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ABSTRACT

Fifty-five raw coal samples from 10 coal seams in Illinois have a mean mercury concentration of 0.18 part per million and a mode that lies between 0.10 ppm and 0.12 ppm. Eleven coal samples from states other than Illinois have mercury concentrations within the same range as Illinois coals, or slightly lower.

The neutron activation method used for the determination of mercury in coal, developed at the Illinois State Geological Survey, has a sensitivity of approximately 0.01 ppm and a precision of about ± 20 percent.

Three coal samples were individually separated into specific gravity fractions in the laboratory, and all exhibited a mercury reduction of at least 50 percent in the lightest coal fraction and a concentration of mercury in the heaviest fraction.

A more detailed study of a single coal sample suggests that a significant part of the mercury is associated with the pyrite in the coal, and the remainder (perhaps up to 50 percent) is in organic association.

INTRODUCTION

Mercury (Hg) in coal and its potential deleterious effects on the environment have received much coverage recently in the popular press. Because the analysis of mercury in coal is difficult, no mercury values for Illinois coals have previously been reported. To procure data that would

allow a reasonable assessment of the effect of mercury in coal upon the environment, we determined the quantity present in Illinois coals. Analyses made for mercury in several coals from eastern and western states also are included to provide a comparison with Illinois coals.

Only a very limited amount of data has been published in the international literature on mercury content of coals. Perhaps the most detailed studies are those concerning the coals of the Donetz Basin in the USSR (Vasilevskaya and Shcherbakov, 1963; Vasilevskaya, Shcherbakov, and Karakozova, 1964; Karasik, Dvornikov, and Petrov, 1963). Karasik, Dvornikov, and Petrov (1963) reported values of less than 1 part per million (ppm), or less than 1 gram per metric ton, for "normal" coals and found values greater than 1 ppm only for coals in the proximity of known mercury mineral deposits.

Headlee and Hunter (1955) analyzed a series of ash samples of West Virginia coals for 35 elements, using optical emission spectrography. They reported an average mercury value for the ash that is more than 500 times greater than the average value reported in this paper for mercury in Illinois coals.

Although mercury concentrations in coal had not been studied at the Illinois State Geological Survey prior to this investigation, we have been determining mercury in Lake Michigan bottom sediments by neutron activation, and a report of that investigation is currently in press (Kennedy, Ruch, and Shimp, 1971). The analytical method used on the Lake Michigan sediments was modified and applied to this investigation of mercury in coal.

ANALYTICAL PROCEDURE

From 0.6 to 1.0 gram of coal (hand-ground to 20 mesh and air dried) was accurately weighed into 1/2-dram polyethylene snap-cap vials that had previously been cleaned with deionized water and acetone. A 1.0 ml aliquot of a 10.3 mg per ml standard solution of Hg^{++} (as nitrate) was put in a similar polyethylene vial. Samples and standard were simultaneously irradiated in the University of Illinois Advanced TRIGA reactor for 110 minutes in a thermal neutron flux of 1.4×10^{12} neutrons cm^{-2} sec^{-1}. During the irradiation all samples and the standard were rotated at 1 rpm to insure equal neutron flux. Approximately one day was allowed for the preferential decay of shorter-lived radioisotopes (such as ^{24}Na, ^{31}Si, ^{56}Mn) to facilitate handling.

Each sample was mixed 1 to 1 with 60-mesh Norton Alundum RR (Al_2O_3), transferred to a 4-inch porcelain boat (Fisher Combax, size A), and covered with Alundum. The boat, previously impregnated with 2 mg Hg^{++} carrier (as nitrate), was placed in a 1-inch diameter Vycor tube and the contents then combusted slowly with a Bunsen burner ($\sim 500^{o}$-600° C) in a flow system, with an oxygen flow through the tube of about 50 to 75 ml per minute. The gaseous and volatilized products were bubbled through two consecutive 100 ml vacuum traps, each containing 20 ml of a 3.25 pH sodium acetate—acetic acid buffer solution (Hinkle, Leong, and Ward, 1966), 40 ml of saturated bromine water, and 30 mg Hg^{++} (as nitrate). The combustion process required about one hour to insure controlled, thorough burning and efficient transfer to the traps. *(NOTE:* Sample should not be burned

too rapidly, as there is danger of violent explosion.) Approximately 250 ml of 2M HCl was used to wash the glassware thoroughly, including the Vycor tube, and was then combined with the trap solutions.

The resulting solution (∿ 450 ml) was then passed through a column bed 1 cm in diameter that contained 3.5 ml of Dowex 2 in the chloride form. After radioactive interferences were eluted with 40 ml water and 40 ml of 2M HCl, the resin was transferred to a 100 ml polystyrene bottle, was allowed to settle uniformly, and was then counted for ^{197}Hg ($t_{1/2}$ = 65 hours, 77 keV gamma ray) with a 3 x 3-inch NaI detector and a 400-channel Nuclear Chicago gamma-ray spectrometer system.

A 0.10 ml aliquot of the irradiated standard was diluted to 100 ml with 2M HNO_3. One ml of the diluted solution was immediately pipetted into a porcelain boat (already impregnated with 2 mg *inactive* Hg^{++}) and allowed to dry. About one gram of inactive coal was mixed with Alundum, put into the boat, covered with Alundum, and burned in the same manner as the irradiated samples.

The over-all recovery in the process was 67 ± 15 percent. The amount of mercury in a sample was calculated by comparing the height of the photopeak of the sample to that of the standard. The precision of the method is about 20 percent and the sensitivity about 0.01 ppm for 1-gram samples and 2-hour irradiations.

Because coal standards with known mercury content are unavailable at this time, evaluation of the method's accuracy is not possible. However, the U. S. Bureau of Mines has begun a study to compare mercury analyses of two coal samples determined by a variety of methods at a number of laboratories. Our values for the two samples were reported during a recent meeting at Bituminous Coal Research, Monroeville, Pennsylvania, and were in good agreement with those values obtained by atomic absorption, mass spectroscopy, and a different neutron activation technique used by other laboratories cooperating in the Bureau of Mines study.

Other approaches were attempted before the final procedure was established. Both whole coal and the low-temperature ash of the whole coal were analyzed by another hot-tube technique previously developed for mercury in sediments (Kennedy, Ruch, and Shimp, 1971), which involved heating the sample to 800° C under iron filings in a nitrogen (N_2) atmosphere. The resultant data agree basically with the reported results except where some possible contamination in the low-temperature ash occurred.

In another series of experiments, irradiated coal was burned in oxygen at 500° C. The residue from this combustion was then treated according to the 800° C-nitrogen technique. No additional mercury was detected, indicating that the lower temperature, oxygen-burn technique quantitatively removed all mercury from the sample.

RESULTS AND DISCUSSION

Mercury Content of Coal

Fifty-five face channel, drill core, or column samples of Illinois coal were analyzed for mercury by the neutron activation method. The samples were all untreated (raw coal) and are representative of coal as it occurs in the ground. The results of these analyses, as well as the percentage of ash, total sulfur, pyritic sulfur, sulfate sulfur, and organic sulfur are given in table 1.

As indicated in table 1, the 55 Illinois coal samples analyzed are from 10 different coal seams. Mines from which these samples were collected are widely distributed geographically and represent all the major mining districts in the state. The samples are identified in table 1 as one of three sample types: composite face channel sample, column sample, or drill core sample. The composite face channel sample is a mixture of two or three samples taken individually, by hand, in a single mine and includes coal from the total coal being mined, with the exception of mineral bands or partings more than 3/8 of an inch thick. The column samples differ from the face channel samples only in that all mineral bands and partings have been included in the sample. Drill core samples also represent the full seam thickness but exclude mineral bands greater than 3/8 of an inch thick.

Mean mercury content of the samples from coal seams currently being mined in Illinois is 0.18 ppm. Thirty-two samples of the Herrin (No. 6) Coal, which is the most extensively mined coal in Illinois, contain an average (mean) of 0.16 ppm mercury. The range in mercury content in the coals currently being mined is from 0.04 ppm to 0.49 ppm, and the mode is between 0.10 ppm and 0.12 ppm (fig. 1). Forty-three samples (78 percent of the total analyzed) lie between 0.07 and 0.24 ppm. Higher values were obtained from two samples of Reynoldsburg Coal, a coal not now being mined. The Reynoldsburg Coal is the oldest coal (lowest stratigraphically) of the Illinois coals sampled. The locality from which it was collected lies the farthest south of all the sample sites. Surprisingly, the Reynoldsburg sample (C-17089), which has the highest mercury concentration of all the coal samples, also has the lowest ash and pyritic sulfur values.

In one test, three face channel samples from one mine were analyzed individually rather than being combined into a composite sample. These three samples of Harrisburg (No. 5) Coal contained 0.20 ppm, 0.23 ppm, and 0.26 ppm mercury. In this case, the local variation approximated the reproducibility of the analytical method. Two samples of Summum (No. 4) Coal, taken approximately 2 miles apart and the second taken a year and a half after the first, gave the same mercury values (0.12 ppm). However, of two samples from the same mine of Herrin (No. 6) Coal taken several years apart, one contained 0.22 ppm mercury and the other 0.12 ppm mercury.

Cinnabar (HgS), the most important mercury mineral in nature, is often found in association with pyrite. However, no correlation of mercury content with ash, total sulfur, or pyritic sulfur can be discerned from the

TABLE 1—CHEMICAL ANALYSES OF ILLINOIS COALS

Coal	Type of sample*	Analysis number	Mercury (ppm)	Ash† (%)	Total sulfur† (%)	Pyritic sulfur† (%)	Sulfate sulfur† (%)	Organic sulfur† (%)
Danville (No. 7) Coal	FC	C-16895	0.39	10.0	3.01	1.16	0.01	1.84
Herrin (No. 6) Coal	FC	C-16954	0.22	12.1	4.25	1.66	0.03	2.56
	C	C-17016	0.12	12.1	5.56	2.87	0.10	2.59
	FC	C-16969	0.22	7.4	2.18	0.94	0.04	1.20
	FC	C-16967	0.18	10.0	2.79	1.44	0.02	1.33
	FC	C-16971	0.23	10.5	2.60	1.29	0.03	1.28
	C	C-16987	0.12	11.8	3.46	2.07	0.02	1.37
	FC	C-16832	0.15	13.0	3.45	1.42	0.01	2.02
	FC	C-16966	0.17	9.5	3.20	1.22	0.04	1.94
	FC	C-16972	0.15	12.9	3.55	1.54	0.05	1.96
	FC	C-16953	0.14	10.3	3.16	1.58	0.08	1.50
	FC	C-16952	0.18	9.1	1.43	0.65	0.01	0.77
	FC	C-16956	0.08	8.8	0.85	0.29	0.00	0.56
	FC	C-16970	0.12	10.3	1.88	1.14	0.02	0.72
	FC	C-16894	0.13	10.1	3.33	1.37	0.07	1.89
	FC	C-16833	0.13	12.7	4.33	2.55	0.05	1.73
	FC	C-16948	0.10	14.1	4.84	2.27	0.11	2.46
	FC	C-16951	0.14	11.1	4.29	2.30	0.01	1.98
	FC	C-16941	0.32	13.6	4.20	2.26	0.08	1.86
	FC	C-16942	0.10	12.0	3.25	0.97	0.33	1.95
	FC	C-16957	0.35	15.3	3.98	2.13	0.07	1.78
	FC	C-16891	0.09	12.4	4.45	2.36	0.06	2.03
	FC	C-16961	0.32	10.8	3.73	1.76	0.03	1.94
	FC	C-16949	0.19	12.6	3.70	1.57	0.01	2.12
	FC	C-16834	0.17	13.1	3.81	1.60	0.08	2.13
	FC	C-16976	0.08	12.2	4.19	1.59	0.04	2.56

(Continued on next page)

TABLE 1—Continued

Coal	Type of sample*	Analysis number	Mercury (ppm)	Ash† (%)	Total sulfur† (%)	Pyritic sulfur† (%)	Sulfate sulfur† (%)	Organic sulfur† (%)
Herrin (No. 6) Coal (Continued)	FC	C-16959	0.14	12.4	3.31	1.79	0.08	1.44
	FC	C-16974	0.23	14.5	3.68	1.81	0.05	1.82
	FC	C-16963	0.10	12.2	1.74	0.98	0.05	0.71
	FC	C-16973	0.19	12.4	4.31	1.69	0.03	2.59
	FC	C-16892	0.12	12.5	3.92	2.42	0.12	1.38
	FC	C-16955	0.11	10.5	1.54	0.99	0.01	0.53
	C	C-16993	0.15	16.5	4.15	2.81	0.02	1.32
Springfield-Harrisburg (No. 5) Coal	FC	C-16975	0.22	12.1	4.01	2.34	0.02	1.65
	FC	C-16945	0.28	12.8	3.68	1.42	0.02	2.24
	FC	C-16965	0.24	12.4	4.52	2.33	0.05	2.14
	FC	C-16946	0.16	12.2	3.90	2.13	0.14	1.63
	FC	C-17082-4	0.23	11.8	4.14	2.62	0.16	1.51
	FC	C-16896	0.10	14.8	4.06	1.96	0.07	2.03
	FC	C-16897	0.04	10.6	4.27	2.47	0.02	1.78
	FC	C-16960	0.38	10.3	1.34	0.74	0.02	0.58
	FC	C-16947	0.09	10.7	4.35	2.67	0.06	1.62
Summum (No. 4) Coal	FC	C-16893	0.12	9.2	3.67	1.26	0.05	2.34
	C	C-16990	0.12	10.5	4.58	2.33	0.04	2.21
	FC	C-16962	0.19	14.3	5.59	3.78	0.06	1.75
Colchester (No. 2) Coal	FC	C-16958	0.49	10.1	4.85	3.38	0.05	1.42
	FC	C-16943	0.16	9.5	4.81	3.38	0.11	1.32
	FC	C-16940	0.27	10.9	4.83	2.72	0.04	2.07
	FC	C-16950	0.22	8.0	3.16	2.27	0.04	0.85

(Continued on next page)

TABLE 1—Concluded

Coal	Type of sample*	Analysis number	Mercury (ppm)	Ash† (%)	Total sulfur† (%)	Pyritic sulfur† (%)	Sulfate sulfur† (%)	Organic sulfur† (%)
DeKoven Coal	DC	C-16835	0.13	15.6	3.72	2.11	0.04	1.57
Davis Coal	DC	C-16890	0.11	12.0	5.46	4.39	0.08	0.99
Murphysboro Coal	FC	C-16968	0.30	11.2	4.90	3.78	0.05	1.07
Rock Island (No. 1) Coal	FC	C-16944	0.10	10.3	5.36	3.21	0.05	2.10
Reynoldsburg Coal	FC	C-16964	0.60	4.6	1.93	1.27	0.00	0.66
	FC	C-17089	1.15	3.8	0.56	Tr	0.00	0.56

* FC - Composite face channel sample; C - column sample; DC - drill core sample.
† Analytical values are given on a moisture-free basis.

analyses of the raw coal samples given in table 1. Nor does the data collected thus far seem to show other than a random areal distribution of the mercury content of Illinois coals.

Eleven raw coal samples from six states other than Illinois were also analyzed for mercury, ash, total sulfur, and varieties of sulfur. The results of the analyses are given in table 2. The three samples from Ohio and the two samples from Pennsylvania lie well within the range of values obtained for Illinois coals, the two from Montana are at the lower end of that range, and the four remaining western coal samples contain smaller amounts of mercury (from 0.02 ppm to 0.04 ppm).

Reduction of Mercury Content in Coal by Washing

Most of the coals mined in Illinois are "washed" or "cleaned" prior to delivery to the consumer. Cleaning involves reducing the ash and sulfur content of the coal by removing a portion of the mineral matter associated with the coal. Because the specific gravities of the minerals in coal are from two to four times as great as that of the coal, most coal cleaning techniques involve a specific gravity separation. Data on the washability of Illinois coals, as well as a description of the techniques used, have recently been published by Helfinstine et al. (1970).

We have analyzed the mercury content of sample sets from these coals that were prepared in our laboratories by various specific gravity separation techniques.

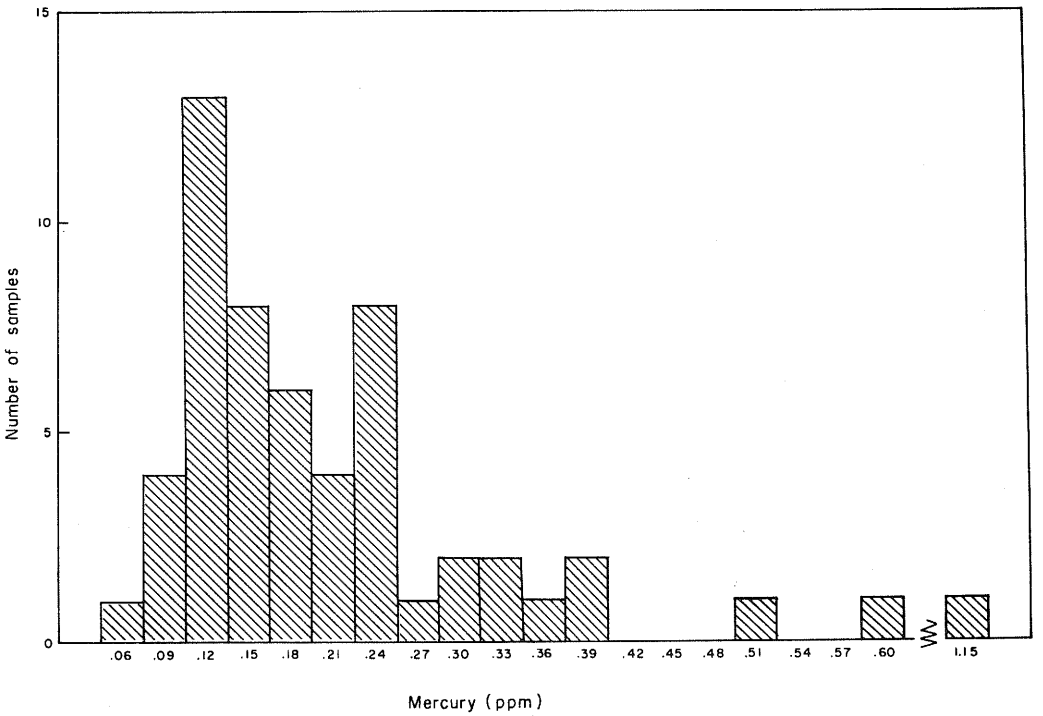


Fig. 1 - Mercury distribution (in 0.03 ppm class intervals) in samples of Illinois coals.

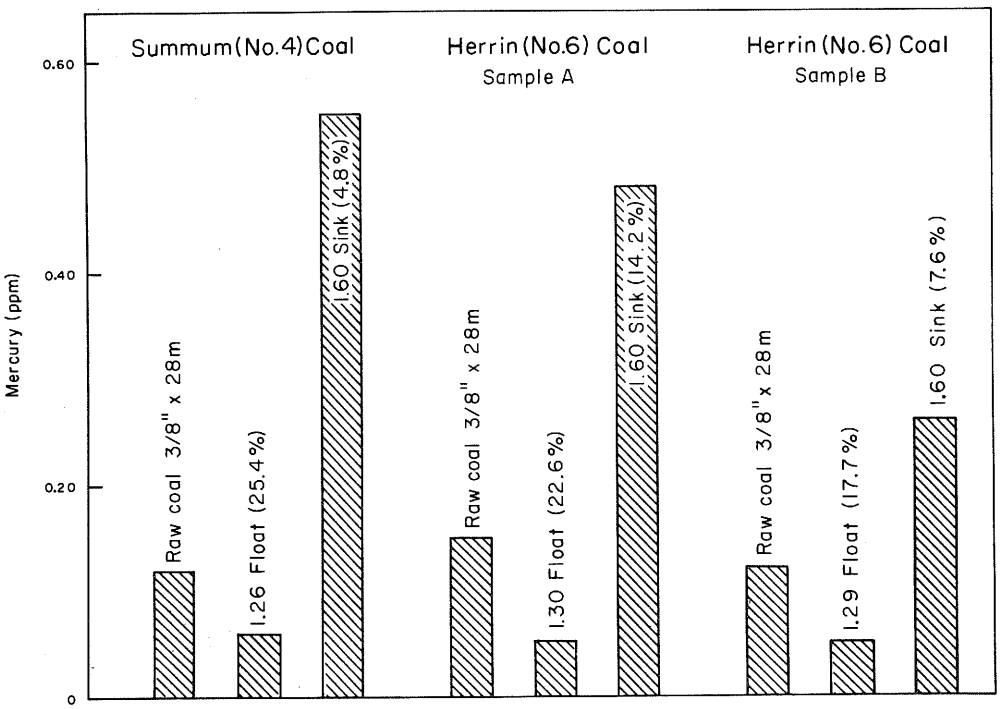


Fig. 2 - Mercury content of raw and prepared fractions of three Illinois coals.

TABLE 2—CHEMICAL ANALYSES OF SEVERAL COALS FROM MINES OUTSIDE ILLINOIS

State	Analysis number	Mercury (ppm)	Ash* (%)	Total sulfur* (%)	Pyritic sulfur* (%)	Sulfate sulfur* (%)	Organic sulfur* (%)
Arizona	C-17045	0.02	14.2	0.44	0.10	0.02	0.31
Colorado	C-17054	0.02	12.5	0.83	0.31	0.03	0.49
	C-17097	0.02	11.6	0.55	0.07	0.02	0.46
Montana	C-17047	0.07	13.9	1.88	1.16	0.02	0.70
	C-17046	0.09	13.3	1.11	0.53	0.02	0.56
Ohio	C-16983	0.15	15.3	3.94	2.37	0.16	1.41
	C-16986	0.13	16.9	4.20	2.59	0.19	1.42
	C-16984	0.10	11.9	2.33	1.26	0.02	1.05
Pennsylvania	C-16836	0.28	15.7	1.53	1.01	0.05	0.46
	C-16837	0.16	10.9	3.04	1.82	0.21	1.01
Utah	C-17096	0.04	7.7	0.69	0.24	0.05	0.40

* Analytical values are given on a moisture-free basis.

TABLE 3—CHEMICAL ANALYSES OF LABORATORY-PREPARED COALS

Coal and type of sample	Percentage of raw coal	Analysis number	Mercury (ppm)	Ash* (%)	Total sulfur* (%)	Pyritic sulfur* (%)	Sulfate sulfur* (%)	Organic sulfur* (%)
Summum (No. 4) Coal								
Raw coal, 3/8" x 28 mesh	100.0	C-16990	0.12	10.5	4.58	2.33	0.04	2.21
Washed, 1.26 s.g. float	25.4	C-16991	0.06	3.0	2.73	0.52	0.02	2.19
Washed, 1.60 s.g. sink	4.8	C-16992	0.55	59.0	21.32	20.44	0.05	0.83
Herrin (No. 6) Coal (Sample A)								
Raw coal, 3/8" x 28 m	100.0	C-16993	0.15	16.5	4.15	2.81	0.02	1.32
Washed, 1.30 s.g. float	22.6	C-16994	0.05	1.5	1.96	0.38	Tr	1.59
Washed, 1.60 s.g. sink	14.2	C-16995	0.48	59.4	10.16	9.79	0.19	0.19
Herrin (No. 6) Coal (Sample B)								
Raw coal, 3/8" x 28 m	100.0	C-16987	0.12	11.8	3.46	2.07	0.02	1.37
Washed, 1.29 s.g. float	17.7	C-16988	0.05	2.3	1.53	0.30	Tr	1.23
Washed, 1.60 s.g. sink	7.6	C-16989	0.26	54.6	15.07	14.39	0.13	0.55

* Analytical values are given on a moisture-free basis.

TABLE 4—CHEMICAL ANALYSES OF LABORATORY-PREPARED FRACTIONS OF SUMMUM (NO. 4) COAL

Type of sample	Wt. % of 3/8" x 28 m	Analysis number	Mercury (ppm)	Ash* (%)	Total sulfur* (%)	Pyritic sulfur* (%)	Sulfate sulfur* (%)	Organic sulfur* (%)
Raw coal, 28 mesh x 0	—	C-17039	0.15	16.8	4.86	1.97	0.65	2.24
Raw coal, 3/8" x 28 m	100.0	C-16990	0.12	10.5	4.58	2.33	0.04	2.21
1.26 float, 3/8" x 28 m	25.4	C-16991	0.06	3.0	2.73	0.52	0.02	2.19
1.26 sink, 1.28 float, 3/8" x 28 m	20.9	C-17031	0.09	5.2	2.98	0.76	0.04	2.18
1.28 sink, 1.30 float, 3/8" x 28 m	17.3	C-17032	0.07	5.3	2.98	0.78	0.04	2.16
1.30 sink, 1.40 float, 3/8" x 28 m	25.7	C-17033	0.11	11.2	3.95	1.62	0.10	2.23
1.40 sink, 1.60 float, 3/8" x 28 m	5.9	C-17034	0.22	21.7	5.15	2.97	0.16	2.02
1.60 sink, 3/8" x 28 m	4.8	C-16992	0.55	59.0	21.32	20.44	0.05	0.83
1.60 sink, 3/8" x 28 m	4.8	C-17087-8	0.68	57.5	20.96	19.52	0.94	0.49
1.60 sink, 2.14 float, 3/8" x 28 m	1.5	C-17085	0.27	41.3	5.85	4.06	0.46	1.33
2.14 sink, 3/8" x 28 m	3.3	C-17086	0.88	65.5	28.51	28.23	0.24	0.06

* Analytical values are given on a moisture-free basis.

Table 3 lists, for each of the three coals, analytical data for the raw coal, for a low specific gravity fraction of the coal, and for the portion of the coal that has a specific gravity greater than 1.60. The gravity separations were, in each case, made on a 3/8-inch by 28-mesh size fraction obtained by crushing the coal to less than 3/8 of an inch and then screening it. All three coals showed a decrease in mercury content of at least 50 percent in the lightest fraction, and all showed a concentration of mercury in the mineral-matter rich fraction with specific gravity greater than 1.60 (fig. 2). The mercury content of the heaviest specific gravity fractions were from two to five times the mercury content of the raw coal.

To investigate further the distribution of mercury in a single coal sample, additional specific gravity fractions of the Summum (No. 4) Coal were analyzed. The results of the various chemical analyses of those samples and their proportions in the total sample are given in table 4. The mercury content of fractions of this coal increases as the specific gravity of the fraction increases (fig. 3). The rate of increase is not linear, but the heavier and more mineral-matter rich fractions contain increasingly larger amounts of mercury. The heaviest fraction (specific gravity greater than 2.14) composes 3.3 percent of the total sample and contains 22 percent of the mercury. The mercury content to be expected at any rate of recovery of this coal (percent float fraction) may also be read from the "washability" curve in figure 4.

The amounts of mercury in the gravity fractions of this sample of Summum (No. 4) Coal correlate well with the pyritic sulfur content, as shown in figure 5. The ash values (recalculated to exclude the contribution of the pyrite) of the higher specific gravity fractions do not correlate with the mercury values (fig. 6). In these high ash, high specific gravity fractions the mercury is apparently concentrated with the pyrite. However, not all of the mercury in this sample of Summum (No. 4) Coal is in the pyrite or in the high ash, high specific gravity fractions. The lightest (less than 1.26 s.g.) fraction contains 0.06 ppm mercury, or half the concentration of the raw coal, which suggests that a portion of the mercury in this coal sample may be in organic association.

CONCLUSIONS

Fifty-five samples of raw coal from 10 coal seams in Illinois have a mean mercury concentration of 0.18 ppm. Eleven coal samples from states other than Illinois have mercury concentrations within the range of Illinois coals, or slightly lower.

An accurate neutron activation analysis method for the determination of mercury in coal has been developed at the Illinois State Geological Survey that has a sensitivity of approximately 0.01 ppm and a precision of about 20 percent.

Three coals were separated in the laboratory into various specific gravity fractions. The mercury content of the lightest fraction was, in each case, 50 percent or less of the mercury concentration in the raw coal; the

Fig. 3 -
Mercury content of specific gravity fractions of laboratory-prepared Summum (No. 4) Coal.

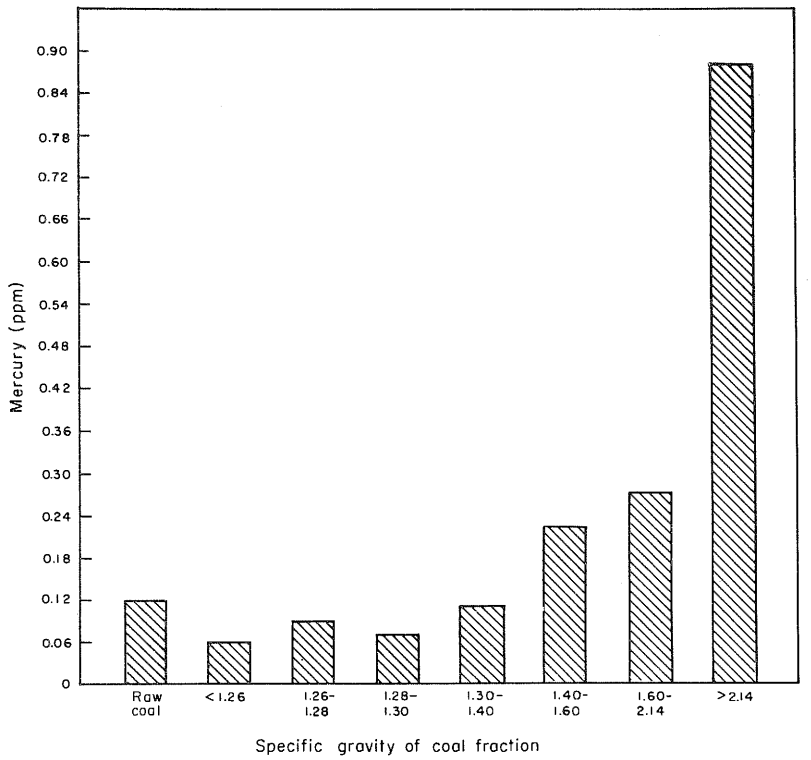
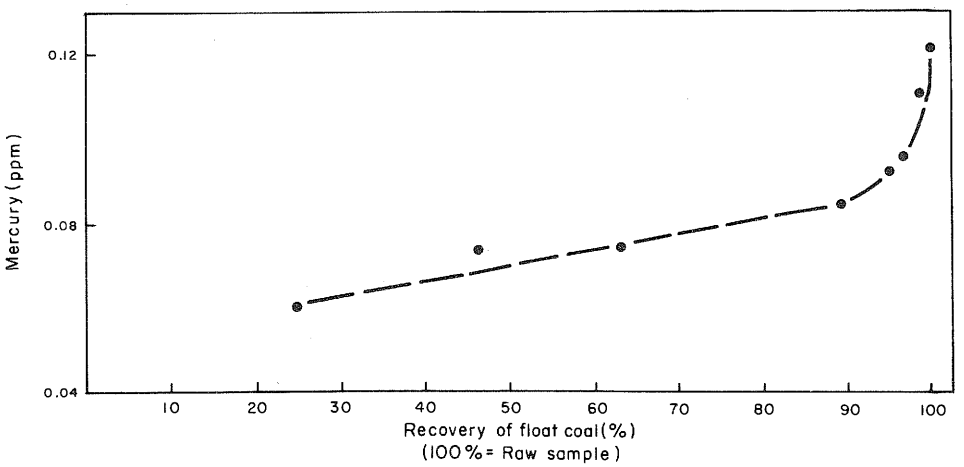


Fig. 4 -
Mercury washability curve for one sample of Summum (No. 4) Coal.



heaviest fraction contained a concentrated amount of mercury. A more detailed investigation of one of these coal samples has suggested that, although a portion of the mercury is concentrated in the heavier, pyrite-rich part of the coal, some of the mercury (perhaps as much as half) may be in organic association.

It would not be prudent to extrapolate such limited amounts of data as are given here into a definite statement concerning the mode of occurrence of mercury in coal. The danger of doing so is vividly demonstrated by the observation that the coal sample with the largest amount of mercury analyzed to date is one sample of Reynoldsburg Coal (C-17089, table 1), which contains no discernible pyritic sulfur.

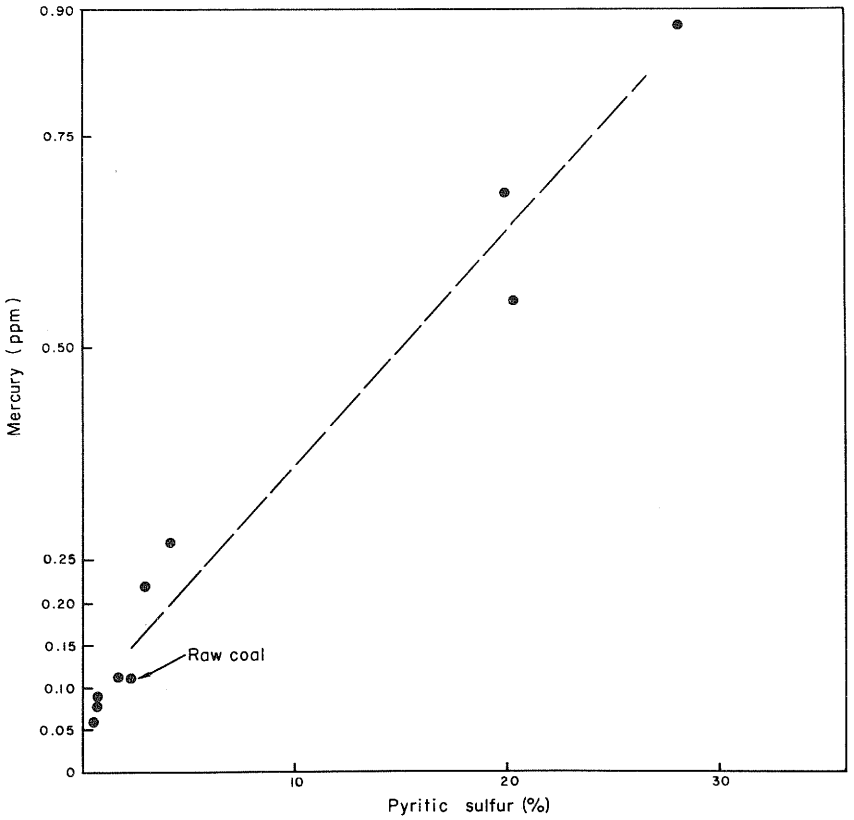


Fig. 5 -
Mercury versus pyritic sulfur content of laboratory-prepared specific gravity fractions of Summum (No. 4) Coal.

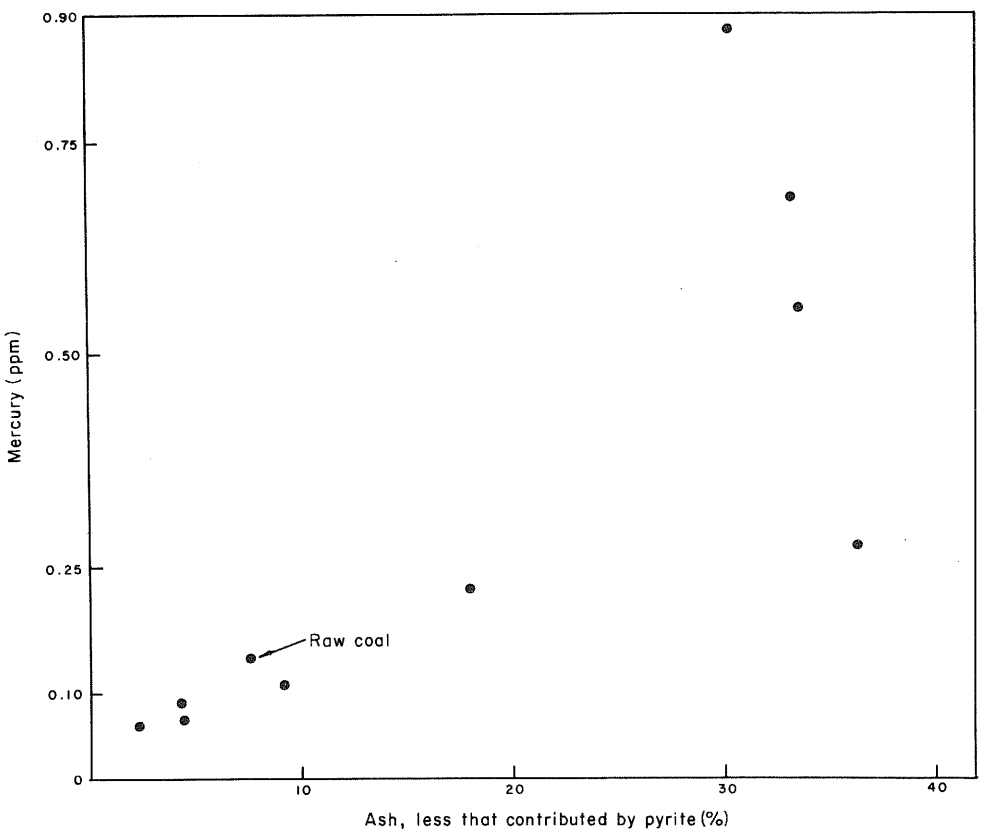


Fig. 6 -
Mercury versus ash (recalculated to a pyrite-free basis) for laboratory-prepared specific gravity fractions of Summum (No. 4) Coal.

Further investigations into the mode of occurrence and distribution of mercury in coals are continuing, specifically attempts to relate mercury to ash, pyrite content, and other trace element concentrations. Before the potential environmental effects of mercury in coal can be further assessed, the behavior of the mercury as the coal is burned under various conditions and the final chemical form in which it is stabilized must be determined.

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