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New mineral occurrences and mineralization processes: Wuda coal-fire gas vents of Inner Mongolia

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ABSTRACT

Five unique mineral assemblages that include the sulfates millosevichite, alunogen, anhydrite, tschermigite, coquimbite, voltaite, and godovikovite, as well as the halide salammoniac and an unidentified phase, according to X-ray diffraction and EDS data, were found as encrustations on quartzofeldspathic sand and sandstone adjacent to coal-fire gas vents associated with underground coal fires in the Wuda coalfield of Inner Mongolia.

The mineral assemblage of alunogen, coquimbite, voltaite, and the unidentified phase collected from the same gas vent, is documented for the first time. Coquimbite also occurs as rosettes secondarily nucleated on a cryptocrystalline mass of alunogen, coquimbite, voltaite, and the unidentified phase during storage in a sealed container at room temperature.

Field observations, analyses of vent gases, SEM images, and mineral compositions suggest that the sulfates millosevichite, alunogen, coquimbite, voltaite, godovikovite, and the unidentified phase, crystallized in response to a complex sequence of processes that include condensation, hydrothermal alteration, crystallization from solution, fluctuating vent temperatures, boiling, and dehydration reactions, whereas the halide salammoniac crystallized during the sublimation of coal-fire gas. Tschermigite and anhydrite formed by the reaction of coal-fire gas with quartzofeldspathic rock or by hydrothermal alteration of this rock and crystallization from an acid-rich aqueous solution.

Variations in the mineral assemblages found at five gas vents are possibly due to differences in coal-bed chemistry, exchange reactions involving coal-fire gas, and the composition of sediment, rock, and aqueous solutions prior to the exhalation of gas at the surface, as well as the temperature and cooling rate at a vent.

Few studies have addressed the interaction of coal-fire gas with sediment, rock, and aqueous solutions and the subsequent mineralization processes. Coal fires present opportunities for discovering rare and new mineral occurrences. These minerals have potentially important environmental significance and may be vectors for the transmission of toxins. Coal fires also provide insight for the recognition in the geologic record of preserved mineral assemblages that are diagnostic of ancient fires.

INTRODUCTION

Underground coal fires are reported from major coal-producing countries including China, the United States, India, Australia, South Africa, Russia, and Indonesia (Prakash and Gupta 1999; Prakash et al. 1999; Stracher 2002; Stracher and Taylor 2004). Gas vents, surface fissures lined by baked rocks, surface thermal anomalies, and dry and barren patches of land with locally high-reflection aureoles are typical expressions of underground fires (Gupta and Prakash 1998). The gas vents are commonly encrusted with materials associated with coal combustion. All of these surface features are important not only in indicating the location of underground fires, but also because they hold clues to the nature of the coal, intensity of burning, subsurface progression of combustion, and other important features that characterize a coal fire.

Mineralization processes at earth's surface associated with coal-fire gas initially involve the exhalation of the gas from vents or fissures (Lapham et al. 1980; Stracher 1995). At times, this is analogous to the exhalation-condensation process in which minerals form in solfataric or other fumarolic environments (Stoiber and Rose 1974). Studies linking the mineralization and chemistry of coal-fire gas are almost non-existent. One exception is the Centralia mine fire in Pennsylvania, where Stracher (2003) reported toxic emissions of CO (1000 ppm) and CO_2 (2200 ppm), in addition to numerous S-based and hydrocarbon gas compounds, associated with mineralization at numerous

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vents and fissures (Stracher et al. 2004).

This paper presents the results of X-ray diffraction (XRD), micro-Dumas carbon-nitrogen (MDCN) analyses, energy dispersive spectrometry (EDS), and scanning electron microscopy (SEM) used to identify and characterize solid encrustations derived from coal-fire gas exhaled from surface vents associated with underground coal fires in the Wuda coalfield in Inner Mongolia. In addition, chromatographic analysis of a gas sample collected from one of the vents is discussed.

As discussed below, the coal fires at Wuda are associated with human activities. Some would therefore argue that the phases we identified are not really minerals, because "natural processes" did not form them. However, in consideration of the fact that natural processes are sometimes defined to include those not occurring "in the laboratory" (Klein 2002, p. 4), the phases described herein are referred to as minerals. Natural processes that trigger coal fires include lightning, forest fires, and spontaneous combustion (Saraf et al. 1995; Stracher and Taylor 2004; Zhang et al. 2004a).

Geologic setting

The Wuda coalfield, 1250–1400 m in elevation, is part of the Eerduosi basin, a 10 × 3 km north-south trending syncline located in the northeastern Helan (Steed) Mountains (Gao 2003; Shanxi Coal Industry Association 2004). Three major mines, Wuhushan, Suhaitu, and Huangbaici, in addition to dozens of smaller mines in this coalfield, are a primary source of coking coal (Dai et al. 2002; Fig. 1). Wuhai (Haibowan) City, at the southeastern edge of the coalfield, serves the mining population and local tourist industry (Wuhai City Government 2004).

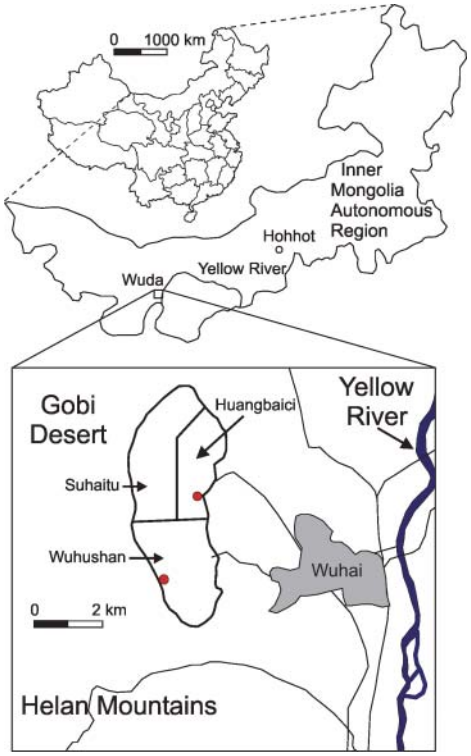


Figure 1. Wuda coalfield of Inner Mongolia, northern China showing the location of the Wuhushan, Suhaitu, and Huangbaici mines. Mineral samples from five coal-fire gas vents (Table 3) were collected within the circular areas shown in SE Huangbaici and SW Wuhushan.

According to Liu (1990), Dai et al. (2002), and Voigt (2003), the Wuda coalfield consists of 1500–2500 m of interbedded Pennsylvanian and Permian coal beds, sandstones, mudstones, and limestones, indicative of a Late Paleozoic migrating shoreline dominated by interacting fluvial, deltaic, and tidal processes. Nearly 80% of the coal mined in Wuda is extracted from ten beds within the Upper Pennsylvanian Taiyuan Formation. Additional coal is mined from seven beds in the Lower Permian Shanxi Formation (Shanxi Coal Industry Association 2004).

Wuda coalfield fires

Coal mining in Wuda began in 1958, and the first reported coal fire broke out in 1961 (Ma 2002). Since the 1980's, the number of coal fires in Wuda has increased together with the number of private mines, about 400 today, and unsafe mining practices continue unabated (Voigt 2003). Prior to 1989, fires were restricted to individual private coal workings. Subsequently, numerous fires coalesced, and currently 8.8% of the coalfield, about 3 km^2, is engulfed in surface and underground fires less than 100 m deep (Table 1). Although the origin of individual fires is unknown, spontaneous combustion is most likely the cause (Ma 2002) as a consequence of exothermic oxidation reactions between sulfide minerals in the coal and air circulating in coalbed joints and tunnels (Stracher and Taylor 2004).

Analytical methodology

Minerals

X-ray diffraction patterns combined with MDCN analyses and qualitative chemical analyses by EDS (Table 2) were used to identify minerals at five gas vents. All X-ray and MDCN data were obtained at the University of Georgia, Athens, whereas EDS data were obtained at the University of Nevada, Reno. The samples X-rayed were first ground to powder in air with an agate mortar and pestle. Each powder was then mounted on a 30 × 30 mm zero-background plate (ZBP), consisting of a single quartz crystal cut ~6° off the **c**-axis. The ZBP permits analysis of thin, small sample masses (~20–50 mg) by eliminating incoherent substrate scatter. Instead, the transmitted beam coherently scatters away from the detector.

All X-ray data were obtained using a Scintag XDS-2000 X-ray diffractometer. Instrumental conditions were Co$K\alpha$ radiation (generated at 40 kV and 40 mA), a 250 mm goniometer radius, 1°/2° primary/scattering slits, 0.5°/0.3° scattering/receiving slits, primary and receiving Soller slits, and a liquid N_2-cooled germanium solid-state detector. XRD data were collected using a scan rate of 0.5° 2θ per minute, with a step increment of 0.01° 2θ.

Diffractogram peaks were calibrated with reference to an external National Institute of Standards and Technology (NIST) reference silicon powder, SRM640a, and peak shapes were analyzed using profile-fitting software. Peak positions were best matched with patterns from the International Centre for Diffractions Data - Powder Diffraction File (ICDD-PDF). ICDD-PDF card numbers were assigned to our best-fit diffractograms after compositional corroboration by EDS analyses. The EDS analyses were obtained using a KEVEX solid-state detector mounted

Table 1. Wuda coal basin reserves of Inner Mongolia autonomous region of northern China

Coal reserves	Amount of coal (Mt)*
Prior to fire	636
Exploited as of 2001	280
Blocked from coal fire	100
Lost to fire as of 2001	3
Current rate loss/annum	0.1–0.2

Notes: Data from Ma (2002). For comparison, up to 200 Mt of coal per annum are lost to fires throughout China (Rosema et al. 1993; Anonymous 1999).
* Mt = million tons.

on a JEOL 840A scanning electron microscope, and digital images were acquired with the same instrument. Operating conditions were 10–15 kV and 1–30 nA, as necessary, to optimize image quality.

The total nitrogen content of solid samples was determined by MDCN analysis (Table 2) using a Carlos-Urba instrument. The analytical procedure is based on the transformation of solid matter to a gas phase by rapid flash combustion (via pure O_2) of a powdered sample (~10 mg) encapsulated in tin at 1200 °C in a furnace (Schroeder and Ingall 1994). The gaseous combustion products, including any N_2 and NO_x compounds, were extracted from the bottom of the furnace by a stream of He gas. The gases were then passed through a reduction column, equipped with granulated Cu wire at 600 °C, where NO_x is reduced to N_2 gas. The gases were then separated in a gas chromatograph, where N was detected in the mineral assemblages from vents 1 and 4. Mineral diffractograms, digital images illustrating the locations of EDS spectra taken on each sample for qualitative analysis, and MDCN data are in the repository of this journal.[1]

Vents 1–4 yielded multiple phases, whereas gas vent 5 yielded only salammoniac (Table 3). Electron microprobe analysis of individual phases in each sample was not possible because of sample deterioration during the preparation of polished thin sections.

[1] Deposit item AM-05-029. Deposit items are available two ways: For a paper copy contact the Business Office of the Mineralogical Society of America (see inside front cover of recent issue) for price information. For an electronic copy visit the MSA web site at http://www.minsocam.org, go to the American Mineralogist Contents, find the table of contents for the specific volume/issue wanted, and then click on the deposit link there.

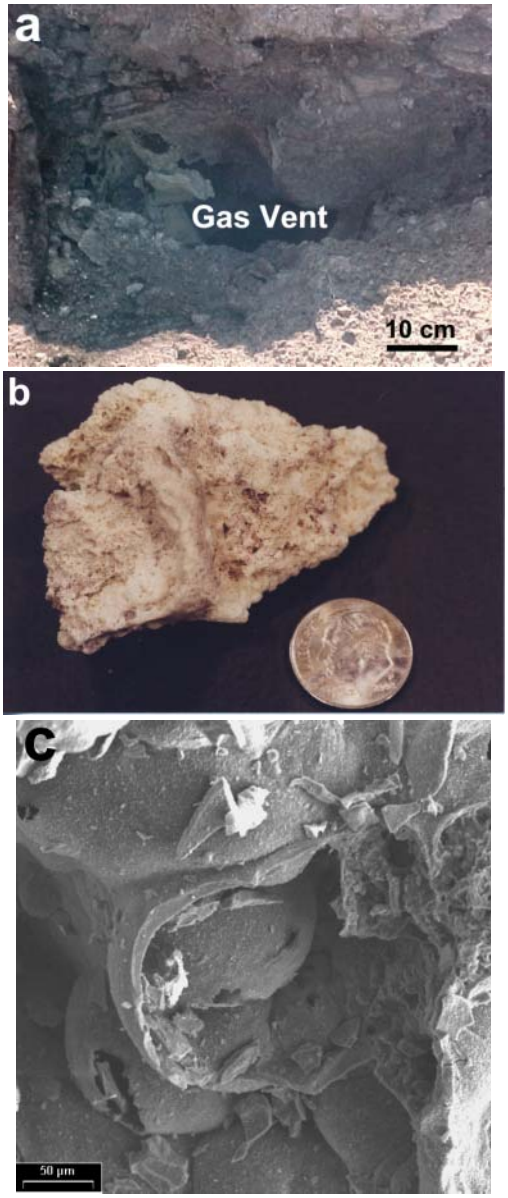


FIGURE 2. Alunogen, coquimbite, and godovikovite: gas vent 1. (**a**) Smoke and gas vent in quartzofeldspathic sandstone. (**b**) Alunogen, coquimbite, and godovikovite collected as a mass projecting inward from the vertical wall of the vent. (**c**) Cryptocrystalline mixture of alunogen, coquimbite, and godovikovite. Bright particles at the top and in the center of the SEM image are loose fragments from breakage of the sample. Bright area on bottom right is a region of strong contrast.

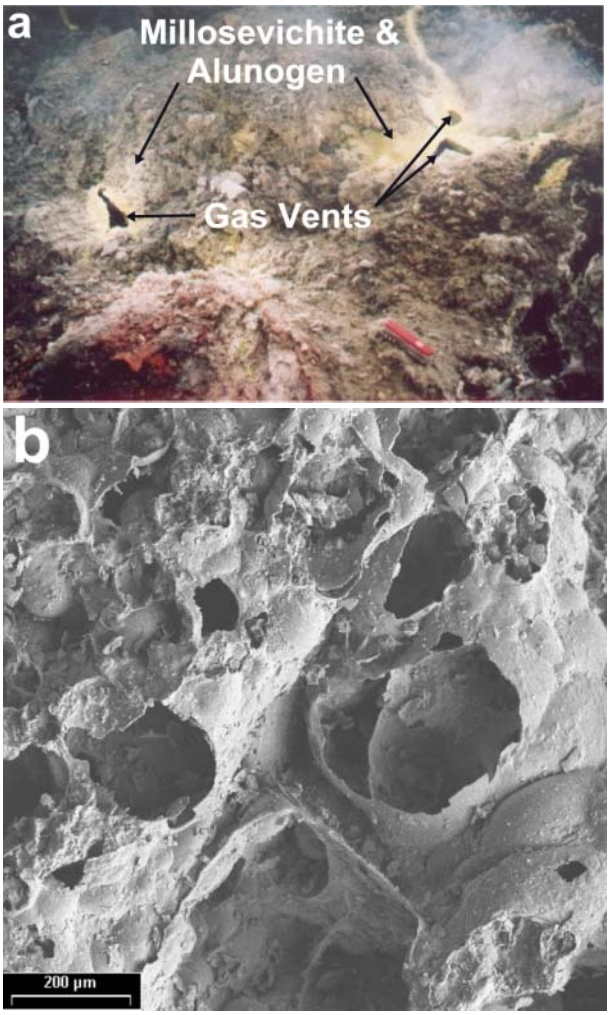


FIGURE 3. Millosevichite and alunogen: gas vent 2. (**a**) Smoke and yellow-white (light-colored) minerals on quartzofeldspathic sand. Distribution of minerals suggests that the gas moved in variable directions, ranging from left to right and upward in the photo. (**b**) Cryptocrystalline mixture of millosevichite and alunogen (SEM).

TABLE 2. Elements* in Wuda coal-fire minerals

Gas vent number	Al	Ca	Cl	Fe	H	Mg	Mn	N	O	K	S	Si
1	+	–	–	+	+	+	–	+†	+	+‡	+	–
2	+	–	–	+	+	+‡	–	–	+	–	+	–
3	+	–	–	+	+	+	+	–	+	+	+	–
4	+‡	+	–	–	+	–	–	+†	+	–	+	+
5	–	–	+	–	+	–	–	+	–	–	–	–

Notes: + = present; – = absent.
* Elements identified by EDS, except hydrogen, whose presence is inferred from mineral identification by XRD.
† MDCN analyses average 0.327 ± 0.02% and 0.427 ± 0.03% by weight at vents 1 and 4, respectively.
‡ Al, Mg, and K not detected in each area of sample analyzed.

TABLE 3. Wuda coal-fire minerals and associated field data

Gas vent number	Mineral X-ray identification	Sample color	Gas *T* (°C) ||	Universal transverse mercator coordinates (zone 48) X	Y	Z (m)
1	alunogen, coquimbite, and godovikovite (Fig. 2)*	yellow-white	260	639343 E	4373707 N	1294
2	millosevichite and alunogen (Fig. 3)†	yellow-white	110	641150 E	4376188 N	1249
3	alunogen, coquimbite, voltaite, and UP (Figs. 4 and 5)§	pink-white	269	641029 E	4376161 N	1241
4	tschermigite and anhydrite (Fig. 6)‡	yellow	239	641025 E	4376161 N	1240
5	salammoniac (Fig. 7)§	white	360	641162 E	4376193 N	1249

* Sample collected immediately inside edge of vent.
†1–5 cm from outside edge of vent.
‡ 1 cm from outside edge of vent.
§ 1–3 cm from outside edge of vent.
|| Temperature measured immediately inside gas vent.

TABLE 4. Wuda coal-fire gas analysis: Vent 1*

Compound	CO	CO_2	OCS	CS_2	CH_4	CH_3Cl	Ethane	Propane
ppmv†	42.69	86 189			2814			
pptv‡			4 057 734	329 770		2840	84 710 844	11 859 307
Detector	FID§	TCD||	MSD#	MSD	FID	FID/ MSD	FID	FID

* Partial analysis, complete gas chromatographic analysis in journal repository.[1] Temperature = 260 °C immediately inside gas vent.
† Parts per million by volume.
‡ Parts per trillion by volume.
§ Flame ionization detector.
|| Thermal conductivity detector.
Mass spectrometer detector.

Gas: extraction and chromatography

Eight gas samples were collected from the Wuda coalfield. Only one such sample was extracted from a vent (no. 1) encrusted with minerals related to coal-fire gas (Fig. 2 and Table 3). The apparatus used for collecting the gas consisted of a stainless steel tube about 65 cm long, attached to an evacuated two-liter electro-polished stainless steel canister. Threaded stainless steel fittings connected the tube and canister. After insertion of the tube several centimeters into the vent, a stainless steel bellows valve, attached to the canister, was opened slightly for about 30 seconds and then closed.

Gas chromatographic analysis of the sample (Table 4) was performed at the University of California, Irvine, using the method of Colman et al. (2001) and Barletta et al. (2002). After cryogenic pre-concentration at −196 °C using liquid nitrogen, the sample was vaporized using a hot water bath and split into five different column/detector combinations contained in three separate Hewlett-Packard (HP) 6890 gas chromatographs. Two electron capture detectors (ECD), a mass spectrometer detector (MSD), and two flame ionization detectors (FID) were used to analyze a variety of compounds including halocarbons and alkyl nitrates (ECD), sulfur- and halogen-based compounds, as well as selected hydrocarbons (MSD), and the non-methane hydrocarbons in addition to CH_4 and CO (FID). CO was methanized by reacting it with H_2 prior to analysis. For CO_2 analysis, an HP 5890 gas chromatograph equipped with a thermal conductivity detector was used. N_2, O_2, and the sulfur oxides, detectable in the field and always present in coal-fire gas (Stracher and Taylor 2004), were not quantifiable due to laboratory analytical conditions. H_2O was not quantified due to its tendency to condense in the canister.

ANALYTICAL RESULTS

Mineralogy at gas vents

Mineral identification was based on a combination of XRD, MDCN, and EDS analyses. The minerals identified, their colors, the gas temperature immediately inside each vent, and the location of each vent are all presented in Table 3.

All the minerals identified are sulfates or hydrated sulfates, except for the halide salammoniac and an unidentified phase referred to herein as "UP," based on its X-ray diffraction pattern and EDS data. Millosevichite, alunogen, anhydrite, tschermigite, coquimbite, voltaite, godovikovite, and salammoniac were previously identified elsewhere in association with coal combustion, but with little attempt to link the minerals with the rank or chemistry of the coal. The mineral assemblage of alunogen, coquimbite, voltaite, and UP at vent 3 is the first known occurrence of this assemblage at a gas vent. In addition, UP may be a new mineral. The sample from vent 3 was too small to collect the requisite chemical analysis and crystallographic data to define the mineral structure of UP.

The mineral assemblages in the Wuda coalfield are unique. For the first time, these assemblages were found in proximity to one another (Fig. 1) and formed by the same geologic process, a coal fire. The distances between samples 1–4, collected in SE Huangbaici, ranged from 5 to 140 meters. Sample 5 is from SW Wuhushan.

Standard mineralogical data (Gaines et al. 1997) for these minerals and their type localities (when known) are presented below. Also presented are additional reported occurrences of these minerals in association with coal and sulfide ore combustion, volcanic, hydrothermal, and evaporative processes, and acid mine drainage.

Godovikovite (hexagonal), $NH_4(Al,Fe^{3+})(SO_4)_2$, was found at

vent 1 (Fig. 2). Named after the Russian mineralogist Aleksandr A. Godovikov, the mineral was discovered in 1988 in burning coal mine dumps at its type locality, the Chelyabinsk coal basin, near the town of Kopeisk, in the southern Ural Mountains. According to Jambor and Grew (1990), it is a primary component of sulfate crust, formed by the reaction of dump material with sulfuric acid.

Millosevichite (hexagonal), $(Al,Fe^{3+})_2(SO_4)_3$, was found at vent 2 (Fig. 3). The discovery of millosevichite (also called lippite) at Wuda confirms its association with coal combustion. Brownfield et al. (1999) and Kolker et al. (2000) noted its occurrence with anhydrite as a coal combustion by-product in fly ash at a Kentucky power plant. First discovered in 1913, the type locality of millosevichite is Alum Grotto, Vulcano Island, Eolie (Lipari) Islands, Messina Province, Sicily (Gaines et al. 1997; Perroud 2004). More recently, it has been found on Mutnovsky Volcano in Kamchatka, associated with hydrothermal and fumarolic activity (Gordeev 2005).

UP was found at vent 3 (Figs. 4 and 5). The X-ray diffractogram of UP shown in Figure 5 and the presence of O, Al, S, and other elements found in the vent 3 assemblage (Table 2) are not correlative with any mineral in the ICDD-PDF database. Consequently, UP currently remains unidentified.

Voltaite (isometric), $K_2Fe^{2+}_5Fe^{3+}_3Al(SO_4)_{12}\cdot 18H_2O$, was found at vent 3 (Figs. 4 and 5). Although rare, voltaite (also called voltait or voltaita) occurs in diverse geologic settings. Pelloux (1927) noted its occurrence as a fumarolic deposit at Mount Vesuvius in Italy, and Anderson (1927) and Lausen (1928) described its occurrence at the United Verde Copper Company in Arizona as the condensation product of gas from burning sulfide ore deposits. Voltaite also is known from numerous coal mines in Germany including the Carolaschacht Mine dump in Freital, Döhlen Basin, Saxony, where it formed as a precipitate from aqueous solutions that reacted with coal-fire gas (Witzke 1990). Nordstrom and Alpers (1999) documented its occurrence in association with acidic mine waters. Discovered in 1792 in solfatara deposits near Naples (Anderson 1927), voltaite was formally named in 1841 after the Italian Physicist, Count Alessandro Volta. The type locality is Pozzuoli, Naples Province, Campania, Italy (EUROMIN Project 2004; Ralph 2004).

Coquimbite (trigonal), $Fe^{3+}_2(SO_4)_3\cdot 9(H_2O)$, was found at vents 1 and 3 (Figs. 2, 4, and 5). The discovery of coquimbite (also called coquimbit, coquimbita) at Wuda is possibly the second confirmed occurrence of this mineral in association with a coal fire, the first being from the mine-waste dump fires in the Chelyabinsk coal basin in Russia (Chesnokov et al. 1998; de Graff 2004). In the vent 3 sample, apparently primary coquimbite was detected as a phase within a cryptocrystalline mass that includes alunogen, voltaite, and UP (Fig. 4b). After storage at room temperature for four months in a sealed container, secondary coquimbite rosettes nucleated within and on the cryptocrystalline mass (Figs. 4c and 4d). Anderson (1927) described coquimbite as an alteration product of voltaite from the United Verde fire mentioned above, whereas Lausen (1928) described it as a condensation product of the fire, deposited in rock cavities and on charred wood. In consideration of these two occurrences, coquimbite in the cryptocrystalline mass from vent 3 may or may not be an alteration product of voltaite, whereas the coquimbite rosettes likely are alteration products of voltaite. Discovered in 1841, the type locality is Coquimbo Province, Chile (EUROMIN Project 2004; Ralph 2004). Its numerous occurrences include the Cretaceous bituminous-coal deposits in Germany (Dill and Pöllman 2002), Carlsbad Caverns, New Mexico as a speleogenetic by-product (Polyak and Provencio 2001), and the banks of the Rio Tinto River in Spain as an evaporite from sulfate-rich river water produced by the oxidation of pyrite (Buckby et al. 2003).

Alunogen (triclinic), $Al_2(SO_4)_3\cdot 17H_2O$, was found at vents 1–3 (Figs. 2, 3, 4, and 5). It occurs globally in diverse geologic settings. Lapham et al. (1980) noted its occurrence at the Glen Lyon anthracite mine fire in eastern Pennsylvania in association with hydrothermal and evaporative processes. Carlson and Whitford (2002) found alunogen as an efflorescence derived from the evaporation of sulfate-rich pore water in Late Devonian shales in northern Ohio. Its association with hydrothermal and gas activity is exemplified by its occurrences on Mutnovsky Volcano in Kamchatka (Gordeev 2005) and at fumarolic vents on Soufriere Hills Volcano, Montserrat (Boudon et al. 1996).

Anhydrite (orthorhombic), $CaSO_4$, was found at vent 4 (Fig. 6). In addition to its association with millosevichite in Kentucky fly ash, as noted above (Kolker et al. 2000), anhydrite was found as an alteration product surrounding fumaroles in joints in burning oil shale on the Dorset Coast, near Kimmeridge Bay, United Kingdom. Sulfur dioxide from burning oil in the shale is thought to react with calcite in calcareous shale to form anhydrite (Cole 1975; West 2001). Discovered in 1804, the type locality is Hall, Innsbruck, Austria (Ralph 2004). Numerous other occurrences of anhydrite, unrelated to burning, are recorded in the literature.

Tschermigite (cubic), $(NH_4)Al(SO_4)_2\cdot(H_2O)_{12}$, was found at vent 4 (Fig. 6). Discovered in 1853, the type locality of tschermigite is the abandoned Schoeller Coal Mine, Cermiky (Tschermig), Kaaden, Bohemia in the Czech Republic (EUROMIN Project 2004; Ralph 2004). Lapham et al. (1980) noted its occurrence at the Glen Lyon and Wanamie anthracite-mine fires in eastern Pennsylvania in association with hydrothermal and evaporative processes.

Salammoniac (isometric), NH_4Cl, was found at vent 5 (Fig. 7). Salammoniac occurs globally as a sublimation product of coal-fire gas and fumes from volcanic vents. Alexander the Great is thought to have found it in association with underground coal fires in Tadzhikistan (Amethyst Galleries, Inc. 2004). According to Lapham et al. (1980), the minimum time for the sublimation of salammoniac at the Kehley's Run anthracite-mine fire in eastern Pennsylvania was less than 24 hours. Encrustations on volcanic rocks such as those on Mount Vesuvius, Italy and Paricutin Volcano, Mexico are common (Gaines et al. 1997). The mineral also has been identified as a condensation product around fumaroles associated with burning oil shale near Kimmeridge Bay on the Dorset Coast (Cole 1975; West 2001).

EDS and XRD comparison

The EDS data in Table 2 for the most part are consistent with the stoichiometric formulae presented for the "identified" minerals in Table 3. There are, however, some variations. For example, Mg occurs in the EDS spectra of the samples from vents 1–3, but not in any of the stoichiometric formulae. The same is true for

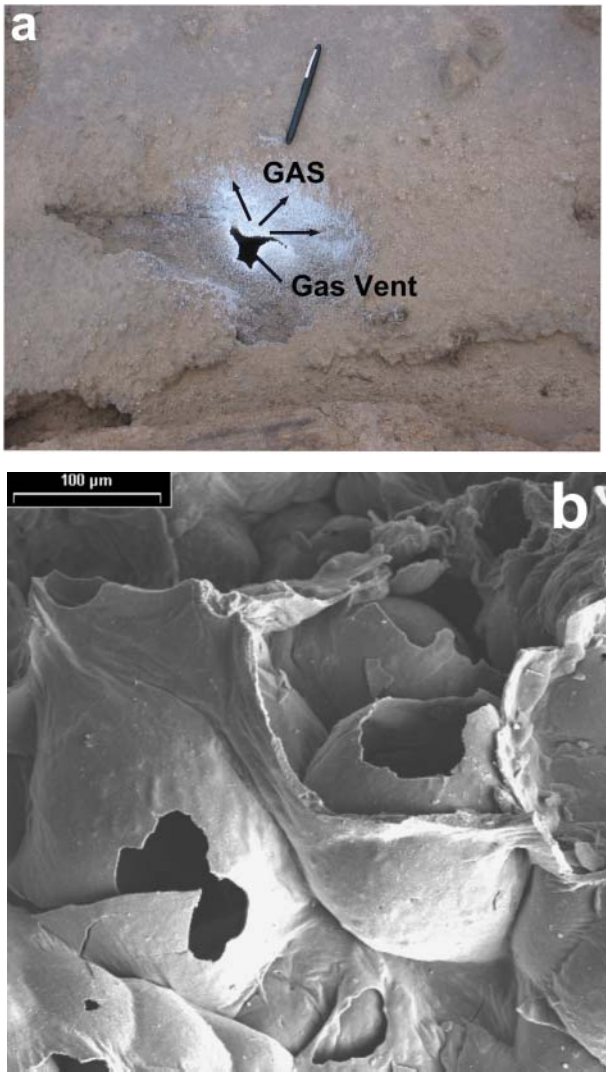


FIGURE 4. Alunogen, coquimbite, voltaite, and UP: gas vent 3. (**a**) Distribution of minerals on quartzofeldspathic sand suggests mineralization occurred in response to a gas moving toward the upper right in the photo. (**b**) Cryptocrystalline mass of alunogen, coquimbite, voltaite, and UP from gas vent 3 (SEM). (**c**) Coquimbite rosettes (small bumps) secondarily nucleated on the cryptocrystalline mass illustrated in b (SEM). (**d**) Magnified image of c illustrating the rosettes (SEM). See also Figure 5.

K and Mn, which occur in the spectra of the samples from vents 1 and 3, respectively, but not in any of the mineral formulae. If voltaite altered to coquimbite in the sample from vent 1, then at least some of the K was contained in the former mineral.

At least two explanations exist for the variations between XRD (bulk-sample) analyses and EDS (point) analyses. The EDS detection of Mg, Mn, and K may indicate the presence of a phase too sparse in the vent assemblages to be detected by XRD. On the other hand, Anderson (1927) noted the presence of Mg in voltaite from the United Verde Copper Company. Consequently, at vent 3, Mg^{2+} and even Mn^{2+}, may instead be in cationic-substitutional solid solution with Fe^{2+} in voltaite.

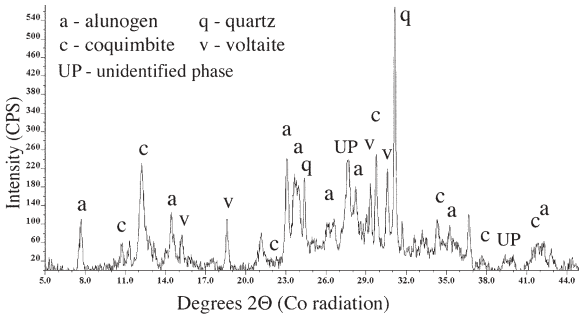


FIGURE 5. X-ray diffractograms for alunogen, coquimbite, UP, quartz, and voltaite: gas vent 3. The UP diffractogram and EDS data (Table 2) do not correlate with any known phases in the ICDD-PDF databank. Consequently, UP remains unidentified (see text). Quartz, part of the quartzofeldspathic substrate, was picked up during collecting. See also Figure 4 and Table 3.

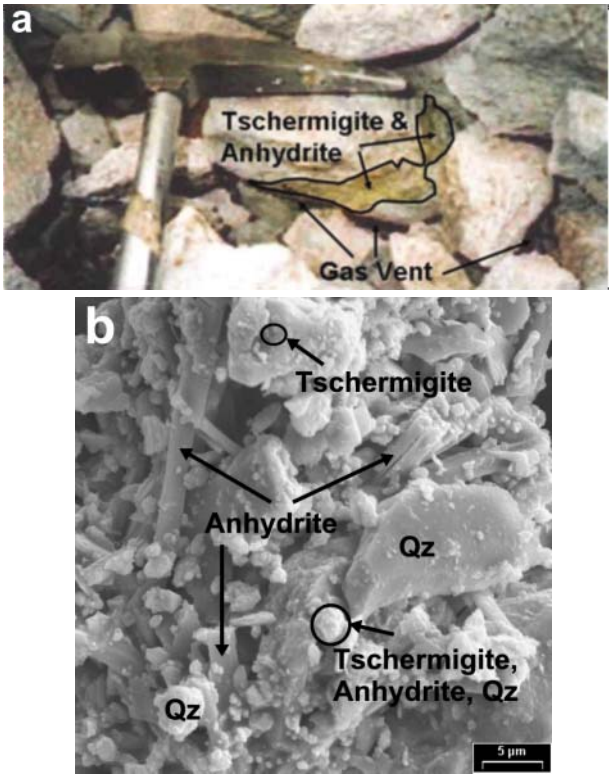
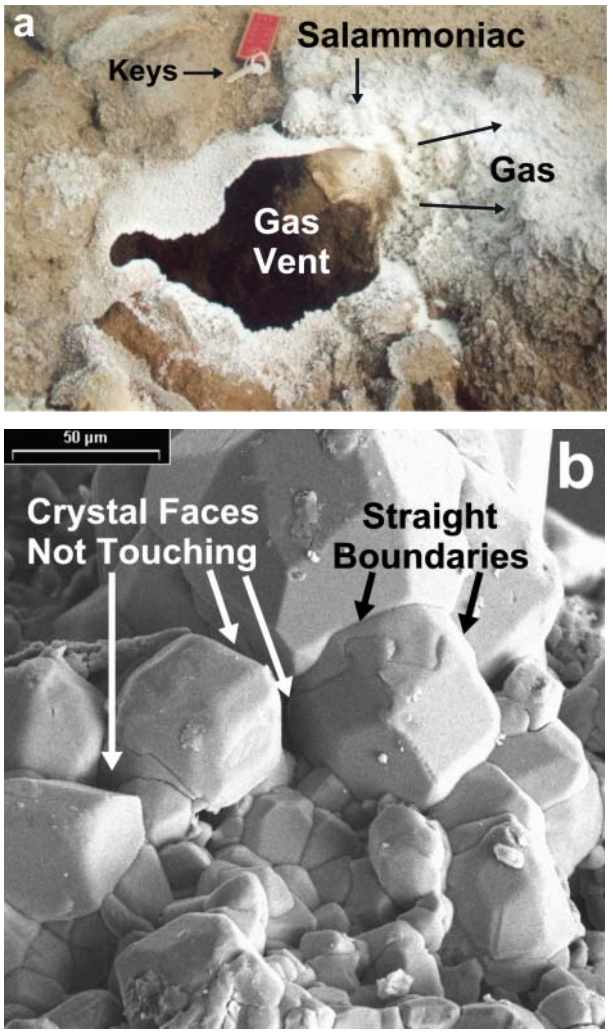


FIGURE 6. Tschermigite and anhydrite: gas vent 4. (**a**) Mineralization associated with coal-fire gas occurs in the area outlined on quartzofeldspathic sandstone, originally facedown over the vent. (**b**) SEM image shows euhedral laths of anhydrite identified by XRD and EDS analyses. The smallest crystals are tschermigite or mixtures of tschermigite, anhydrite, and quartz (Qz). The intermediate to large anhedral quartz crystals also occur with a kaolin-group mineral and K-feldspar, identified in a different cut from the same sample. These were picked up from the quartzofeldspathic substrate during collecting.



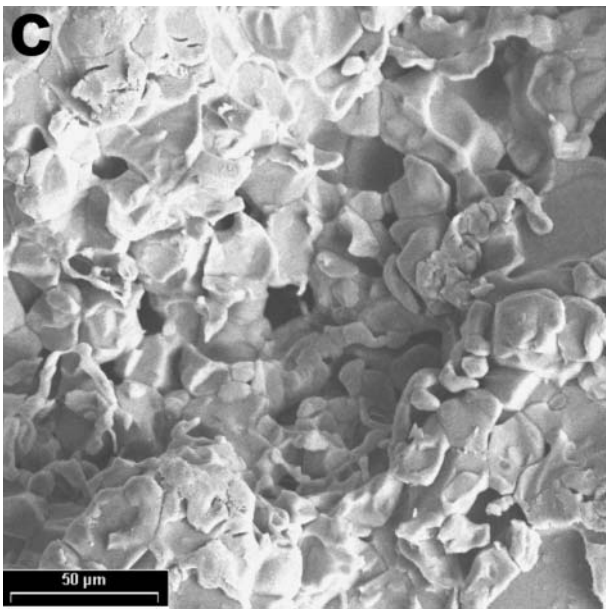


FIGURE 7. Salammoniac: gas vent 5. (**a**) Distribution of salammoniac (white mineral) on quartzofeldspathic sand suggests mineralization occurred in response to a gas moving primarily toward the right in the photo. (**b**) Crystal faces of euhedral salammoniac do not touch or are in contact along straight-grain boundaries, suggesting sublimation from a gas, as discussed in the text (SEM). (**c**) Cryptocrystalline salammoniac from the same sample as b; finer-grained texture is possibly due to a faster sublimation rate (SEM).

Gas chemistry and metallic ions

Table 4 lists the fractional analysis of a gas sample extracted from vent 1. Fifty-five compounds were found in the gas.[1] Some of the compounds contain one or more of the elements S, O, and H, all found in the mineral assemblage alunogen, coquimbite, and godovikovite encrusting the inside margin of the vent (Fig. 2). Although N, which occurs in godovikovite, was not quantified, this element is a common constituent of coal-fire gas (Stracher and Taylor 2004). None of the metallic cations in the stoichiometric formulae presented for the assemblages at vents 1–4 occur in the analyses of the eight gas samples collected at various locations in the Wuda coalfield.

Lausen (1928) claimed that metallic ions in the hydrous sulfates lining surficial cracks associated with the United Verde mine fire were transported to the surface as gaseous oxides or molecules of the sulfates. However, these do not occur in the gas analyses reported by him. Furthermore, we know of no published coal-fire gas analyses (for example, Chamberlain and Hall 1973; Kim 1973; Giardino 1999), including the eight gas samples from Wuda, where metal-bearing phases were ever found. With the possible exceptions of Hg, As, and Se, the ambient heat energy necessary to keep metal-bearing phases volatilized would result in temperatures exceeding those of any known coal fire.

In consideration of the above, as well as extensive field observations of other rocks and sediments altered to varying degrees by coal-fire gas and encrusted by fumarolic deposits, it appears that prior to mineralization at vents 1–4, metallic cations were extracted from quartzofeldspathic sediment and rock when the gas or a liquid condensed from the gas, reacted with these materials.

Mineralization processes

In the field, the distribution of minerals adjacent to gas vents 1–5 suggests that mineralization occurred when the gas cooled and condensed at the surface. This is true for the salammoniac found at vent 5, as discussed below. However, the mineralization processes at vents 1–4 were more complex. The minerals collected there contain metallic ions likely acquired from the sediment and rock they nucleated on. Although the temperatures of and the mineralization sequences at vents 1–4 are unknown, field observations, SEM images, and the metallic ions in the mineral assemblages from these vents provide evidence for the mineralization processes that occurred there. In addition, it is plausible that the minerals at a particular vent formed by multiple processes.

Gas vents 1–3

The sample from vent 1 was collected as a mass projecting inward from the vertical wall of the vent (Figs. 2a and 2b). This apparent inward growth is in accord with a cooling gas that initially condensed on or reacted with the wall of the vent, prior to exhalation of the remaining gas at the surface. The distribution of fumarolic minerals adjacent to vents 2 and 3 (Figs. 3a and 4a) is indicative of a mineralization process that initially involved either simple condensation of a cooling gas or reaction of a gas with the surface of the substrate it passed over. In either case, the gas moved in a preferential direction at vents 2 and 3. Moreover, Figures 2c, 3b, 4b, and 4c reveal that the mineral assemblages at vents 1–3 are vesicular-globular masses. This texture is reminiscent of that developed in some shallow gas-charged intrusions and lava flows (Williams et al. 1982, p. 64–65) during cooling and crystallization. By analogy, the vesicles in the assemblages from vents 1–3 suggest that a fluid phase was released in association with the crystallization of these assemblages.

These observations and the fact that the mineral assemblages at vents 1–3 contain metallic ions likely acquired from the substrates on which they were collected, indicate that two possible mineralization processes occurred at these vents: (1) Coal-fire gas condensed as a liquid on the substrate at each vent, perhaps as microscopically thin layers. If so, the resulting aqueous solution, acid-rich because of the N, S, and C compounds and water vapor originally in the gas, hydrothermally altered metal-bearing minerals in the substrate, such as the feldspars, by leaching metallic ions into solution. As the solution cooled, the mineral assemblages crystallized; (2) The gas reacted directly with the substrate, extracting metallic cations prior to cooling and crystallization of the vent assemblages.

Origin of vesicular texture

Two processes, each associated with fluctuating vent temperatures, may account for the vesicles in the fumarolic deposits that crystallized at each vent. Based on field measurements, we have found such fluctuations to be common at the openings of and in the substrate adjacent to coal-fire gas vents.

A vesicular texture would result, for example, if during the formation of a vent assemblage from a cooling acid-rich aqueous solution, there was a momentary temperature reversal, causing water to boil and escape from the crystallizing solution. On the other hand, regardless of the actual mineral formation process, a momentary temperature increase subsequent to the formation of a vent assemblage could trigger dehydration reactions, "blowing holes" in the assemblage as water vapor or liquid water escaped. If so, then dehydration reactions suggested by the mineral assemblages at vents 1–3 and the stoichiometric formulae presented previously, include:

$$\underset{\text{Tschermigite}}{(NH_4)Al(SO_4)_2\cdot(H_2O)_{12}} \rightarrow \underset{\text{Godovikovite}}{NH_4(Al)(SO_4)_2} + 12H_2O\uparrow \quad \text{Vent 1}$$

$$\underset{\text{Alunogen}}{Al_2(SO_4)_3\cdot 17H_2O} \rightarrow \underset{\text{Millosevichite}}{Al_2(SO_4)_3} + 17H_2O\uparrow \quad \text{Vent 2}$$

$$\underset{\text{Voltaite}}{K_2Fe^{2+}_5Fe^{3+}_3Al(SO_4)_{12}\cdot 18H_2O} \rightarrow \underset{\text{Coquimbite}}{Fe^{3+}_2(SO_4)_3\cdot 9(H_2O)} + XH_2O\uparrow + \quad \text{Vent 3}$$

$$K_2AlFe^{2+}_5\,Fe^{3+}(SO_4)_9\cdot YH_2O\,(?)$$

If tschermigite dehydrated to godovikovite at vent 1, the reaction went to completion because the sample does not contain tschermigite. If millosevichite at vent 2 formed by the dehydration of alunogen, the reaction did not go to completion because the sample from this vent contains both minerals. The incomplete breakdown is explained by a temperature decrease subsequent to the increase that triggered dehydration. The fluctuation accounted for by changing combustion or atmospheric conditions or, in this case, even removal of the assemblage from the vent. The actual dehydration reaction for voltaite at vent 3 is unknown.

Larsen (1921, p. 155) and Anderson (1927) noted coquimbite as one of the possible decomposition products of this mineral. Although the reaction as written is balanced when X + Y = 9, the K-bearing product of the reaction, $K_2AlFe^{2+}_5Fe^{3+}(SO_4)_9{\cdot}YH_2O$, is hypothetical, serving merely to balance the reaction. Besides coquimbite, the additional unidentified-reaction product could be UP, detected in the vent 3 assemblage by XRD.

Gas vent 4

The mineral assemblage from vent 4 was collected from the bottom surface of a slab of quartzofeldspathic sandstone hanging over the vent. A piece of the rock broken from the slab was partially encrusted with minerals on the face exposed to coal-fire gas (Fig. 6a). XRD revealed the presence of tschermigite and anhydrite in the sample (Table 3). Micro-Dumas analysis confirmed the presence of N (Table 2) in further confirmation of tschermigite. XRD also indicated the presence of quartz, a kaolin-group mineral, and K-feldspar, likely acquired from the sandstone during the collection process (Fig. 6b). The specific kaolin-group mineral (kaolinite, dickite, or halloysite) could not be determined because of low abundance and preferred orientation. The best diffractogram fit for K-feldspar is sanidine, but microcline is also possible.

The sample from vent 4 has a granular texture and consists of poorly sorted and unconsolidated mineral grains (Fig. 6b). EDS spot analyses of euhedral laths in the assemblage revealed the presence of Ca, S, and O. The laths are therefore inferred to be the anhydrite, identified by XRD. The smallest grains are tschermigite or mixtures of tschermigite, anhydrite, and quartz inferred from the Al, S, Ca, O, and Si in the EDS spectra, N in the MDCN analysis, and the XRD data. The intermediate to largest grains are anhedral quartz. Both tschermigite and anhydrite contain metallic cations likely acquired from the substrate the sample was collected from. Unlike the assemblages from vents 1–3, the vent 4 assemblage shows no evidence of a fluid-phase that escaped during crystallization. On the other hand, tschermigite is hydrous and anhydrite is not. This suggests two possible mineralization processes for the vent 4 assemblage: (1) Gaseous N, S, and C compounds together with water vapor, reacted with Ca- and Al-bearing minerals in the sandstone, such as feldspar, and produced tschermigite and anhydrite. Some K-feldspar possibly then altered to the kaolin-group mineral; (2) The mineralization process occurred because, like vents 1–3, coal-fire gas condensed to an acid-rich aqueous solution that hydrothermally altered the quartzofeldspathic substrate, leaching Ca and Al ions into solution. As the solution cooled, tschermigite and anhydrite crystallized.

Gas vent 5

The distribution of salammoniac on quartzofeldspathic sand adjacent to vent 5 (Fig 7a) is, as at vents 1–4, indicative of either mineralization associated with condensation from a cooling gas or of mineralization that occurred in response to a gas reacting with the substrate over which it passed. Because salammoniac contains no metallic ions, and the elements in its formula occur in all eight-gas samples from Wuda, the composition of salammoniac seems independent of any gaseous or liquid reactions with the depositional substrate. This fact, in addition to the observation that salammoniac in the sample is characterized by euhedral isometric crystals with adjacent faces that either do not touch or are in contact along straight-grain boundaries (Fig. 7b), suggests that salammoniac crystallized in a non-restrictive environment due to sublimation, analogous to the exhalation-condensation mechanism by which some minerals form in some solfataric or other fumarolic environments (Stoiber and Rose 1974).

Variations between gas vent assemblages

At least three explanations exist for the differences in mineral assemblages at gas vents 1–5. The first one is differences in the chemistry of coal in the subsurface. Samples of coal from the subsurface in the vicinity of vents 1–5 are unavailable to the authors. However, partial analyses presented by Ma (2002) for 12 beds, from undisclosed locations in the Wuda coalfield, reveal that the coal contains from 0.59 to 3.42 wt% S (sum of organic and non-organic) and from 0.87 to 1.12 wt% H_2O. There may be variations among other elements in the coal. The second explanation is chemical diversity within and among the sandstones, mudstones, carbonates, and possibly among aqueous solutions encountered by the gas en route to the surface. The solutions may reside in subsurface joints, along fault planes, or in local saturation zones perched above impermeable rocks such as mudstones. Exchange reactions between the gas, rocks, and solutions would continuously change the chemistry of gas en route to the surface and therefore, influence the mineral assemblages that crystallized in association with the gas. The third explanation is differences in the temperature and cooling rate at the opening of each vent, both of which fluctuate in response to changes in subsurface combustion and atmospheric conditions. They are important in determining whether condensation, dehydration, or some other process associated with coal-fire gas will occur (Stracher 1995).

Discussion

In this study, field observations, SEM images, and X-ray, EDS, MDCN, and gas analyses were used to propose several processes responsible for the formation of mineral assemblages found in the Wuda coalfield. These processes include condensation, hydrothermal alteration, crystallization from aqueous solution, fluctuating vent temperatures, boiling, dehydration reactions, sublimation, and the reaction of coal-fire gas with country rock. Additional studies are necessary to identify the chemical reactions that occur during these processes. Once identified, the reactions will provide crucial information about the origin of specific "coal-fire mineral assemblages" by revealing the requisite gas, aqueous solution, and substrate chemistry necessary for the formation of a specific mineral assemblage. In addition, such studies coupled with the techniques utilized herein may reveal whether the same mineral assemblage is always associated with the same mineralization processes. Indeed, the Wuda study demonstrates that investigating coal fires may result in the discovery of rare minerals or new mineral occurrences. Furthermore, the potential for discovering new minerals exists, considering the breadth of possible variations in both the chemistry of coal-fire gas and the substrates encountered by the gas during exhalation at the surface.

In addition to their uniqueness, an important reason for

studying coal-fire mineral assemblages is their environmental significance. Stracher (1995) derived a *P-T* stability diagram for the condensation of orthorhombic sulfur from anthracite gas associated with the Centralia, Pennsylvania mine fire. The significance of any such stability diagram is that it serves as an environmental indicator of conditions that tend to favor the formation of the solid by-products of coal combustion that contribute to soil and water pollution as opposed to the absorption of pollutants into the atmosphere.

Clinker, rock baked and metamorphosed by burning coal, is evidence for the occurrence of natural burning coal fires in the geologic past, including those during the Pliocene (Coates and Heffern 2000; Heffern and Coates 2004) and Pleistocene (Zhang and Kroonenberg 1996; Kroonenberg and Zhang 1997). Regardless, opencast and deep-coal mining since the industrial revolution has promoted the worldwide proliferation of coal fires, some burning for decades (Stracher 2002). The environmentally catastrophic effects subsequent to industrialization include trace elements that promote soil and water pollution and toxic gases that poison the atmosphere and contribute to the formation of acid rain (Stracher 2002, 2003; Stracher and Taylor 2004). Such pollutants, including As, F, Hg, Tl, Se, and organic compounds, have destroyed floral and faunal habitats, forced entire communities to relocate, killed people, and are responsible for a variety of human diseases including hyperkeratosis (As poisoning), dental and skeletal fluorosis (osteosclerosis), Tl poisoning, lung cancer, and pulmonary heart disease (Johnson et al. 1997, p. 19; World Resources Institute 1999, p. 63–67; Finkelman et al. 1999, 2001, 2002; Finkelman 2004). The identification and trace-element analyses of minerals formed as coal combustion by-products may reveal specific minerals that are vectors for toxins transmitted to humans by food grown in soils that contain these minerals or even by dust particles that are inhaled.

An additional motivation for the study of coal-fire mineral assemblages is to understand the long-term geologic record better and to establish mineralogical evidence for paleo-coal fires. Berner et al. (2000) have noted the need for negative feedback mechanisms for maintaining atmospheric O_2 at biologically permissible levels. Missing from their models is the potential for large-scale O_2 consumption from coal fires. Recognizing coal-fire mineral assemblages in modern systems such as in Wuda, Inner Mongolia is a first and requisite step to garnering evidence for large-scale coal fires throughout geologic time (Zhang et al. 2004b).

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