Geochemical Data for Illinois Basin Coal Samples, 2015–2018

Data Series 1135

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Abbreviations

Actlabs	Activation Laboratories, Ltd.
Ag	silver
As	arsenic
ASTM	American Society for Testing and Materials (now ASTM International)
Au	gold
B	boron
Bi	bismuth
C	carbon
°C	degrees Celsius
Cd	cadmium
Co	cobalt
Cr	chromium
Cu	copper
EERSC	USGS Eastern Energy Resources Science Center
EPA	U.S. Environmental Protection Agency
EPMA	electron probe microanalysis
Fe	iron
FE-SEM	field emission scanning electron microscope/microscopy
g	grams
H	hydrogen
HCl	hydrochloric acid
Hg	mercury
$HgCl_2$	mercury chloride
ICP-AES	inductively coupled plasma atomic emission spectroscopy
ICP-MS	inductively coupled plasma mass spectrometry
ICP-OES	inductively coupled plasma optical emission spectroscopy
ICCP	International Committee for Coal and Organic Petrology
IGWS	Indiana Geological and Water Survey
INAA	instrumental neutron activation analysis
Ir	iridium
IR	infrared
kV	kilovolts
LA-ICP-MS	laser ablation inductively coupled plasma mass spectrometry
Li	lithium

MATS	Mercury and Air Toxics Standards
mg	milligrams
mL	milliliter
mm	millimeter
Mn	manganese
Mo	molybdenum
μm	micrometer (or micron)
ng	nanograms
Ni	nickel
NIST	National Institute of Standards and Technology
O	oxygen
Pb	lead
ppb	parts per billion
ppm	parts per million
QMS	Laboratory Quality Management System (USGS)
REE	rare earth elements
S	sulfur
Sb	antimony
Se	selenium
SEM	scanning electron microscope/microscopy
SIUC	Southern Illinois University Carbondale
SO_2	sulfur dioxide
SOP	standard operating procedure
SRM	standard reference material
Ti	titanium
Tl	thallium
USGS	U.S. Geological Survey
V	vanadium
Zn	zinc

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Abstract

Researchers at the U.S. Geological Survey (USGS) and their collaborators conducted a study of the geochemical properties of coals currently produced for electric power generation in the Illinois Basin in Illinois and Indiana. The study follows from recommendations by an expert panel for the USGS to investigate the distribution and controls of trace constituents such as mercury (Hg) in Illinois Basin coals and the behavior of these constituents in coal preparation. A total of 72 new samples were collected by USGS collaborators between 2015 and 2017. These samples include raw coals, prepared coals, and waste coals from coal preparation. To understand the geochemistry and cleaning behavior of these coals, these samples were subjected to an integrated series of analyses described here, including microanalysis of coal constituents and bulk sample chemical analysis. Of the procedures used, whole-sample Hg analysis quantified overall mercury contents and its reduction by coal preparation. Laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS) of pyrite in coal quantified Hg and other potentially harmful elements contained in pyrite, the most likely host of these constituents. Trace elements investigated include those whose emissions are regulated under the U.S. Environmental Protection Agency (EPA) Mercury and Air Toxics Standards (MATS; U.S. Environmental Protection Agency, 2011). This report and the corresponding data release (Kolker and others, 2021), serve as an archive for geochemical data obtained in our study of the geochemistry of Illinois Basin coals. Material included in this report also define approaches used by the USGS over the period of study to characterize coal samples, requiring combined use of results from USGS and non-USGS laboratories.

Introduction

Near the conclusion of the first U.S. Geological Survey (USGS) Geochemistry of Energy Fuels project (Oct. 1, 2010, to Sept 30, 2015), a panel of 25 experts in coal geochemistry was surveyed regarding priorities for new USGS project work on mercury (Hg) in U.S. coals, whose emission had become regulated under the U.S. Environmental Protection

Agency (EPA) Mercury and Air Toxics Standards (MATS) (U.S. Environmental Protection Agency, 2011). The consensus of the panel was that new studies of Hg in U.S. coals were beneficial to the USGS and the Nation (Kolker, 2016). The panel identified the Illinois Basin as one of the highest priority areas for new studies of Hg and associated trace elements in coal. This report and the recent accompanying data release (Kolker and others, 2021) are the result of new sampling of Illinois Basin coals in the succeeding second USGS Geochemistry of Energy Fuels project (Oct. 1, 2015, to Sept. 30, 2020). The goals of this sampling are to better understand the controls on Hg and trace element distribution in Illinois Basin coals, and the behavior of these elements during coal preparation. These data also serve as a partial update to geochemical results included in the 2002 USGS Illinois Basin coal resource assessment (Hatch and Affolter, 2002). While U.S. coal production has declined in recent years as coal use for power generation has been supplanted by gas-fired capacity, that decline has been least pronounced in the Illinois Basin, as Illinois Basin coals are currently (2018–2019) the most cost-competitive U.S. coal source. The high sulfur content of some Illinois Basin coals is no longer an impediment to their use in power generation as sulfur dioxide scrubbers are increasingly employed in modern coal-fired power stations. For these reasons, the expert panel recommended that among potential new study areas, the USGS investigate controls on Hg and trace element distribution in Illinois Basin coals.

Sample Descriptions and Collection Methods

A total of 72 new coal, waste coal, and prepared coal samples were obtained from the southern and east-central portions of the Illinois Basin in Illinois, and from the eastern margin of the basin in southwest Indiana (fig. 1). Samples include raw coal, cleaned coal, and waste (refuse) coal samples collected from working operations, from both surface and underground workings (Kolker and others, 2021, tables 1.1 and 1.2). Illinois coals represented in this sampling include the Herrin #6 (30 samples), Springfield #5 (5 samples), Colchester #2 (1 sample), De Koven (4 samples), Davis #2

(1 sample), and Murphysboro (2 samples), listed in descending stratigraphic order (Willman and others, 1975). Indiana coals sampled include the Danville #7 (2 samples), Hymera #6 (1 sample), Springfield #5 (6 samples), Bucktown #5a (1 sample), Buffaloville (4 samples), Upper Block (4 samples) and Lower Block (6 samples), listed in descending stratigraphic order. In addition, five samples from Indiana consist of mixed coals from different stratigraphic horizons. No new samples were obtained from the Kentucky part of the Basin. The 2002 USGS Illinois Basin coal resource assessment emphasized the three most important producing horizons, the Danville-Baker, Herrin, and Springfield coals (Hatch and Affolter, 2002). Coals below the Springfield #5 were not assessed, and results for these coals were included only as averages by formation, including multiple non-assessed units. In the present study, major and trace element data are provided for the coals listed above, with the exception of the Buffaloville coal in Indiana, which is not an important commercial producer.

Sample Preparation

Initial sample preparation for whole sample (bulk) analysis and microanalysis was conducted at Geochemical Testing, Inc., of Somerset, Penn., (hereafter, referred to as Geochemical Testing). Samples collected by USGS collaborators at Southern Illinois University Carbondale (SIUC) and the Indiana Geological and Water Survey (IGWS) were returned directly to Geochemical Testing, where raw samples were prepared according to ASTM D2013/D2013M–20 (ASTM International, 2020). Bulk sample splits were ground to pass (95 percent) 60 mesh (0.250 millimeters [mm]) for bulk chemical analysis, and ground to pass (95 percent) 20 mesh (0.841 mm) for microanalysis. These representative splits were then provided to the USGS, together with archival material used to prepare the splits. Prior to providing prepared sample material to the USGS, Geochemical Testing also conducted proximate, ultimate, and sulfur forms analysis using ASTM methods (Kolker and others, 2021, tables 2.1 and 2.2).

Polished pellets of 20-mesh (petrographic) fractions were prepared in Reston, Va., at the USGS Coal Petrography Lab. Pellets were prepared in duplicate, for elemental microanalysis, and coal maceral analysis, respectively. Polished sections for elemental microanalysis were used in scanning electron microscope (SEM), electron microprobe, and laser-ablation inductively coupled plasma mass spectrometry (LA-ICP-MS) analysis. Duplicate polished pellets were sent to IGWS, in Bloomington, Ind., for coal maceral analysis. Polished sample pellets were prepared according to ASTM D2799–13 (ASTM International, 2013a), in which pellets are prepared from crushed coal cast in a thermoplastic acrylic powder. Samples were ground and polished using an automatic grinder/polisher. The abrasion sequence used was 240-grit, 400-grit, 600-grit, and 1200-grit silicon carbide paper. The polishing sequence used was a 0.3-micron (µm) alumina polish on non-woven, pressed cloth, with a final polish using 0.06-µm colloidal silica on synthetic velvet cloth.
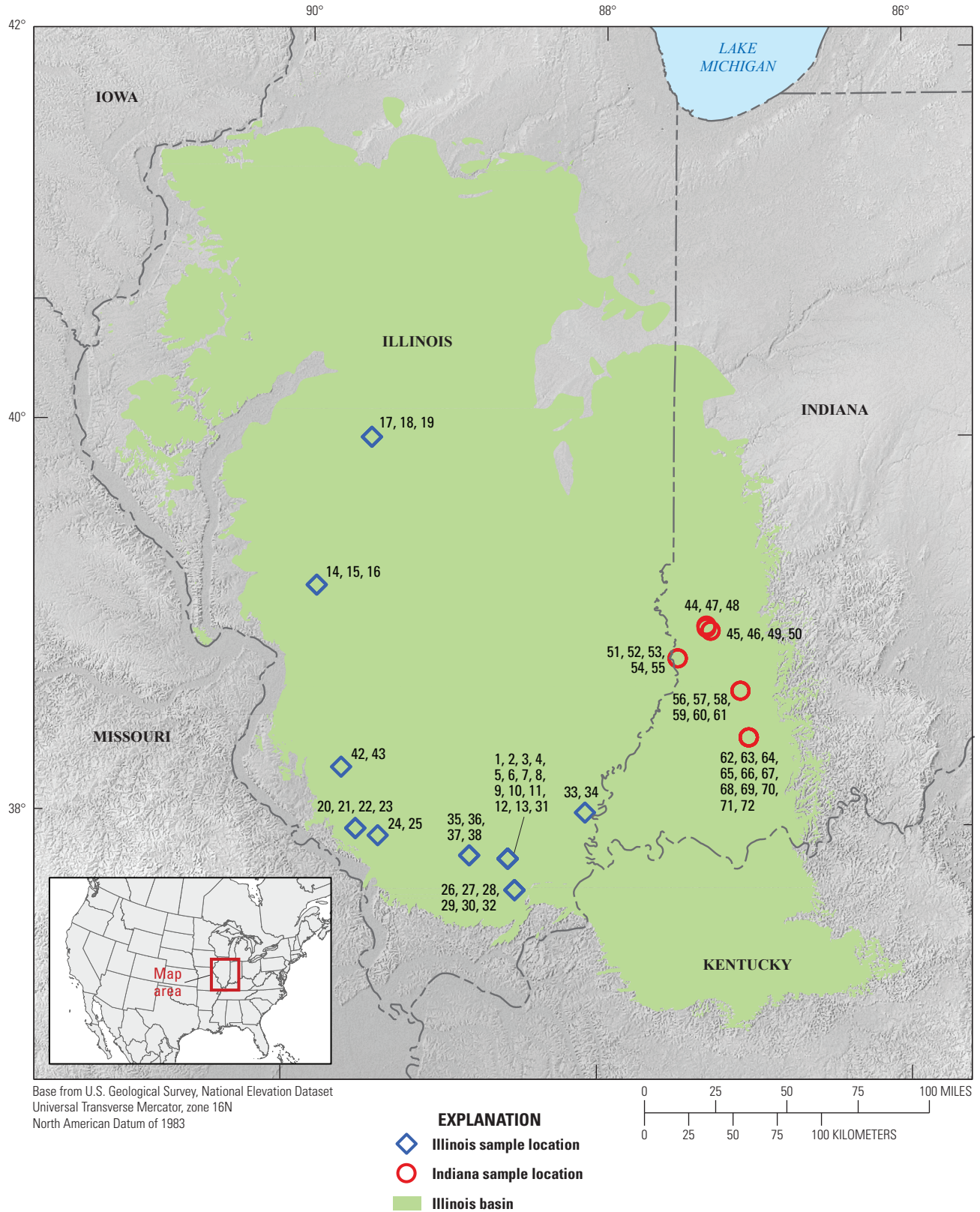
Figure 1. Map showing the distribution of Illinois Basin coal samples collected in Illinois and Indiana. Samples are numbered according to their listing in Kolker and others (2021, tables 1.1 and 1.2). Sample numbers 39, 40, and 41 are not shown as their location is proprietary.

Analytical Methods

Approach

A combination of bulk sample analysis and microanalysis methods were used. These approaches encompass use of both USGS and commercial labs, and for coal maceral determination, a USGS collaborator laboratory. Where applicable, sampling, characterization, and analysis of coal samples followed ASTM methods. Labs operated by the USGS Eastern Energy Resources Science Center (EERSC), now the Geology, Energy and Minerals Science Center, are subject to the Eastern Energy Quality Management System (QMS), implemented on May 1, 2017. These include the Reston Electron Beam Laboratory (electron microprobe, field emission scanning electron microscope), the Reston Coal Petrography Lab, and the Reston Trace Element Instrument Laboratory (mercury analyzer). These laboratories follow QMS-approved standard operating procedures (SOPs). Other USGS laboratories, including the Denver laser ablation ICP-MS laboratory, follow independent quality assurance procedures in advance of USGS Bureauwide QMS implementation.

Bulk Analysis

Proximate Analysis, Ultimate Analysis, and Sulfur Forms

Using ASTM methods, a series of tests giving information on overall coal quality for each sample was provided to the USGS by Geochemical Testing. Proximate analysis (ASTM D3172–13; ASTM International, 2013b) encompasses the determination of moisture, volatile matter, ash yield, and the calculation of fixed carbon by difference, each having specific ASTM methods. Moisture (ASTM D3302/D3302M–17; ASTM International, 2017) and ash-yield (ASTM D3174–12(2018); ASTM International, 2018a) are determined by mass balance following drying and combustion, respectively, of a sample of coal having a known mass. Ultimate analysis (ASTM D3176–15; ASTM International, 2015) is a bulk chemical assay, and includes determination of hydrogen (H), carbon (C), nitrogen (N), oxygen (O), and total sulfur (S), together with ash yield from proximate analysis (Kolker and others, 2021, tables 2.1 and 2.2). Total S (ASTM D4239–18e1; ASTM International, 2018b) is measured as SO_2 (grams [g]) using infrared (IR) spectrometry following combustion. Heating value (ASTM D5865–13; ASTM International, 2013c), determined as part of coal characterization, is measured using a calibrated calorimeter. Forms of sulfur (ASTM D2492–02(2012); ASTM International, 2012) include sulfate, sulfide, and organic forms. Sulfate S is determined gravimetrically following extraction via dilute hydrochloric acid (HCl). Sulfide S is assumed to be dominantly pyritic (pyritic sulfur) and is therefore calculated as a stoichiometric combination with iron (Fe). Organic sulfur is a calculated parameter, determined by subtraction of sulfate and sulfide from the total sulfur measured above. Pyritic sulfur is especially useful as a measure of the proportion of total sulfur in coal occurring as pyrite.

Major and Trace Element Analysis

Major and trace element data were determined by Activation Laboratories, Ltd. (Actlabs), Ancaster, Ontario, Canada using a dedicated coal analysis package that includes element determination by inductively coupled plasma atomic emission spectroscopy (ICP-AES) and (or) inductively coupled plasma optical emission spectroscopy (ICP-OES; hereafter, referred to as ICP), as well as inductively coupled plasma mass spectrometry (ICP-MS), and instrumental neutron activation (INAA; table 1; Kolker and others, 2021, tables 3.1, 3.2, and 3.3). Prior to analysis, 60-mesh splits of bulk samples were combusted by Actlabs at 525°C for 36 hours to produce laboratory ash used in ICP and ICP-MS determinations. Compared to the ASTM D3174–12(2018) (ASTM International, 2018a) ash yield determination at 750°C, coal combustion at 525°C is thought to limit loss of moderately volatile trace elements such as arsenic (As), and antimony (Sb), while retaining all but the most volatile elements such as mercury (Hg) and selenium (Se). This approach to preparation of laboratory ash for analysis was followed in past elemental analysis of coal by the USGS (Golightly and Simon, 1989; Palmer, 1997).

After laboratory combustion at 525°C, the resulting ash was digested using a lithium metaborate/tetraborate fusion and brought into solution for ICP and ICP-MS analysis. Major elements were determined by ICP following fusion digestion. The detection limit for individual major element oxides by ICP is 0.01 percent, except for MnO and TiO_2, for which the detection limit is 0.001 percent. For elements primarily associated with metals (Ni, Cu, Zn, Pb, S), a multi-acid digestion (including nitric, perchloric, hydrofluoric and hydrochloric acids), was used prior to determination by ICP. To ensure that elements with potential for volatilization are quantified, such as Se and Sb, these were determined on aliquots of whole coal, by INAA (table 1).

For trace elements, detection limits by ICP, ICP-MS, and INAA range from 5 parts per million (ppm) for V, Cr, Pb, to the parts per billion (ppb) range for Ir and Au by INAA (table 1). Most trace elements determined by these combined techniques with contents in the ppm have detection limits in the 0.1 to 1.0 ppm range (table 1). Results determined by ICP and ICP-MS on coal ash reported in Kolker and others (2021) are converted to a whole-coal basis using values obtained for moisture and ash yield immediately prior to analysis. As an internal check for decomposition of rare earth element (REE)-bearing trace phases such as zircon and monazite, chondrite-normalized plots of sample REE contents were included with quality assurance documentation provided by the laboratory.

Table 1. Digestion and analysis methods for trace elements in coal and their detection limits.

[Source: Activation Laboratories Ltd. (Actlabs) (2020), at https://actlabs.com/. ICP, inductively coupled plasma; ICP-MS, inductively coupled plasma mass spectrometry; INAA, instrumental neutron activation analysis; ppb, parts per billion; ppm, parts per million]

Element(s)	Digestion	Analysis	Detection limit, in ppm
V	Fusion	ICP	5
Pb	Acid	ICP	5
Cr	None	INAA	5
Se	None	INAA	3
Sr, Zr, Ba	Fusion	ICP	2
Rb, Mo	Fusion	ICP-MS	2
Be, Y	Fusion	ICP	1
Co, Ga, Ge, Nb, Sn, W	Fusion	ICP-MS	1
Ni, Cu, Zn	Acid	ICP	1
Ag, Cs	Fusion	ICP-MS	0.5
Cd	Acid	ICP	0.5
As, Br	None	INAA	0.5
Bi	Fusion	ICP-MS	0.4
In, Hf	Fusion	ICP-MS	0.2
Sb	None	INAA	0.2
La, Ce, Nd, Sm, Gd, Tb, Dy, Ho, Er, Yb, Ta, Tl, Th, U	Fusion	ICP-MS	0.1
Sc	None	INAA	0.1
Pr, Eu, Tm	Fusion	ICP-MS	0.05
Lu	Fusion	ICP-MS	0.01
Ir	None	INAA	5 ppb
Au	None	INAA	2 ppb

In addition to the procedure described above, for a subset of 13 ashed coal samples (IL-1 to IL-8, and SIUC-2 to SIUC-6) a sodium peroxide fusion digestion approach was used (Kolker and others, 2021, table 3.1) prior to determination by ICP and ICP-MS. Like the lithium metaborate/tetraborate fusion procedure, sodium peroxide fusion is superior to acid digestion for decomposition of insoluble trace phases that carry REEs (Kolker, 2018). Detection limits obtained by ICP and ICP-MS following sodium peroxide fusion, are comparable to those obtained in the procedure using lithium metaborate/tetraborate fusion. However, with addition of sodium peroxide, sodium cannot be determined as a major oxide. And without separate determination on whole coal by INAA, Se is assumed to be all or partially lost in the ashing process. Preparation of ash at 525°C minimizes loss of other, moderately volatile trace elements such as Sb, which were determined by INAA on whole coal (together with Se) in the procedure used for all samples except for samples IL-1 to IL-8 and samples SIUC-2 to SIUC-6. Use of lithium metaborate/tetraborate fusion for the larger group of samples does not allow determination of lithium (Li) and boron (B), which cannot be measured following addition of lithium metaborate/tetraborate.

Mercury Analysis

Mercury (Hg) contents for Illinois Basin coal samples were determined at the USGS EERSC in Reston, Va., using a Nippon MA-3000 instrument (Kolker and others, 2021, tables 4.1 and 4.2). This and similar benchtop Hg analyzers utilize EPA Method 7473, in which samples are heated and the evolved Hg is selectively captured as an amalgam and measured by atomic absorption spectrophotometry (U.S. Environmental Protection Agency, 2007). Mercury results were acquired under the USGS Eastern Energy QMS, according to a QMS-approved standard operating procedure (SOP) (Scott and Kolker, 2018). The SOP broadly follows operating procedures outlined in the Nippon MA-3000 instrument manual. The MA-3000 has an absolute detection limit of 0.002 nanograms (ng) Hg, a linear range from 0.002 to 2000 ng Hg, and an upper, non-linear range of 25,000 ng Hg. Two calibration ranges (low, 0–10 ng; high, 20–200 ng) are used. The instrument features automatic switching between low and high dynamic ranges as needed. A typical working range for the method is 0.05 to 600 ng Hg. For a routine sample loading of 50 milligrams (mg), this corresponds to a

nominal detection limit of 1 ppb Hg. All samples that were determined fall within the range of the primary calibration standards and the linear range for the instrument.

Quantification is achieved using a series of calibration standards prepared from mercury chloride ($HgCl_2$) stock solution having a concentration of 1.0 mg Hg/mL. A calibration curve is obtained by running primary calibration standards prepared by diluting a $HgCl_2$ stock solution with an L-cysteine preservative solution to contain 0.0, 0.5, 1.0, 5.0, 10.0, 20.0, 50.0, 100, and 200 ng Hg. NIST standard reference materials (SRMs) including NIST 1632d (Trace Elements in Coal (bituminous) NIST, 2014) and NIST 1633c (Trace Elements in Coal Fly Ash, NIST, 2011) are used as internal standard reference materials. In a typical sample analysis sequence, following initial instrument calibration, two SRMs and a blank are run before and after each series of 10 unknowns (Scott and Kolker, 2018). Results obtained prior to- and following implementation of the QMS are highly comparable (table 2).

Microanalysis

Scanning Electron Mcroscopy

All microanalyses (from SEM, electron microprobe (EPMA), and LA-ICP-MS) were conducted on the same set of polished pellets as described above. For scanning electron microscopy, all samples included in the study were analyzed using a Hitachi SU5000 variable pressure field-emission scanning electron microscope (FE-SEM) at the USGS Reston Electron Beam Laboratory. The FE-SEM was used in both the SEM and backscattered modes to identify potential hosts of elements of environmental interest in coal, such as pyrite (Kolker, 2012). Backscattered electron imaging is especially useful in identifying non-silicate minerals such as pyrite, as well as trace metal zoning within pyrite, due to their contrast in mean atomic number compared to carbonaceous or aluminosilicate hosts. Operating procedures for the Hitachi SU5000 FE-SEM are given in the QMS-approved laboratory SOP (Valentine, 2018). In the present study, the instrument was operated at 15 kilovolts (kV), an instrument-specific unit-less spot size of 30 (range 1 to 100), and a working distance of 10 mm.

Electron Microprobe Elemental Mapping

Representative pyrite grains identified in the coal samples identified by the FE-SEM were checked for minor element compositional variation by backscattered electron imaging and wavelength-dispersive elemental mapping, using the JEOL JXA 8900R electron microprobe at the USGS Reston Electron Beam Laboratory. The operating conditions for elemental mapping were 20-kV accelerating voltage and 300 nanoamps (nA) of beam current. Pixel dwell time for all maps was 30 milliseconds per pixel, whereas step size (that is pixel size) varied between 0.5 and 2 µm depending on the map, with most maps collected with a 1-µm step size; map dimensions were adjusted according to the size of the region of interest. Raw intensity maps were acquired in two analytical sessions. In the first session Fe, S, Cu, Zn, and As were analyzed and in the second session Se was analyzed instead of Zn. For both sessions the analyzing crystals and X-ray lines were: spectrometer 1, Fe K-alpha (lithium fluoride [LIF] analyzing crystal); spectrometer 2, Cu K-alpha (LIFH analyzing crystal; also lithium fluoride, but curved to fit an H-type spectrometer with a Rowland circle of 100 mm); spectrometer 3, As La-alpha (thallium acid phthalate [TAP] analyzing crystal). For session 1, additional analyzing crystals were: spectrometer 4, Zn K-alpha (LIF analyzing crystal); and spectrometer 5, S K-alpha (PET analyzing crystal). For session 2, additional analyzing crystals were: spectrometer 4, S K-alpha (pentaerythritol [PET] analyzing crystal); and spectrometer 5, Se L-alpha (TAP analyzing crystal). Mapping at these conditions

Table 2.   Results of mercury determination of coal samples from the Herrin #6 Coal Prep Plant.

[Results are from the U.S. Geological Survey, Eastern Energy Resources Science Center (EERSC) in Reston, Va., using a Nippon MA-3000 instrument. [1]Results obtained prior to May 1, 2017, are for informational purposes only. [2]Mixed product includes IL-2, IL-4, and IL-6 clean coal samples. Hg, mercury; ppb, parts per billion (on an as-received basis)]

Sample number	Material	Hg, in ppb, analyst A, April 10, 2018	Hg, in ppb, analyst B, June 25, 2015[1]	Percent deviation
IL-1	Raw prep plant feed	228.6	236.1	-3.2
IL-2	Coarse clean	105.2	102.3	+2.8
IL-3	Coarse refuse	295.4	296.7	-0.4
IL-4	Intermediate clean	78.8	78.6	+0.3
IL-5	Intermediate refuse	168.0	169.0	-0.6
IL-6	Fine clean	73.1	71.2	+2.7
IL-7	Fine refuse	129.8	133.7	-2.9
IL-8	Final mixed product[2]	84.7	86.3	-1.9

revealed little or no discernable minor element variation in Illinois Basin pyrite, suggesting that concentrations of each minor element mapped were below the electron microprobe detection limit of approximately 100 ppm. As nearly all map images of pyrite only show counts for iron and sulfur, these are omitted from this publication.

Laser Ablation ICP-MS Analysis

Pyrite grains observed in FE-SEM, such as those shown in appendix 1, were analyzed by laser ablation ICP-MS (LA-ICP-MS) spot analysis at the USGS Geology, Geophysics and Geochemistry Science Center in Denver, Colorado. A greater range of elements was determined by LA-ICP-MS than by electron microprobe mapping, including V, Cr, Mn, Co, Ni, Cu, Zn, As, Se, Mo, Ag, Cd, Sb, Au, Hg, Tl, Pb, and Bi (Kolker and others, 2021, table 5.1). Pyrites were ablated using spot sizes of 25 µm or 18 µm as necessary, based on the size of targeted grains. Instrument detection limits were obtained daily for each element determined, defined as three times the standard deviation of the blank (instrument response with the laser turned off), and converted to a concentration using the external calibration reference material. Detection limits in the sub-ppm to single ppm range were obtained for elements of interest such as V, Co, As, Sb, Hg, Tl, and Pb. Detection limits are generally lower for results determined at 25 µm than at 18 µm because a larger volume of material is ablated. Calibration uses USGS synthetic sulfide reference material MASS-1, which is homogeneous at a minimum of 20 µm (Wilson and others, 2002). In addition, as an internal standard, Fe in pyrite was fixed at its stoichiometric proportion (47 weight percent) and used to correct for yield variation from spot to spot. Response for ^{12}C was monitored during the runs as a qualitative measure of the relative contribution by the organic host during the analysis. This was not quantifiable due to the absence of carbon in the MASS-1 external calibration reference material. Results confirm that trace elements most commonly enriched in pyrite, such as arsenic, are generally present at concentrations below electron microprobe detection limits, and that mercury is commonly present in pyrite at ppm levels, demonstrating its enrichment in pyrite relative to values obtained for Illinois Basin whole coals.

Coal Maceral Analysis

Maceral analysis of coal was carried out at the Indiana Geological and Water Survey, Indiana University in Bloomington, Ind. The analysis utilized polished pellets prepared at the USGS coal petrography lab in Reston, Va., as described in the section "Sample Preparation." A reflected light microscope Leica DFC310 FX with an oil immersion objective of 50x was used to identify macerals in reflected and fluorescent light. A standard point-counting technique was used with 500 points counted on all samples; the counts were then recalculated into volume percentages. Maceral

identification followed established International Committee for Coal and Organic Petrology (ICCP) System 1994 methods (International Committee for Coal and Organic Petrology, 1998, 2001; Pickel and others, 2017). Data and results presented in Kolker and others (2021, tables 6.1 and 6.2), include coal maceral content, mineral matter (MM), and vitrinite reflectance (R_o), in percent. Maceral groups include vitrinite, liptinite, and inertinite.

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Appendix 1.   FE-SEM Images of Pyrite in Illinois Basin Coal Samples

Appendix 1 contains backscattered electron (BSE, and [or] BSE-ALL) images (figs. 1.1, 1.2, 1.3, 1.4, 1.6, 1.7, 1.8, and 1.9) and a secondary electron (SE, and [or] SE[L]) image (fig. 1.5) of pyrite (FeS$_2$) in Illinois Basin coal samples that were obtained by field emission scanning electron microscopy (FE-SEM).

USGS 10.0mm BSE-ALL 02/23/2018   10.0µm

Figure 1.1.   Image of a single pyrite framboid. The framboid is composed of an aggregate of approximately 2-µm pyrite crystals that exhibit the octahedral crystal structure of pyrite. The 10-µm-diameter size of the pyrite framboid is typical of Illinois basin coals. Image from sample IL-2, Herrin #6 coal. Abbreviations: BSE-ALL, backscattered electron-image; IL, Illinois; mm, millimeters; µm, micrometers (microns); USGS, U.S. Geological Survey.
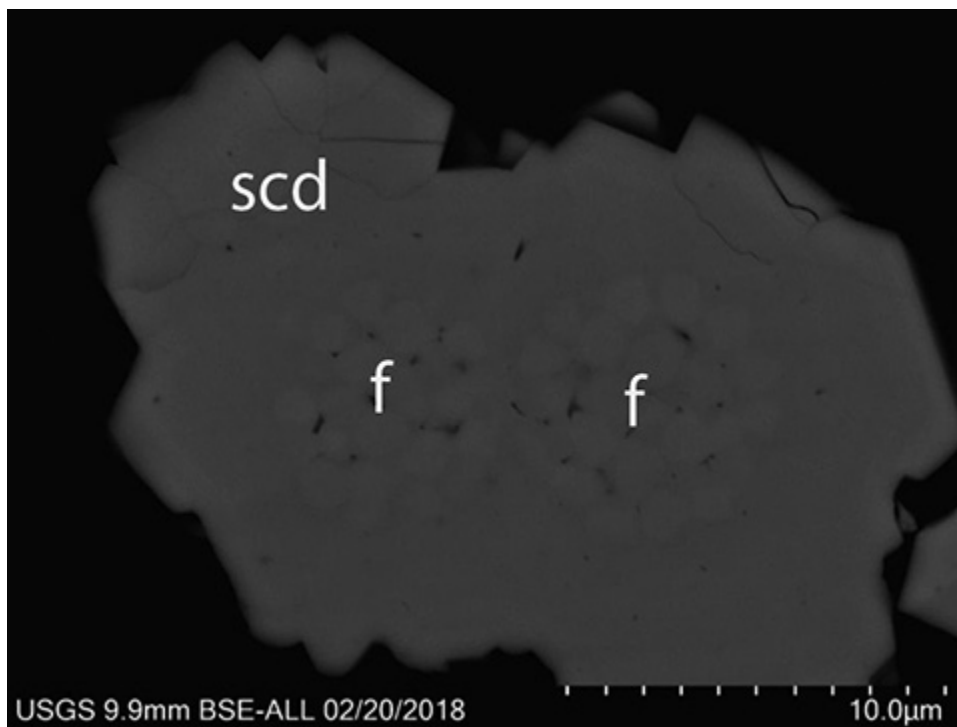
Figure 1.2.   Image of a cluster of pyrite framboids from sample IL-2, Herrin #6 coal. Abbreviations: BSE-ALL, backscattered electron-image; IL, Illinois; mm, millimeters; μm, micrometers (microns); USGS, U.S. Geological Survey.
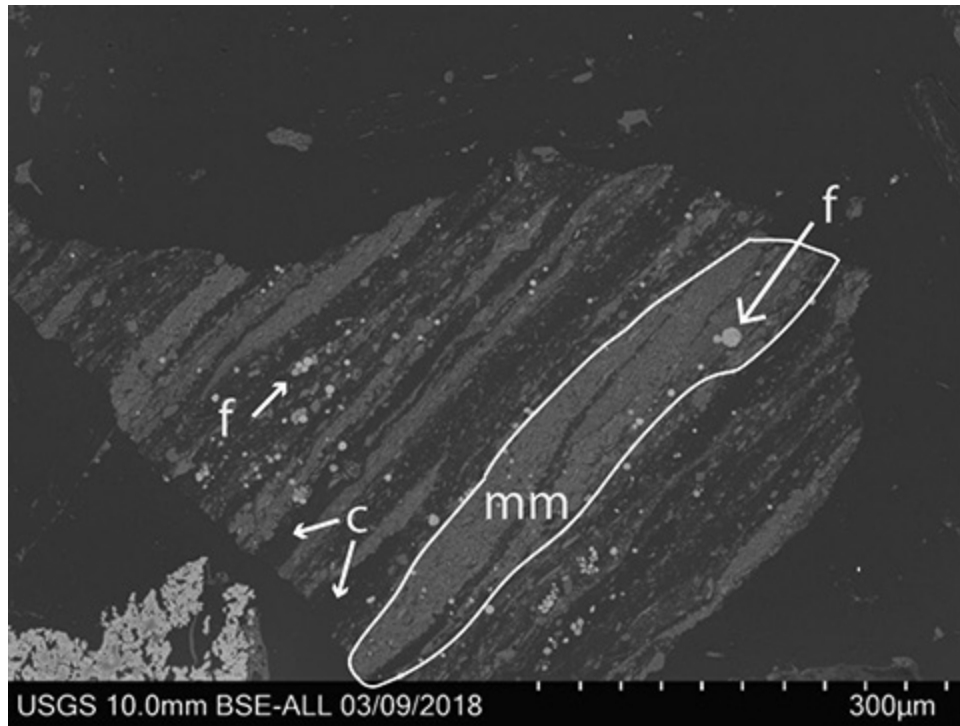

Figure 1.3.   Image of two small pyrite framboids (f) that are overgrown by secondary pyrite (scd) from sample IL-1, Herrin #6 coal. Abbreviations: BSE-ALL, backscattered electron-image; IL, Illinois; mm, millimeters; μm, micrometers (microns); USGS, U.S. Geological Survey.
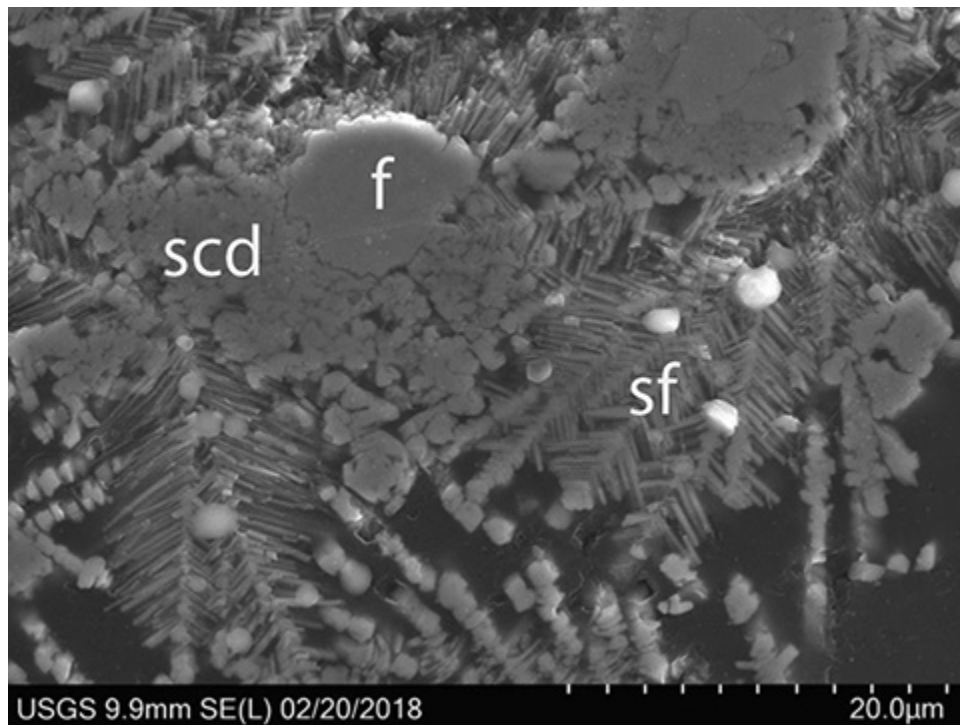
Figure 1.4. Image of a coal fragment hosting pyrite framboids (f) from sample IL-1, Herrin #6 coal. Dark and gray bands are coal (c) and mineral matter (mm), respectively. Abbreviations: BSE-ALL, backscattered electron-image; IL, Illinois; mm, millimeters; μm, micrometers (microns); USGS, U.S. Geological Survey.

Figure 1.5. Image of a pyrite framboid (f, upper-middle left) surrounded by secondary sulfides (scd) and possible sulfate salts (sf). The image is from sample IL-1, Herrin #6 coal. Abbreviations: IL, Illinois; mm, millimeters; SE(L), secondary electron image, μm, micrometers (microns); USGS; U.S. Geological Survey.
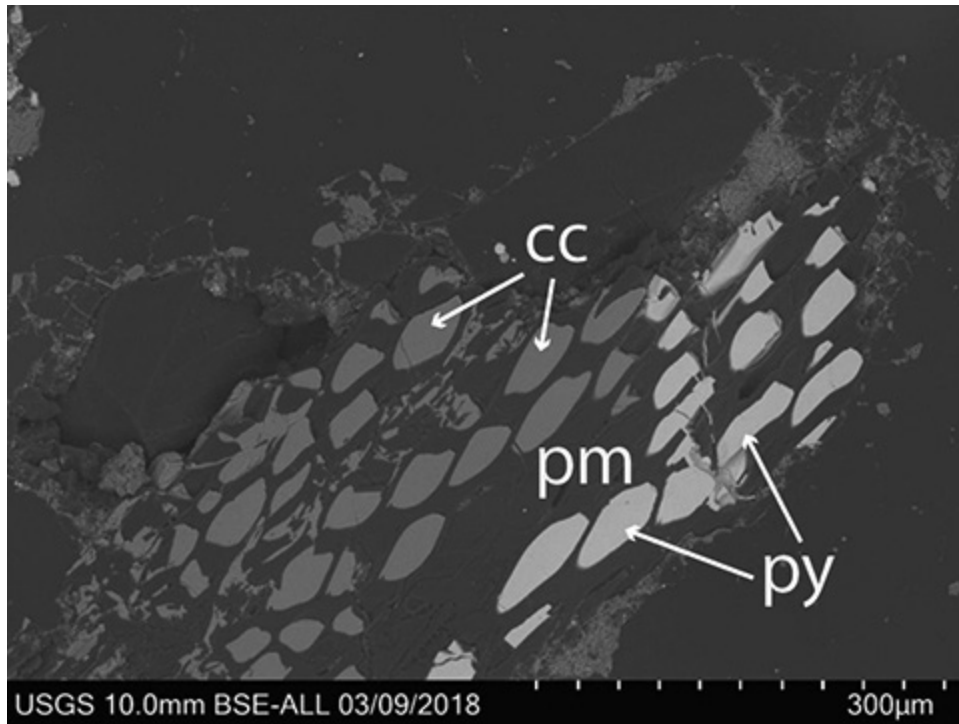
Figure 1.6.   Image of plant material (pm) with pyrite (py, high contrast) and calcite (cc, medium contrast) infilling. The image is from sample IL-1, Herrin #6 coal. Abbreviations: BSE-ALL, backscattered electron-image; IL, Illinois; mm, millimeters; μm, micrometers (microns); USGS, U.S. Geological Survey.

Figure 1.7.   Image of a fragment of a secondary pyrite cleat (high contrast) from sample IL-2, Herrin #6 coal. Abbreviations: BSE-ALL, backscattered electron-image; IL, Illinois; mm, millimeters; μm, micrometers (microns); USGS, U.S. Geological Survey.
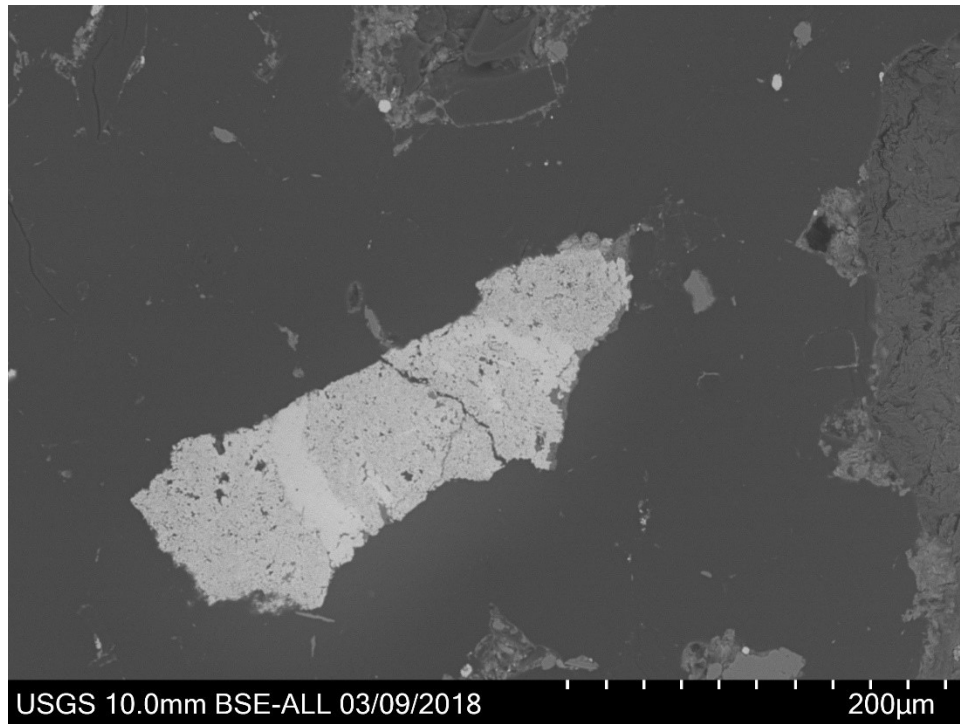
USGS 10.0mm BSE-ALL 03/09/2018   200μm

Figure 1.8.   Image of a fragment of a secondary pyrite cleat (high contrast) from sample IL-1, Herrin #6 coal. Abbreviations: BSE-ALL, backscattered electron-image; IL, Illinois; mm, millimeters; μm, micrometers (microns); USGS, U.S. Geological Survey.
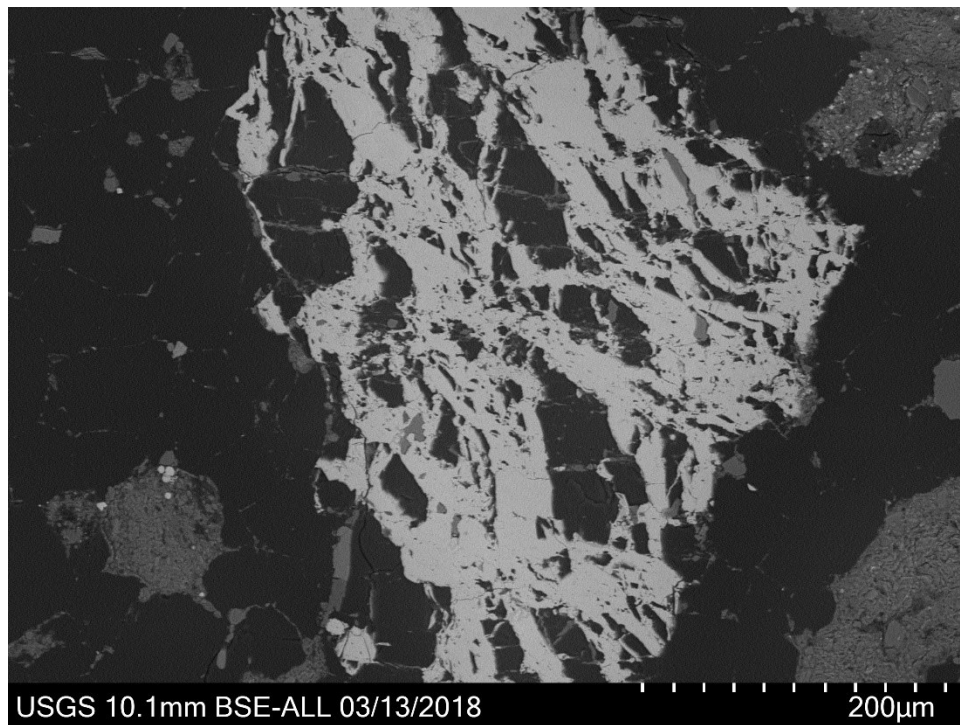

USGS 10.1mm BSE-ALL 03/13/2018   200μm

Figure 1.9.   Image of a fragment of a secondary pyrite cleat (high contrast) from sample IL-5, Herrin #6 coal. Abbreviations: BSE-ALL, backscattered electron-image; IL, Illinois; mm, millimeters; μm, micrometers (microns); USGS, U.S. Geological Survey.