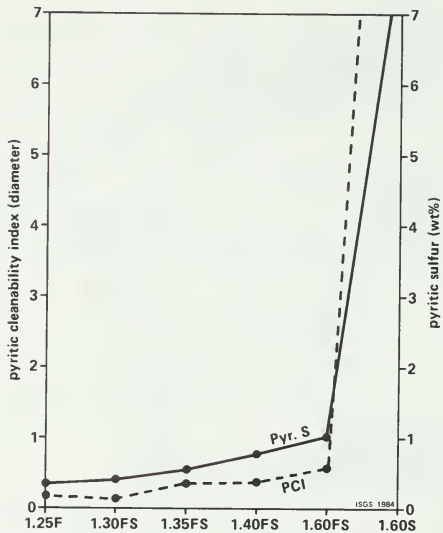


Figure 13 PCI and pyritic sulfur content for float-sink set C22232-C22237 (feed sample C22169).

EVALUATION OF RESULTS FOR PYRITE CLEANABILITY

Pyrite Cleanability Index

The pyrite size and association data can be used to generate an index that reflects the potential cleanability of the sample tested. The pyrite cleanability index (PCI) is calculated by dividing a measure of those grains that are potentially easy to remove from the coal by a measure of those grains that are difficult to remove from the coal.

The pyrite grains judged easy to remove are free grains and grains enclosed in carbominerite or pyritic coal. Pyrite grains judged hard to remove are those enclosed in vitrite, inertite, liptite, or bi- and trimacerites. The ratio of these two groups of associations, each expressed as the total percentage of grain-characteristic in each association of the group, yields a value here defined as the pyrite cleanability index (PCI):

$$PCI = \frac{P_f + P_p + P_c}{P_v + P_i + P_l + P_t}$$

P = pyrite grain characteristic

f = free grain

p = $\geq 20\%$ pyrite (pyritic coal)

c = carbominerite

v = vitrite

i = inertite

l = liptite

t = tri- and bimacerites

Note that the classification of pyrite grains as to ease of removal is largely density based, with the lower density and more maceral-rich particles in the denominator and the higher density and more pyrite-rich particles in the numerator. In this study we have expressed our measurements in terms of grain by count (number), diameter, area, and volume (fig. 9), so the PCI can be expressed in one or more of these measures, PCI_n (by number), PCI_d (by diameter), or PCI_v (by volume). Comparison of the ratios determined for the float-sink sets reveals a relatively wide range for each of the four measures, and the relative ranking between the samples changes for only a few samples in a set for each measure. However, both of the extreme measures PCI_n and PCI_v , are less desirable to use. PCI_n tends to overemphasize the significance of the many tiny grains that are of low mass. The PCI_v , on the other hand, overemphasizes the largest grains that have the highest mass and are measured with the least accuracy. The large grains also are the easiest to reject by commonly used methods of coal preparation. For these reasons and because diameter data are commonly used in minerals preparation research, the PCI_d (measured by diameter) was chosen as the best measure for the project samples (table 6). In the table and discussion that follows we drop the subscript d from the PCI term, but it always carries the meaning of diameter.

A plot of PCI and corresponding pyritic sulfur values for a float-sink set shows a close match through all the middle range of specific gravity values (fig. 12). Only the sink fraction shows a poor match, and this may be due to sample bias rather than to the method of calculation of PCI. Similar plots of the other float-sink sets confirmed this correlation (fig. 13). It appears that the PCI will be a useful value to compare petrographic and chemical data for samples from various coal preparation methods, and may well have predictive value when enough data are assembled on feed coals.

Based on the limited group of high-sulfur coal samples studied here, the general characteristics of the PCI are as follows: uncleaned coal of typical size distribution, as well as refuse material, generally have PCI values above 1.0; partly cleaned or inefficiently cleaned coal and coal relatively clean to begin with generally fall in the 0.33 to 1.0 PCI range; well-cleaned coal generally has a PCI value of 0.33 or below. The PCI is partly a function of the particle size of the coal. If we start with the same feed sample and crush representative splits of it to progressively fine top sizes, the PCI should increase as the top particle size decreases because of the progressive liberation of pyrite. In theory, the PCI would approach infinity if the coal could be crushed fine enough. The actual point(s) at which the PCI curve changes slope during grinding will be the subject of future research.

The PCI of the feed samples ranges from 0.65 to 1.24, and each feed sample has a PCI intermediate between that of its low and high density fractions.

Table 6 Pyrite cleanability index according to grain diameter.

Sample No.	Sample Type	PCI
C22169	Mine run	0.65
C22232	1.25F	0.18
C22233	1.30FS	0.12
C22234	1.35FS	0.37
C22235	1.40FS	0.38
C22236	1.60FS	0.59
C22237	1.60S	20.9
C22173	Channel	1.24
C22238	1.25F	0.52
C22239	1.30FS	0.30
C22240	1.35FS	0.36
C22241	1.40FS	0.50
C22242	1.60FS	1.56
C22243	1.60S	7.08
C22419	Channel	0.85
C22458	3/8"x30M	0.92
C22422	1.25F	0.35
C22423	1.31FS	0.32
C22424	1.35FS	0.29
C22425	1.40FS	0.93
C22426	1.60FS	1.59
C22427	1.80FS	3.17
C22428	1.80S	7.49
C22457	-30Mx200M	0.80
C22429	1.25F	0.34
C22430	1.31FS	0.22
C22431	1.35FS	0.33
C22432	1.40FS	0.26
C22433	1.60FS	1.10
C22434	1.80FS	4.63
C22435	1.80S	16.5
C22459	-200x0	4.85
C22420	washed	.75

This trend of the PCI suggests that this means of characterizing coals may be useful for comparing the washability of pyrites in coals.

Another aspect of the PCI which bears on its use is its independence of actual percentage of pyritic sulfur. It is determined from the physical proportions of sulfide grains in specific maceral-mineral associations observed by microscopic methods. It should be noted that the same PCI number could result for two samples with quite different abundances of pyrite. Therefore, the PCI can best be used to compare treated splits and cleaned fractions from the same parent sample. Nonetheless, comparison of PCI for the two feed samples of comparable particle size (30 X 20 M) shows differences that correlate with the float-sink cleanability of the raw samples:

Sample	PCI	Float-sink behavior
C22169	0.65	cleanable (fig. 1a)
C22173	1.24	more cleanable (fig. 1b)

While only two of the project samples have been compared, the PCI does vary with the general float-sink behavior--the higher PCI value corresponds to a more cleanable feed coal (C22173), the lower value to a less cleanable sample (C22169).

On the basis of the limited data generated in this study, the PCI reflects the trend of pyritic sulfur reduction exhibited in washability tests. However, much more data will be needed before prediction of washability behavior from a single PCI is possible.

CONCLUSIONS AND RECOMMENDATIONS

The size of pyrite grains in coal was characterized on the basis of their diameter (Martin's Statistical Diameter) with a reflecting light microscope equipped with an oil immersion lens. A minimum of 1000 grains had to be measured to obtain a mean diameter that was characteristic of the sample. The maceral-mineral association of each pyrite measured was classified according to one of seven different associations (modified microlithotypes): vitrite, inertite, liptite, bi- and trimacerite, carbominerite, pyritic coal, and free pyrite grains. The grain's association was determined by visually estimating the composition (area percentage) of the entire particle of coal enclosing the measured grain. This method was used to characterize three feed samples and six to seven density fractions of each. The results provided the following observations.

- The mean diameter of pyrite grains in feed samples varies from 8 to 11 μm , but the grains vary in diameter from less than 1 μm to more than 200 μm . The largest grains are associated with other such grains in pyritic coal particles, which are most abundant in high density particles.

- Free grains average smaller than others in the same sample. Only one sample departed from this trend--the 1.8 sink of a finely crushed feed sample. The free grains are thought to have been crushed products of cleat-fill or nodular pyrite forms in the coal seam. Evidently these forms are relatively easy to crush into their constituent grains, mostly 10 μm or smaller. However, free pyrites comprise only 9 to 17 percent of all pyrites in the feed samples.
- Pyrites are most abundant in bi- and trimacerite associations in all samples except for the heavy fractions (>1.40 g/cc), wherein carbominerite and pyritic coal types predominate. The mean diameter of grains in trimacerite particles in feed samples ranges only from 7 to 9 μm ; on the other hand, those in carbominerite and pyritic particles range from 10 to 32 μm .
- The characteristic found most useful for evaluating the float-sink behavior of coal was the summed diameter of grains in two groups of associations: (1) free, carbominerite, pyritic coal; and (2) vitrite, inertite, liptite, and bi- and trimacerites. The ratio of the summed diameters of grains with these two groups of associations gives a value defined here as the pyritic cleanability index (PCI). It is proposed that the PCI provides a measure of the coal's potential for pyrite removal.

It is recommended that further work be applied in three aspects: (1) improvements in specimen preparation and/or counting procedures need to be made to increase the correlation between the petrographically determined pyrite content and the chemically determined pyritic sulfur content; (2) many additional samples need to be tested for their PCI and this index compared with results of various pyrite removal processes; and (3) a more rapid method of measuring the size and maceral association of pyrites in coal needs to be developed. About 9 hours are required to complete the microscopic and computer entry tasks for each sample. Computer controlled image analysis of microscopic specimens might well enable the PCI to be determined in much less time.

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APPENDIX. Computer programs: PSTORE, PCHAR, and SIZEFREQ

```
'Program PSTORE
```

```
'AUTHOR: Ernie Colantonio
```

```
'WRITTEN: September 9, 1983
```

```
'LANGUAGE: Microsoft BASIC-80 (compiler)
```

```
'DESCRIPTION:
```

```
' This program gathers sample pyrite grain data from the user
' and stores it in a specified format in a file. It queries the
' user for the sample number and date, and allows the entry of the
' number of ocular divisions for each of up to 500 grains in a
' sample. For each grain, the user must indicate if it is free or
' enclosed, and if it is enclosed, the user must enter an additional
' association code letter. PSTORE presents the raw data of the
' last 6 grains entered and allows the user to go back and change
' any previous entry. After all the grain data has been entered,
' the user can enter up to 3 lines of comments to be stored with
' the data. Upon program completion, the data is written to an
' output file named by the sample number with the extension ".P".
```

```
' MODIFICATIONS: Sally Lincoln January, 1984
```

```
' Made the program more user-friendly by speeding up
' data entry and extensively checking for errors. No
' invalid flags should appear in the output file now.
```

```
'INPUT:
```

- from user
- magnification factor
- sample number
- date
- number of ocular divisions for each grain
- "F" for free or "E" for enclosed for each grain
- if enclosed, one additional association code letter
- up to 3 lines of comments

```
'OUTPUT:
```

- to file "sample#.P"
- line 1
 - cols 1-10 - sample number
 - cols 11-20 - date
 - cols 21-23 - number of grains in the sample
 - cols 26-30 - magnification factor (### format)
- lines 2 - 4
 - cols 1-80 - comments
- remaining (up to 50) lines
 - 10 to a line:
 - number of ocular divisions (### format), "F" or "E",
 additional letter if enclosed, 1 space
 - For example:

10F	10EV	20F	100ET	15F	20EV	30F	40F	25ET	100F
-----	------	-----	-------	-----	------	-----	-----	------	------

```
'VARIABLES:
'
'DIVISION% - array of number of ocular divisions for each grain
'TYPE$ - array of flags "F" for free, "E" for enclosed
'ASSOCIATION$ - array of association codes
'DATE$ - date
'SAMPLE$ - sample number
'MAGFACT - magnification factor
'N% - number of grains
'VIEW% - starting index of data to be presented at CRT
'I% - loop control variable
'TYPEOUT$ - translation of TYPE$ element
'ENTRY$ - holds user response
'CHANGE% - holds index of data to be re-entered
'FILE$ - output file name
'
```

```
.....
'Introduction
```

```
PRINT
PRINT "Program PSTORE"
PRINT "          - gather and store pyrite grain data"
PRINT
INPUT "Magnification factor"; MAGFACT
PRINT
INPUT "Sample number"; SAMPLE$
INPUT "          Date"; DATE$
PRINT
PRINT "Enter the number of ocular divisions and whether";
PRINT " free or enclosed."
PRINT "If enclosed, enter additional association code letter."
PRINT "Up to 500 grains may be entered for this sample."
PRINT
PRINT "Answer with 'S' at any time (except while editing)";
PRINT " to stop entering data."
PRINT
```

```
.....
'Initializations
```

```
OPTION BASE 1
DIM DIVISION%(500), TYPE$(500), ASSOCIATION$(500), COMMENTS(3)
N% = 1
```

```
.....
'Data entry and display loop
```

```
100PRINT
PRINT "Answer with 'C' at any time to change a previous entry."
PRINT
IF N% = 1 GOTO 200
```

```

IF N% < 8 THEN VIEW% = 1 ELSE VIEW% = N% - 6
FOR I% = VIEW% TO (VIEW% + 5)
  TYPEOUT$ = "No Entry"
  IF TYPE$(I%) = "E" THEN TYPEOUT$ = "Enclosed"
  IF TYPE$(I%) = "F" THEN TYPEOUT$ = "Free"
  PRINT " (";
  PRINT USING "###"; I%;
  PRINT ") d' = ";
  PRINT USING "###"; DIVISION%(I%);
  PRINT " ";TYPEOUT$;
  PRINT TAB(50); ASSOCIATION$(I%)
NEXT I%

200PRINT
PRINT "Grain (";
PRINT USING "###"; N%;
PRINT ")? ";
INPUT; "Divisions"; ENTRY$
E$ = LEFT$(ENTRY$,1)
IF E$ = "C" GOTO 1000
IF E$ = "S" GOTO 2000
DIVISION%(N%) = VAL(ENTRY$)
PRINT
250INPUT " Enter E + V,I,L,P,C,T or just F"; ENTRY$
E$ = LEFT$(ENTRY$,1)
IF E$ = "C" GOTO 1000
IF E$ = "S" GOTO 2000

TYPE$(N%) = LEFT$(ENTRY$,1)
IF TYPE$(N%) = "F" GOTO 340
IF (TYPE$(N%) = "E") AND (LEN(ENTRY$) <> 1) GOTO 300
PRINT
PRINT CHR$(7)
PRINT "*** Incorrect entry. Try again."
GOTO 250

300ASSOCIATION%(N%) = MID$(ENTRY$,2,1)
IF INSTR("V,I,L,P,C,T", ASSOCIATION%(N%)) <> 0 GOTO 350
PRINT
PRINT CHR$(7)
PRINT "*** Incorrect entry. Try again."
GOTO 250

340ASSOCIATION%(N%) = " "
350PRINT

N% = N% + 1
IF N% > 501 GOTO 2000
IF N% < 501 GOTO 100
PRINT
PRINT CHR$(7)
PRINT CHR$(7)
PRINT "*** 500 GRAIN LIMIT. Change previous entry, if "
PRINT "needed. Then stop (S). Else data will be lost."
PRINT
GOTO 100

```

.....

'Handle the changing of a previous entry, displaying changed
'entry up to last entry at the CRT.

```

1000PRINT
INPUT; "Grain Number"; CHANGE%
IF (CHANGE% >= 1) AND (CHANGE% <= 500) GOTO 1010
  PRINT CHR$(7)
  PRINT
  PRINT "**** Invalid Grain Number. Try again."
  GOTO 1000
1010INPUT; "  Divisions"; DIVISION%(CHANGE%)
1020INPUT "    Enter E + V,I,L,P,C,T or just F"; ENTRY$
TYPE$(CHANGE%) = LEFT$(ENTRY$,1)
IF TYPE$(CHANGE%) = "F" GOTO 1040
IF (TYPE$(CHANGE%) = "E") AND (LEN(ENTRY$) > 1) GOTO 1030
  PRINT
  PRINT CHR$(7)
  PRINT "**** Invalid entry. Try again."
  GOTO 1020

1030ASSOCIATION$(CHANGE%) = MID$(ENTRY$,2,1)
IF INSTR("V,I,L,P,C,T", ASSOCIATION$(CHANGE%)) <> 0 GOTO 1050
  PRINT
  PRINT CHR$(7)
  PRINT "**** Invalid entry. Try again."
  GOTO 1020
1040ASSOCIATION$(CHANGE%) = " "
1050PRINT
PRINT
FOR I% = CHANGE% TO (N% - 1)
  IF TYPE$(I%) = "E" THEN TYPEOUT$ = "Enclosed"
  IF TYPE$(I%) = "F" THEN TYPEOUT$ = "Free"
  PRINT "  (";
  PRINT USING "###"; I%;
  PRINT ")    d' = ";
  PRINT USING "###"; DIVISION%(I%);
  PRINT "    "; TYPEOUT$;
  PRINT TAB(50); ASSOCIATIONS$(I%)
NEXT I%
GOTO 200

```

.....

2000'Finished entering grains, gather comments and write to file

N% = N% - 1

```

PRINT
PRINT "Enter up to 3 lines of comments - if none, hit RETURN."
PRINT
FOR I% = 1 TO 3
  LINE INPUT "?"; COMMENTS$(I%)
NEXT I%

```

PRINT

'Write out raw data

FILE\$ = SAMPLE\$ + ".P"

OPEN "O", #1, FILE\$

PRINT #1, LEFT\$(SAMPLE\$ + SPACE\$(10), 10);

PRINT #1, LEFT\$(DATE\$ + SPACE\$(10), 10);

PRINT #1, USING "###"; N%;

PRINT #1, USING "####.##"; MAGFACT

FOR I% = 1 TO 3

PRINT #1, LEFT\$(COMMENT\$(I%), 80)

NEXT I%

FOR I% = 1 TO N%

PRINT #1, USING "###"; DIVISION%(I%);

PRINT #1, TYPE\$(I%); ASSOCIATIONS\$(I%); " ";

IF (I% MOD 10) = 0 THEN PRINT #1,

NEXT I%

.....

'Exit program

PRINT

PRINT "Data for sample ";SAMPLE\$;" has been written to file ";

PRINT "";FILE\$;"".

PRINT

PRINT "Exit program PSTORE."

PRINT

END

'Program PCHAR

'AUTHOR: Ernie Colantonio

'WRITTEN: September 21, 1983

'LANGUAGE: Microsoft BASIC-80 (compiler)

'DESCRIPTION:

' This program gathers data from a file created by program
' PSTORE and computes and reports pyrite characterization summary
' statistics. Subtotals, percentages, means and standard deviations
' are computed for free, enclosed and total pyrite grains as well
' as for vitrite, inertite, liptite, trimacerite, carbominerite
' and 50% or more pyrite maceral-mineral associations. All
' information is output directly to the printer as a formatted
' report.

'MODIFICATIONS:

' S. Lincoln January, 1984

- ' - The word LENGTH in the output table was changed to DIAMTR.
' Therefore whenever length is referred to in this program, in
' commenting or variables, actually "diameter" is meant.
- ' - The percentages under DIAMTR, AREA, and VOLUME now total
' to the ENCLOSED percentage for each of the above categories,
' instead of to 100%.
- ' - The length constant LENGTHC was changed from 1.26 to
' 1.00 so that it would not have an effect on the calculations;
' but, it was not removed because it may be used at a later date.

' S. Lincoln September, 1984

- ' - A routine was written to calculate the Pyrite Cleanability
' Index for each of 4 categories: grain count, diameter, area, and
' volume. A formula specified by PJD was followed. Then changes
' were made to print the values in the final report.

'INPUT:

- ' - from user
- ' - filename of data produced by program PSTORE
- ' - from PSTORE output file
- ' - line 1
 - ' - cols 1-10 - sample number
 - ' - cols 11-20 - date
 - ' - cols 21-23 - number of grains in the sample
 - ' - cols 26-30 - magnification factor
- ' - lines 2 - 4
 - ' - cols 1-80 - comments
- ' - remaining (up to 50) lines


```

' - 10 to a line:
'   number of ocular divisions (### format), "F" or "E",
'   additional letter if enclosed, 1 space
'   For example:
'     10F  10EV 20F  100ET 15F  20EV 30F  40F  25ET 100F

'OUTPUT:
' - Pyrite Characterization Report directly to printer

'SUBROUTINES:
'INITIALIZATIONS - dimensions arrays & initializes variables
'GATHER-DATA     - gets data from input file
'COMPUTE-TOTALS  - computes necessary subtotals from input data
'COMPUTE-STATISTICS - computes pyrite characterization statistics
'COMPUTE-PCI     - computes Pyrite Cleanability Indices
'                 (see MODIFICATIONS for September, 1984)
'PRINT-REPORT   - outputs report directly to printer

'VARIABLES:
'COMMENTS$      - array of 3 comment lines
'ENTRIES$       - array of data entries for grains from input file
'NASSOC%        - how many associations there are
'ASSOCIATION$   - array of the association names
'ACODE$         - array of the 1-letter association codes
'FILE$          - name of input file
'LINE$         - entire line from input file
'SAMPLE$        - sample number
'DATE$          - date
'MAGFACT        - magnification factor
'               NSTORED% - number of grain entries in input file
'I%, J%, K%     - loop control variables
'NREAD%         - number of entries read from input file
'GTYPES$       - pyrite grain type code (F or E)
'ASSOC$        - association type code letter
'ANSWERS$      - user response
'RFORMAT$      - real number output format
'IFORMAT$      - integer number output format
'TOTAL         - temporary variable for computing percentages
'GRAINS        - 2 dimensional real array (9,2)
'               dimension 1, subscripts 1-9
'               - FREE, ENCLOSED, TOTAL, VITRITE, INERTITE,
'               LIPTITE, TRIMACERITE, CARBOMINERITE, 50%
'               or more PYRITE
'               dimension 2, subscripts 1-2
'               - grain count, grain percentage
'SIZE          - 3 dimensional real array (3,9,4)
'               dimension 1, subscripts 1-3
'               - DIAMTR, AREA, VOLUME
'               dimension 2, subscripts 1-9
'               - FREE, ENCLOSED, TOTAL, VITRITE, INERTITE,
'               LIPTITE, TRIMACERITE, CARBOMINERITE, 50%
'               or more PYRITE

```

```

'          dimension 3, subscripts 1-4
'          - sum total, percentage, mean, standard deviation
'ONESIZE   - 1 dimensional real array (3)
'          dimension 1, subscripts 1-3
'          - computed length, area and volume
'LABEL$    - 2 dimensional string array
'          report output labels
'LENGTHC   - length (diameter) computation constant =
'          cross sectional factor (1.00) *
'          magnification factor (MAGFACT)
'AREAC     - area computation constant = pi / 4
'VOLUMEC   - volume computation constant = pi / 6
'COUNTPCI - Pyrite Cleanability Index (PCI) for grain count
'PCI       - PCI for diameter, area, and volume only
'PCILABEL$ - array of labels for values in variable PCI above
'TEMP      - temporary variable

```

```

.....
'Main Program

```

```

PRINT
PRINT "Program PCHAR"
PRINT "          - Pyrite Characterization Report Program"
PRINT

```

```

GOSUB 1000 'INITIALIZATIONS
GOSUB 2000 'GATHER-DATA
PRINT "Data successfully loaded into memory."
GOSUB 3000 'COMPUTE-TOTALS
PRINT "Subtotals successfully computed."
GOSUB 4000 'COMPUTE-STATISTICS
PRINT "Statistics successfully computed."
GOSUB 5000 'COMPUTE PYRITE CLEANABILITY INDECES
GOSUB 6000 'PRINT-REPORT

```

```

PRINT
PRINT "Exit program PCHAR"
PRINT

```

```

END

```

```

.....
1000'Subroutine INITIALIZATIONS

```

```

'This routine does the array dimensioning and variable
'initializations.

```

```

OPTION BASE 1

```

```

DIM COMMENTS$(3)
DIM ENTRY$(2000)

```

```

NASSOC% = 6
DIM ASSOCIATIONS$(6)
ASSOCIATIONS$(1) = "Vitrite           "
ASSOCIATIONS$(2) = "Inertite           "
ASSOCIATIONS$(3) = "Liptite           "
ASSOCIATIONS$(4) = "Trimacerite and Bimacerite"
ASSOCIATIONS$(5) = "Carbominerite    "
ASSOCIATIONS$(6) = "20% or more Pyrite  "
DIM ACODE$(6)
ACODE$(1) = "V"
ACODE$(2) = "I"
ACODE$(3) = "L"
ACODE$(4) = "T"
ACODE$(5) = "C"
ACODE$(6) = "P"

DIM GRAINS(9,2)
DIM SIZE(3,9,4)
DIM ONESIZE(3)

DIM PCI(3)

DIM PCILABEL$(3)
PCILABEL$(1) = "DIAMETER"
PCILABEL$(2) = "AREA"
PCILABEL$(3) = "VOLUME"

DIM LABEL$(3,2)
LABEL$(1,1) = "DIAMTR"
LABEL$(2,1) = " AREA"
LABEL$(3,1) = "VOLUME"
LABEL$(1,2) = " %"
LABEL$(2,2) = " mean"
LABEL$(3,2) = " s.d."

FOR I% = 1 TO 3
  FOR J% = 1 TO 9
    FOR K% = 1 TO 4
      SIZE(I%,J%,K%) = 0
    NEXT K%
  NEXT J%
NEXT I%

LENGTHC = 1.00
AREAC = 0.785
VOLUMEC = 0.5236

RETURN

```

```

.....
2000'Subroutine GATHER-DATA

```

```

'This routine gathers the data from the input file.

```

```

2010PRINT
INPUT "Enter filename. Use the correct extension->",FILE$
PRINT
PRINT "You want to use file ";FILE$;" in PCHAR."
INPUT "Correct (Y/N)";ANSWER$
ANSWER$ = LEFT$(ANSWER$,1)
IF ANSWER$ <> "N" GOTO 2020
  PRINT "I will ask until you answer 'Y'."
  GOTO 2010
2020OPEN "I", #1, FILE$

```

```

LINE INPUT #1, LINE$
SAMPLE$ = LEFT$(LINE$, 10)
DATE$ = MID$(LINE$, 11, 8)
NSTORED% = VAL(MID$(LINE$, 20, 4))
MAGFACT = VAL(MID$(LINE$, 26, 6))

```

```

FOR I% = 1 TO 3
  LINE INPUT #1, COMMENT$(I%)
NEXT I%

```

```

NREAD% = 1
WHILE NOT EOF(1)
  LINE INPUT #1, LINE$
  FOR I% = 1 TO 10
    ENTRY$(NREAD%) = MID$(LINE$, 6*I%-5, 5)
    IF NREAD% = NSTORED% GOTO 1100
    NREAD% = NREAD% + 1
  NEXT I%
WEND

```

```
1100RETURN
```

```
.....
```

```
3000'Subroutine COMPUTE-TOTALS
```

```
'This routine computes the necessary subtotals.
```

```
LENGTHC = LENGTHC * MAGFACT
```

```
FOR I% = 1 TO NREAD%
```

```
' store whether FREE or ENCLOSED
GTYPE$ = MID$(ENTRY$(I%), 4, 1)
' store which association
ASSOC$ = RIGHT$(ENTRY$(I%), 1)

```

```
' compute length, area and volume
```

```

ONESIZE(1) = VAL(LEFT$(ENTRY$(I%),3)) * LENGTHC
ONESIZE(2) = ONESIZE(1) 2 * AREAC
ONESIZE(3) = ONESIZE(1) 3 * VOLUME

```

```

IF GTYPE$ <> "F" GOTO 2100
  increment number of free grains
  GRAINS(1,1) = GRAINS(1,1) + 1
  FOR J% = 1 TO 3
    increment free grain length, area and volume
    SIZE(J%,1,1) = SIZE(J%,1,1) + ONESIZE(J%)
    increment length, area, volume squared
    SIZE(J%,1,4) = SIZE(J%,1,4) + ONESIZE(J%)  2
  NEXT J%
2100  'Endif

IF GTYPE$ <> "E" GOTO 2200
  FOR J% = 1 TO NASSOC%
    IF ASSOC$ <> ACODE$(J%) GOTO 2300
    increment association grain count
    GRAINS(J%+3,1) = GRAINS(J%+3,1) + 1
    FOR K% = 1 TO 3
      increment association length, area, volume
      SIZE(K%,J%+3,1) = SIZE(K%,J%+3,1) + ONESIZE(K%)
      increment association length, area, volume squared
      SIZE(K%,J%+3,4) = SIZE(K%,J%+3,4) + ONESIZE(K%)  2
    NEXT K%
2300  'Endif
  NEXT J%
2200  'Endif

NEXT I%

'sum up associations to get sums of ENCLOSED grains

FOR I% = 4 TO (NASSOC% + 3)
  GRAINS(2,1) = GRAINS(2,1) + GRAINS(I%,1)
  FOR K% = 1 TO 3
    SIZE(K%,2,1) = SIZE(K%,2,1) + SIZE(K%,I%,1)
    SIZE(K%,2,4) = SIZE(K%,2,4) + SIZE(K%,I%,4)
  NEXT K%
NEXT I%

'sum up FREE and ENCLOSED to get TOTAL

FOR I% = 1 TO 3
  SIZE(I%,3,1) = SIZE(I%,1,1) + SIZE(I%,2,1)
  SIZE(I%,3,4) = SIZE(I%,1,4) + SIZE(I%,2,4)
NEXT I%

GRAINS(3,1) = GRAINS(1,1) + GRAINS(2,1)

RETURN
.....

4000'Subroutine COMPUTE-STATISTICS

```

'This routine computes the necessary pyrite characterization
'statistics.

'Compute grain percentages

```
FOR I% = 1 TO (NASSOC% + 3)
  IF I% <= 3 THEN TOTAL = GRAINS(3,1) ELSE TOTAL = GRAINS(2,1)
  IF TOTAL = 0 GOTO 4050
  IF I% <= 3 THEN GRAINS(I%,2) = GRAINS(I%,1) / TOTAL * 100
  IF I% > 3 THEN GRAINS(I%,2) = GRAINS(I%,1) / TOTAL * GRAINS(2,2)
4050  NEXT I%
```

'Compute percentages, means and standard deviations of length,
'area and volume.

```
FOR I% = 1 TO (NASSOC% + 3)
  FOR J% = 1 TO 3
  '    compute percentages

    IF I% <= 3 THEN TOTAL = SIZE(J%,3,1) ELSE TOTAL = SIZE(J%,2,1)
    IF TOTAL = 0 GOTO 4080
    IF I% <= 3 THEN SIZE(J%,I%,2) = SIZE(J%,I%,1) / TOTAL * 100
    IF I% > 3 THEN SIZE(J%,I%,2) = SIZE(J%,I%,1) / TOTAL * SIZE(J%,2,2)

  '    compute means

4080  IF GRAINS(I%,1) = 0 GOTO 4100
    SIZE(J%,I%,3) = SIZE(J%,I%,1) / GRAINS(I%,1)

  '    compute standard deviations

    IF GRAINS(I%,1) = 1 GOTO 4100
    SIZE(J%,I%,4) = SIZE(J%,I%,4) - (SIZE(J%,I%,1) 2/GRAINS(I%,1))
    SIZE(J%,I%,4) = SQR(ABS(SIZE(J%,I%,4)/(GRAINS(I%,1)-1)))
4100  IF GRAINS(I%,1) = 1 THEN SIZE(J%,I%,4) = 0
    NEXT J%
  NEXT I%
```

RETURN

.....

5000'Subroutine COMPUTE-PCI

'This routine computes the Pyrite Cleanability indeces
'for grain count, diameter, area, and volume.

'Compute PCI based on grain count percentages.

```
TEMP = GRAINS(4,2) + GRAINS(5,2) + GRAINS(6,2) + GRAINS(7,2)
IF TEMP = 0 GOTO 5010
COUNTPCI = (GRAINS(1,2) + GRAINS(8,2) + GRAINS(9,2)) / TEMP
```

'Compute a PCI for (1) diameter, (2) area, and (3) volume.

```
5010FOR I% = 1 TO 3
      TEMP = SIZE(I%,4,2) + SIZE(I%,5,2) + SIZE(I%,6,2) + SIZE(I%,7,2)
      IF TEMP = 0 GOTO 5020
      PCI(I%) = (SIZE(I%,1,2) + SIZE(I%,8,2) + SIZE(I%,9,2)) / TEMP
5020  NEXT I%

      RETURN
```

6000'Subroutine PRINT-REPORT

'This routine outputs the pyrite characterization report
'directly to the printer

```
PRINT
PRINT CHR$(7)
PRINT "Adjust paper on printer & make sure it is 'on line'."
INPUT "OK"; ANSWER$

RFORMAT$ = "#####.##"
IFORMAT$ = "#####"

LPRINT SPC(28); "PYRITE CHARACTERIZATION"
LPRINT
LPRINT
LPRINT SPC(35); "Date: "; DATE$
LPRINT SPC(26); "Sample Number: "; FILE$
LPRINT SPC(19); "Magnification factor: ";
LPRINT USING "##.##"; MAGFACT
LPRINT
LPRINT "Comments:"
FOR I% = 1 TO 3
  LPRINT COMMENT$(I%)
NEXT I%

LPRINT
LPRINT
LPRINT SPC(24); "PYRITE";SPC(24);"PYRITE CLEANABILITY INDEX"
LPRINT SPC(60); "Pf + Pp + Pc"
LPRINT SPC(15); "FREE ENCLOSED TOTAL"; SPC(15); "Pv + Pi + Pl + Pt"
LPRINT
LPRINT "GRAINS";
LPRINT USING IFORMAT$; GRAINS(1,1); GRAINS(2,1); GRAINS(3,1);
LPRINT SPC(20); "COUNT"
LPRINT " %";
LPRINT USING RFORMAT$; GRAINS(1,2); GRAINS(2,2); GRAINS(3,2);
LPRINT SPC(13);
LPRINT USING RFORMAT$; COUNTPCI
FOR I% = 1 TO 3
  LPRINT
  LPRINT LABEL$(I%,1);
```



```

LPRINT USING IFORMAT$; SIZE(I%,1,1); SIZE(I%,2,1); SIZE(I%,3,1);
LPRINT SPC(20); PCILABEL$(I%)
FOR J% = 2 TO 4
  LPRINT LABEL$(J%-1,2);
  LPRINT USING RFORMAT$;SIZE(I%,1,J%);SIZE(I%,2,J%);SIZE(I%,3,J%);
  LPRINT SPC(13);
  IF J% = 2 THEN LPRINT USING RFORMAT$; PCI(I%) ELSE LPRINT
  NEXT J%
NEXT I%

LPRINT
LPRINT
LPRINT SPC(17); "MACERAL-MINERAL ASSOCIATION OF ENCLOSED PYRITE"
LPRINT
LPRINT SPC(17); "V"; SPC(11); "I"; SPC(11); "L";
LPRINT SPC(11); "T"; SPC(11); "C"; SPC(11); "P"
LPRINT
LPRINT "GRAINS";
FOR I% = 4 TO 9
  LPRINT USING IFORMAT$; GRAINS(I%,1);
  NEXT I%
LPRINT
LPRINT "      %";
FOR I% = 4 TO 9
  LPRINT USING RFORMAT$; GRAINS(I%,2);
  NEXT I%
LPRINT

FOR I% = 1 TO 3
  LPRINT
  LPRINT LABEL$(I%,1);
  FOR J% = 4 TO 9
    LPRINT USING IFORMAT$; SIZE(I%,J%,1);
    NEXT J%
  LPRINT
  FOR J% = 2 TO 4
    LPRINT LABEL$(J%-1,2);
    FOR K% = 4 TO 9
      LPRINT USING RFORMAT$; SIZE(I%,K%,J%);
      NEXT K%
    LPRINT
    NEXT J%
  NEXT I%

LPRINT

FOR I% = 1 TO NASSOC%
  LPRINT SPC(29); ACODE$(I%); " = "; ASSOCIATION$(I%)
  NEXT I%

LPRINT

RETURN

```

.....

```
=====
PROGRAM SIZEFREQ
```

```
AUTHOR:  Sally Lincoln      November, 1983
```

```
LANGUAGE:  Microsoft BASIC-80 (compiler)
```

```
DESCRIPTION:
```

```
'SIZEFREQ extrapolates grain diameters for a particular sample
'from a user-specified PSTORE file by reading only the rele-
'vant columns. Each diameter is multiplied by a magnification
'factor (read from the input file) to convert it to microns.
'This number is then converted to a phi unit to determine if
'the diameter fits into one the phi unit ranges. This program
'determines the number of diameters fitting into each PU range
'and prints out a table of related information (see OUTPUT).
```

```
INPUT:
```

```
'A user-specified PSTORE file. The filename has a format of
'                               Cxxxx.lll
'where x is a digit and l is a letter. Proper length of the
'filename is checked. This file is left unaltered.
```

```
OUTPUT:
```

```
'A Pyrite Grain Size Frequency table with the following columns:
```

- '1) Phi Unit ranges considered
- '2) Micrometer ranges considered
- '3) Number of diameters falling in each PU range
- '4) Percentage of column 3 to the total number
 of diameters considered
- '5) Percentage of the diameter sum in each PU range
 to the total sum of all diameters considered
- '6) Cumulative percentage of column 5 above
- '7) Bar graph describing column 5

```
'The following information is printed beneath the table:
```

- '1) The number of grain diameters qualifying
 for any Phi Unit range
 - '2) The number of grain diameters in the file
 - '3) The total sum of the grain diameters
 - '4) A description of the * symbol used in the
 bar graph. Description appears only if the
 * appears in the table. The * symbol appears
 only if there are > 10 units in the graph.
- 'note: if (1) and (2) above are not equal, this
indicates an error in the diameter data

```
SUBROUTINES:
```

- ```
'1000(INITIALIZE) - initializes constants or sets var's to 0
'2000(CONVERT) - converts the grain diameter to a phi unit
```

```

'3000(UPDATE_SUM) - finds the phi unit category in which the
' grain fits, if any, and updates a counter.
'4000(RAWPERCENT) - determines the percentage of the number of
' grains in a category to the total number.
'5000(CUMPERCENT) - determines the cumulative percentage
'6000(PRINTTABLE) - prints out the table
'
' VARIABLES:
'
'ANSWER$ - user response to a Y/N question
'CUMPERCENT - cumulative percentage of RAWPERCENTD
'DIAMETER - original diameter of a grain before conversions
'DMTRTOTAL% - total sum of all diameters falling in a PU range
'FILE$ - name of the user-specified file
'FRACTION% - # of data entries in last line of file if < 10
'LINE$ - entire line of data
'LN2 - natural log of 2
'MAGFACT - magnification factor read from 1st line of input file
'MICRANGES$ - micrometer ranges considered in program
'NUMLINES% - number of complete lines in file (10 entries/line)
'NUMUNITS% - number of units printed in the bar graph (10 max)
'PHINUM - phi unit calculated for a particular grain diameter
'PHIRANGES$ - phi unit ranges considered in program
'RANGEDMTR% - array holding diameter sum from each PU range
'RANGE% - array holding the # of diameters in each PU range
'RANGETOTAL% - total # of diameters falling in any PU range
'RAWPERCENT - percentage of the # of diameters in each RANGE%
' to the total # of diameters considered (RANGETOTAL%)
'RAWPERCENTD - percentage of the sum of the diameters in each
' PU range to the total sum of the diameters
'SUM - temporary variable helping to get cumulative percentage
'TEMP - temporary variable
'TOTGRAIN% - total # of grains in the file
'UPLIMIT% - upper limit used in FOR loop for a FRACTION.
'=====
'MAIN PROGRAM

PRINT
PRINT
PRINT "-Pyrite Grain Size Frequency Table-"
PRINT
PRINT "Please answer the initial questions. Then when the"
PRINT "table is ready to be printed, a bell will sound and"
PRINT "you will be asked for further information."
PRINT

100PRINT
PRINT "Enter the filename for which you want the table."
INPUT "Include the extension (eg Cxxxxx.LLL)->", FILE$
NUMLEN% = LEN(FILE$)
IF NUMLEN% <= 10 GOTO 150
PRINT "***Format is Cxxxxx.LLL (x=digit, L=letter)."
```

```
GOTO 100
```

```

150PRINT
PRINT
PRINT "You want a SIZEFREQ table for file ";FILE$
INPUT "Correct (Y/N)"; ANSWER$
ANSWER$ = LEFT$(ANSWER$,1)
IF ANSWER$ = "N" GOTO 100

200PRINT
PRINT "Be patient...I am working."
OPEN "I", #2, FILE$
GOSUB 1000 'Initializations

LINE INPUT #2, LINE$
TOTGRAIN% = VAL(MID$(LINE$,20,4)) 'Total # of grains in file
MAGFACT = VAL(MID$(LINE$,27,4)) 'Magnification factor in file
LINE INPUT #2, LINE$ 'Ignore 3 comment lines
LINE INPUT #2, LINE$
LINE INPUT #2, LINE$

NUMLINES% = TOTGRAIN% / 10 'Get number of complete data lines
FRACLINE% = TOTGRAIN% / MOD 10 '# of entries in unfilled last line
FOR I% = 1 TO NUMLINES%
 LINE INPUT #2, LINE$
 FOR J% = 1 TO 60 STEP 6 'Strip diameter off data entry
 DIAMETER = VAL(MID$(LINE$,J%,3))
 GOSUB 2000 'Calculate PHI UNIT of diameter
 GOSUB 3000 'Increment count in correct phi unit range
 NEXT J%
 NEXT I%

IF FRACLINE% = 0 GOTO 300 'Handle line with < 10 data entries
 LINE INPUT #2, LINE$
 UPLIMIT% = FRACLINE% * 6
 FOR I% = 1 TO UPLIMIT% STEP 6
 DIAMETER = VAL(MID$(LINE$,I%,3))
 GOSUB 2000 'Calculate phi number
 GOSUB 3000 'Increment count in phi unit range
 NEXT I%

300GOSUB 4000 'Each RANGE is what % of total # of data entries
GOSUB 5000 'Determine cumulative percentages
PRINT CHR$(7)
400PRINT
INPUT "Is lineprinter on and ready (Y/N)";ANSWER$
ANSWER$ = LEFT$(ANSWER$,1)
IF ANSWER$ = "N" GOTO 400

500GOSUB 6000 'Print table
PRINT "All finished."
END

.....
1000 'SUBROUTINE INITIALIZE

```

'This routine sets specified variables to initial values.

```

DIM RANGE%(16),RANGEDMTR%(16),PHIRANGE$(16),MICRANGE$(16)
FOR I% = 1 TO 16
 RANGE%(I%) = 0
 RANGEDMTR%(I%) = 0
NEXT I%

```

LN2 = LOG(2)

```

PHIRANGE$(1) = "0.00 - 2.00"
PHIRANGE$(2) = "2.01 - 4.00"
PHIRANGE$(3) = "4.01 - 4.25"
PHIRANGE$(4) = "4.26 - 4.50"
PHIRANGE$(5) = "4.51 - 4.75"
PHIRANGE$(6) = "4.76 - 5.00"
PHIRANGE$(7) = "5.01 - 5.25"
PHIRANGE$(8) = "5.26 - 5.50"
PHIRANGE$(9) = "5.51 - 5.75"
PHIRANGE$(10) = "5.76 - 6.00"
PHIRANGE$(11) = "6.01 - 6.25"
PHIRANGE$(12) = "6.26 - 6.50"
PHIRANGE$(13) = "6.51 - 7.00"
PHIRANGE$(14) = "7.01 - 7.50"
PHIRANGE$(15) = "7.51 - 8.00"
PHIRANGE$(16) = "8.01 - 9.00"

```

```

MICRANGE$(1) = "1000 - 250"
MICRANGE$(2) = " 250 - 63 "
MICRANGE$(3) = " 63 - 53 "
MICRANGE$(4) = " 53 - 44 "
MICRANGE$(5) = " 44 - 37 "
MICRANGE$(6) = " 37 - 31 "
MICRANGE$(7) = " 31 - 26 "
MICRANGE$(8) = " 26 - 22 "
MICRANGE$(9) = " 22 - 19 "
MICRANGE$(10) = " 19 - 16 "
MICRANGE$(11) = " 16 - 13 "
MICRANGE$(12) = " 13 - 11 "
MICRANGE$(13) = " 11 - 8 "
MICRANGE$(14) = " 8 - 6 "
MICRANGE$(15) = " 6 - 4 "
MICRANGE$(16) = " 4 - 2 "
RETURN

```

.....  
2000'SUBROUTINE CONVERT

'This routine converts the grain diameter to its phi unit.

```

TEMP = DIAMETER * MAGFACT / 1000
PHINUM = - (LOG(TEMP) / LN2) 'Use this trick to get
RETURN 'LOG BASE 2 of TEMP

```

.....

3000'SUBROUTINE UPDATE\_SUM

'This routine increments a counter in one of the 16 phi unit ranges depending on the value of the phi unit of the diameter. This determines the total number of diameters falling in each phi unit range. Also the diameters in each PU range are summed and stored for later use.

```
P = PHINUM
FLAG% = 1
IF (P >= 0.0) AND (P <= 2.00) GOTO 3050
FLAG% = 2
IF (P > 2.00) AND (P <= 4.00) GOTO 3050
FLAG% = 3
IF (P > 4.00) AND (P <= 4.25) GOTO 3050
FLAG% = 4
IF (P > 4.25) AND (P <= 4.50) GOTO 3050
FLAG% = 5
IF (P > 4.50) AND (P <= 4.75) GOTO 3050
FLAG% = 6
IF (P > 4.75) AND (P <= 5.00) GOTO 3050
FLAG% = 7
IF (P > 5.00) AND (P <= 5.25) GOTO 3050
FLAG% = 8
IF (P > 5.25) AND (P <= 5.50) GOTO 3050
FLAG% = 9
IF (P > 5.50) AND (P <= 5.75) GOTO 3050
FLAG% = 10
IF (P > 5.75) AND (P <= 6.00) GOTO 3050
FLAG% = 11
IF (P > 6.00) AND (P <= 6.25) GOTO 3050
FLAG% = 12
IF (P > 6.25) AND (P <= 6.50) GOTO 3050
FLAG% = 13
IF (P > 6.50) AND (P <= 7.00) GOTO 3050
FLAG% = 14
IF (P > 7.00) AND (P <= 7.50) GOTO 3050
FLAG% = 15
IF (P > 7.50) AND (P <= 8.00) GOTO 3050
FLAG% = 16
IF (P > 8.00) AND (P <= 9.00) GOTO 3050
GOTO 3999
```

```
3050RANGE%(FLAG%) = RANGE%(FLAG%) + 1
RANGEDMTR%(FLAG%) = RANGEDMTR%(FLAG%) + DIAMETER
3999RETURN
```

.....

4000'SUBROUTINE RAWPERCENT

'This routine determines the total # of diameters which fell into any PU range. It also figures the total sum of the diameters which fell into a PU range. It then calculates percentages based on these figures.

```
DIM RAWPERCENTC(16), RAWPERCENTD(16)
```

```

RANGETOTAL% = 0
DMTRTOTAL% = 0
FOR I% = 1 TO 16
 RANGETOTAL% = RANGETOTAL% + RANGE%(I%)
 DMTRTOTAL% = DMTRTOTAL% + RANGEDMTR%(I%)
NEXT I%

FOR I% = 1 TO 16
 RAWPERCENTC(I%) = RANGE%(I%) / RANGETOTAL% * 100
 RAWPERCENTD(I%) = RANGEDMTR%(I%) / DMTRTOTAL% * 100
NEXT I%
RETURN

```

```

.....
5000 'SUBROUTINE CUMPERCENT
' This routine calculates the cumulative percentage of
' the percentage of summed diameters. It is found from
' the progressive sum of RAWPERCENTD.

```

```

DIM CUMPERCENT(16)

```

```

TEMP = 0
FOR I% = 1 TO 16
 CUMPERCENT(I%) = TEMP + RAWPERCENTD(I%)
 TEMP = TEMP + RAWPERCENTD(I%)
NEXT I%
RETURN

```

```

.....
6000 'SUBROUTINE PRINTTABLE
' This routine prints out the table.

```

```

RFORMAT$ = "###.###" 'Format for real numbers
IFORMAT3$ = "###" 'Format for 3-digit integers
IFORMAT4$ = "####" 'Format for 4-digit integers
FLAG% = 0

```

```

LPRINT " -PYRITE GRAIN SIZE FREQUENCY-"

```

```

LPRINT
LPRINT " SAMPLE FILE ";FILES

```

```

LPRINT

```

```

LPRINT SPC(17);"MICROMETER";SPC(3);"# OF";SPC(6);"RAW";
LPRINT SPC(4);"% OF SUMMED";SPC(2);"CUM DMTR";SPC(3);"BAR"

```

```

LPRINT "PHI UNIT RANGE RANGE GRAINS PERCENT DIAMETERS PERCENT GRAPH"
LPRINT "-----"

```

```

FOR I% = 1 TO 16
 LPRINT SPC(1); PHIRANGE$(I%); SPC(4); MICRANGE$(I%); SPC(4);
 LPRINT USING IFORMAT3$; RANGE%(I%);
 LPRINT SPC(5);
 LPRINT USING RFORMAT$; RAWPERCENTC(I%);
 LPRINT SPC(5);
 LPRINT USING RFORMAT$; RAWPERCENTD(I%);
 LPRINT SPC(5);

```



```

LPRINT USING RFORMAT$; CUMPERCENT(I%);
LPRINT SPC(4);

' Bar graph
 NUMUNITS% = INT (RAWPERCENTD(I%) / 5)
 IF NUMUNITS% <= 10 GOTO 6010 'If > 10 units,
 LPRINT "***" 'signal this with stars
 FLAG% = 1
 GOTO 6020 'in the bar graph
6010 FOR J% = 1 TO NUMUNITS% 'Else print units
 LPRINT "=";
 NEXT J%
 LPRINT
6020NEXT I%
LPRINT
LPRINT SPC(6);"TOTAL NO. OF SORTED GRAINS: ";
LPRINT USING IFORMAT4$; RANGETOTAL%;
LPRINT SPC(7); "SUM OF DIAMETERS: ";
LPRINT USING IFORMAT4$; DMTRTOTAL%
LPRINT
LPRINT SPC(6);"CLAIMED ENTRIES FOR SAMPLE: ";
LPRINT USING IFORMAT4$; TOTGRAIN%;
LPRINT SPC(7);
IF FLAG% = 1 THEN LPRINT "*** MEANS > 10 UNITS IN GRAPH"
LPRINT
LPRINT
RETURN

```





