Trace Elements in Coal - Modes of Occurrence Analysis

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Abstract

The overall objective of this project is to provide modes of occurrence information for the CQ Inc. (CQ) effort being performed under DOE Contract No. DE-AC22-95PC95153, entitled "HAPs-R_x: Precombustion Removal of Hazardous Air Pollutant Precursors." This work attemps to provide semi-quantitative data on modes of occurrence of 15 elements. Coals investigated include as-mined coals and cleaned fines from the Northern Appalachian and Southern Appalachian, and Eastern Interior coal regions, and as-mined and natural fines from the Powder River Basin. Study techniques include scanning electron microscopy, electron microprobe analysis, and leaching procedures. Microprobe data indicate that pyrite grains in the Northern Appalachian, Eastern Interior and Powder River Basin coals and most of the pyrite grains in the Southern Appalachian coal contain low As concentrations, generally in the 100-500 ppm range. However, the Southern Appalachian coal contains some pyrite grains with much higher As contents, in excess of 4.0 wt. percent As. Microprobe analyses and data from leaching experiments indicate that arsenic is primarily associated with pyrite in the bituminous coals. These techniques also indicate that Cr is primarily associated with illite. Other HAP's elements have multiple associations.

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Introduction

The overall objective of this project, "Trace elements in Coal- Modes of Occurrence Analysis" project is to provide analytical support for the CQ Inc. (CQ) effort being performed under DE-AC22-95PC95153 and entitled "HAPs-R_x: Precombustion Removal of Hazardous Air Pollutant Precursors". Project goals include (1) developing fundamental trace-element mode of occurrence information and developing mechanisms for trace-element removal by coal cleaning to aid in realizing the full potential of HAPs control technologies, and (2) reducing this knowledge to engineering practice, and (3) assembling the information in a form that can be used by industry on a routine basis. In support of this effort, the United States Geological Survey (USGS) will analyze a number of coal samples utilizing the techniques described below, to provide information necessary to achieve a better understanding of toxic element behavior.

Phase I

As a complement to the analyses performed by CQ under DOE Contract No. DE-AC22-95PC95153, data from a unique protocol developed by the USGS is used to analyze selected coal size and density fractions for trace element forms of occurrence. In Phase I, the four Phase I coals were analyzed. The protocol incorporates the elements described below.

All of the samples have been treated by a selective leaching procedure, a powerful technique for approximating modes of occurrence using differing combinations of solvents at various temperatures and concentrations. Splits of the coal have been leached with these solvents (ammonium acetate, hydrochloric acid, hydrofluoric acid, nitric acid) according to the methods developed at the USGS. Results from these leaching tests provide essential information on chemical bonding of the elements present. Elements that are leached by hydrofluoric acid are generally associated with silicates, those that are leached by nitric acid generally occur in sulfides, and those that are leached by hydrochloric acid generally occur in carbonates and mono-sulfides. Ammonium acetate will leach elements that are weakly attached to exchangeable sites or are water soluble.

Experiments have also been conducted to determine volatility of the elements by heating the coal samples to temperatures ranging from less than 200^o C to more than 1,000^{o}C. A split of each coal sample was ashed using a low temperature ashing device. This procedure includes oxidation of the coal at temperatures of less than 200^o C, resulting in a residue of unaltered minerals. This low temperature ash residue will then be chemically analyzed to determine the volatility of the elements at low temperatures. This information, in conjunction with other tests, provides insight into chemical bonding of the elements present. The low temperature ash is also being used for semi-quantitative mineralogical determination by X-ray diffraction.

The above procedures provide indirect evidence, or approximations of the modes of occurrence of trace elements in coal. They will be complemented by direct procedures such as manual scanning electron microscopy (SEM), with energy dispersive analysis (EDX) of polished pellets of coal. The advantage of the conventional SEM methods over automated, computer-controlled SEM is that the operator can intelligently select appropriate phases for analysis by EDX and can apply instantaneous interpretation of the textural relations of the phases being analyzed. The mineralogical, geological, and geochemical expertise of the USGS personnel provide unique and essential insights.

For a more sensitive and quantitative analysis, an electron microprobe analyzer was used. Other, non-routine methods, such as analytical transmission electron microscopy, secondary ion mass spectrometry and infrared spectroscopy, were not used in Phase I, but may used in Phase II as necessary.

The Agency shall not proceed with any of the work under the Phase II program until formal notification is provided.

Phase II

In Phase II, a second group of representative coals will be analyzed. Detailed analysis of coal splits (size and density fractions) from both Phase I and Phase II coals will also be conducted, as required. The standard protocol to be used in Phase II is nearly identical to that used in Phase I; the only significant difference is in the samples to be analyzed. In Phase II, some samples may be subjected to separation procedures and subsequent analysis. For example, density or magnetic separations may be used, or handpicking of specific mineral grains. The protocol to be followed in Phase II incorporates the techniques described below.

Using a methodology similar to that of Phase I, all of the samples will undergo selective leaching, using the solvents ammonium acetate, hydrochloric acid, hydrofluoric acid, and nitric acid. Experiments to determine the volatility of specific elements will also be conducted by heating the coal samples to temperatures ranging from less than 200^{o} C to more than $1,000^{o}$ C, using the same procedures as described in Phase I.

The leaching and heating procedures provide indirect evidence, or approximations of the modes of occurrence of the trace elements in coal. As in Phase I, these procedures will be complemented by direct determinations on polished pellets of coal using conventional SEM analysis with the EDX analyzer . For a more sensitive and quantitative analysis, an electron microprobe analyzer will be used. In each procedure the mineralogical, geological, and geochemical expertise of the USGS personnel will provide unique and essential insights. Other, non-routine methods, such as analytical transmission electron microscopy, secondary ion mass spectrometry and infrared spectroscopy, will be used as necessary. Also in Phase II, some samples may be subjected to separation procedures and subsequent analysis. For example, density or magnetic separations may be used, or where required, hand picking of specific mineral grains. In Phase II we would like to propose two addition items. Because regulation of mercury is still being considered by EPA, understanding the modes of occurrence of mercury is critical. Unfortunately, due to laboratory contamination, volatility, and a host of other factors, the precision of mercury analysis is not well understood. Because this precision is critical to our evaluation of the modes of occurrence we propose to evaluate mercury precision and make improvements as necessary. Secondly, we would like to develop procedures to streamline our analyses for each element by trying to reduce the number of steps necessary to determine modes of occurrence. These streamlined procedures will become critical if laboratories need modes of occurrence information in order to determine cleanability of their coals. We will work with CQ to determine the minimum amount of information needed for their predictive methods for each element and develop cost effective procedures to provide that information.

Methods

The sequential selective leaching procedure used in this study is similar to one described by Palmer et al. (1993) which was modified from Finkelman et al. (1990). Duplicate 5g samples were sequentially leached with 35 ml each of 1N ammonium acetate (CH_3COONH_3), 3N hydrochloric acid (HCl), concentrated hydrofluoric acid (HF; 48%) and 2N (1:7) nitric acid (HNO_3) in 50 ml polypropylene tubes. Each tube was shaken for 18 hrs on a Burrell[1] wrist action shaker. Because of the formation of gas during some of the leaching procedures it is necessary to enclose each tube in double polyethylene bags, each closed with plastic coated wire straps. The bags allow gas to escape, but prevent the release of liquid. Approximately 0.5 g of residual solid was removed from each tube for instrumental neutron activation analysis (INAA) and cold vapor atomic absorption for mercury. The solutions were saved for inductively coupled argon plasma (ICP) analysis and inductively coupled argon plasma mass spectroscopy (ICP-MS) analysis.

SEM and Microprobe

1 - Coal pellet casting and polishing

The pellet formation procedure follows the ASTM D2797-85 technique for anthracite and bituminous coal as modified by Pontolillo and Stanton (1994). The casting procedure impregnates, under pressure, approximately 7-8 grams of crushed sample with Armstrong C4 epoxy. The resultant mold is cured overnight at 60E C. A label is incorporated with the sample.

The pellet block is ground and polished using ASTM D2797-85 standards as

[1]Use of trade names and trademarks in this publication is for descriptive purposes only and does not constitute endorsement by the U. S. Geological Survey.

modified by Pontolillo and Stanton (1994). The epoxy-coal pellet is ground with a 15 μm diamond platen and 600- grit SiC paper until flat and smooth. Rough polishing is done with 1 μm alumina and final polishing is completed with 0.06 μm colloidal silica. Ultrasonic cleaning between and after the various steps insures a final product free of extraneous abrasive material.

Two pellets were prepared from each sample. Each pellet was sectioned with a thin, slow-speed diamond saw and carbon coated for SEM and microprobe analysis.

2 - Scanning electron microscope analysis.

A JEOL-840 scanning electron microscope equipped with a Princeton Gamma-Tech. energy-dispersive X-ray analytical system and/or an ETEC Autoscan with Kevex EDX were used for SEM examination of project coals. Mineral identifications were based on morphology, and major-element composition of grains. Both secondary electron imaging (SEI) and backscattered electron imaging (BSE) modes were used in coal sample characterization. The BSE mode is especially sensitive to variation in mean atomic number, and is useful for showing within-grain compositional variation. By optimizing the BSE image, the presence of trace phases containing elements with high-atomic number can be revealed. Samples were scanned initially to obtain an overall view of the phases present, as with a petrographic microscope. This initial scanning was followed by a series of overlapping traverses in which the relative abundance of the phases were assessed. EDX analysis provides information on elements having concentrations roughly the tenth-of-percent level or greater. Typically operating conditions for SEM analysis were: accelerating potential of 10-30 kV, magnifications of ~50->10,000 times and working distances ranging from 15 to 20 mm (ETEC)and 25 or 39 mm (JEOL).

Scanning electron microscopy preceded electron microprobe analysis. SEI or BSE images taken at low magnification were used as a guide to locate phases of interest for microprobe analysis. SEM images taken at higher magnifications provided records of the points analyzed. Images at higher magnifications commonly reveal the presence of interstices or other imperfections in mineral grains that are not visible in reflected light microscopy. SEI/BSI mapping enabled us to avoid features in mineral grains that would adversely affect the quantitative analysis using the microprobe.

3 - Electron microprobe analysis.

A fully-automated, 5 spectrometer instrument (JEOL JXA 8900L Superprobe[1]) was used to quantitatively determine element concentrations in sulfides and clays by the wavelength-dispersive technique. In our preliminary microprobe work with the program coals, we measured the following elements: Fe, S, As, Ni, Cu, Zn, Se, and Cd. Natural and synthetic standards were used. Beam current used was 2 x 10^{-8} to 3.0 x10^{-8} amps; voltage was 20 KeV. The probe diameter was set as a focused beam; the actual working diameter was about 3-5 micrometers. In this study, we considered the minimum detection limit for the microprobe was about 100 ppm for each of the elements analyzed, using counting times of 60 seconds for peak and 30 seconds for background for most of the elements. For arsenic in pyrite, counting times of 90 seconds for peak and 45 seconds for background were used. Trace elements analyzed using the microprobe can be detected at the 100 ppm level; however, counting statistics at this concentration have a large uncertainty. In the probe analysis, we attempted to detect compositional differences among different pyrite morphologies. Microprobe data collected are shown in Appendix I. In addition to quantitative analyses, the JEOL 8900L was used to produce color maps of elemental distribution in project sulfides.

X-Ray Diffraction analysis

In order to obtain semi-quantitative information on the minerals present in the study coals, samples of low- temperature (<200^o C) ash were pressed into pellets and X-rayed using an automated diffractometer. The samples were diffracted over an interval from 4^o to 60^o 2θ. Counts were collected at 0.5 seconds per step. The data were processed using a computer program for semi-quantitative mineral analysis by X-ray diffraction (Hosterman and Dulong, 1985).

Results and Discussion

SEM and Microprobe Analysis

SEM analysis indicate the presence of the major minerals illite, kaolinite, quartz, calcite, and pyrite in each of the program coals and corresponding cleaned or natural fines (Appendix I). In addition to these five minerals, iron oxides were found in the as-mined coal and cleaned fines of the Southern Appalachian coal and the as-mined Powder River Basin coal, apatite was found in the as-mined Eastern Interior coal and barite was found in the as-mined and natural fines of the Powder River Basin coal. Other minerals were found in minor and trace amounts (Appendix I). The SEM was used to examine textural relationships as well. For example, differences in amount and size of minerals in the as-mined coal and cleaned fines of the Eastern Interior Basin are shown in Figure 1 (a and b). The SEM was also used to observe differing morphologies of pyrite in the program coals; these morphologies include subhedral grains, euhedral grains, composite grains and framboids.

1 - Microprobe Analysis of Fe-sulfides

Microprobe data for most pyrites indicate trace-element concentrations that are at or below the detection limit of ~100 ppm (Appendix II). Of the 7 trace elements determined (Se, Cu, Ni, As, Zn, Cd, and Co), only Cu, As, and Ni are commonly present at levels that exceed the detection limit. Concentrations of these 3 elements were determined for all of the pyrites analyzed.

As and Ni concentrations in pyrites from each of the as-mined and fine coal fractions are shown in Figure 2. The Eastern Interior coal shows the tightest data cluster

(Fig. 2a), with As values less than 0.1 weight percent, and Ni values less than about 0.15 weight percent. As and Ni values for pyrites in the Northern Appalachian coals are similar to those in the Eastern Interior samples, but show slightly greater scatter (Fig. 2b). Overall, pyrites from these two coals are not distinguishable based on As and Ni concentrations. The As contents do not appear to vary according to morphology of pyrite grains, but in each coal, pyrite framboids appear to be more likely to contain measurable levels of Ni. For pyrite in the Powder River Basin coal, As and Ni are both less than about 0.05 weight percent, with the exception of two framboids in the natural fines having As values of 0.12 and 0.27 weight percent, and Ni values of 0.05 and 0.29 weight percent, respectively (Fig. 2c)

Arsenic contents of pyrite in the Southern Appalachian coal and cleaned fines are generally higher than those of the other coals, and As distribution is far more heterogeneous, ranging from less than 0.01 weight percent to greater than 4 weight percent (Fig. 2d). Individual high arsenic (> 1 weight percent) pyrites were found to have multiple forms, including subhedral, irregular, and possibly cleat-filling pyrites (Appendix II). The As content of framboids in the Southern Appalachian coal is however, less than about 0.3 weight percent. The non-framboidal pyrite varieties generally exclude Ni, whereas the as-mined Southern Appalachian framboids trend towards Ni contents in excess of 0.4 weight percent (Fig. 2d). Microprobe elemental mapping reveals that formation of the high-As, low-Ni pyrite grains occurred after framboid formation (Fig. 3).

Arsenic and nickel contents of pyrite grains are plotted vs. their maximum dimensions in Figures 4 and 5, respectively. Although the figures are not meant to represent the complete grain-size distribution of the pyrites, the diagrams suggest that pyrites larger than 100 microns are present only in the as-mined samples, and in the natural (Powder River) fines. The largest pyrites analyzed in the three cleaned fines are less than about 70 microns, suggesting that the cleaning process is effective in removing the largest pyrites from the fines. This may have important consequences for As removal from the Southern Appalachian coal, as the highest-arsenic (>2 weight percent) pyrites have maximum dimensions ranging from 70 to 150 microns (Fig. 4d).

Using the average As and Ni concentrations obtained by electron microprobe, mass-balances were calculated for each of the as-mined coals. The contribution of pyrite to the mass-balance of a given trace element was calculated by multiplying its concentration in pyrite by the amount of pyrite (in weight percent), calculated from pyritic sulfur values. The amount obtained is expressed as a percentage of the whole-coal value for that element. Results for As are shown in Figure 6, in which mass-balance fractions obtained by microprobe are compared with those obtained by leaching experiments (see below). The best agreement is for the Eastern Interior as-mined coal, in which electron microprobe data accounts for 75% of the arsenic in the coal, compared to 60% as indicated by selective leaching (HNO_3 fraction; discussed later). For the Powder River as-mined coal, 50% (microprobe) to 25% (leaching) of As resides in pyrite (Fig. 6). This agreement is within the uncertainty of the method because the leaching value requires an average As in pyrite of about 50 ppm, about half the microprobe detection limit. The microprobe result includes many below-detection-limit values that were each taken as 70% of the detection limit (70 ppm) in the calculation which is likely to over-account for the arsenic in the pyrite. The poorest agreement for As is for the

Northern Appalachian as-mined coal, in which a 70% fraction in pyrite is indicated by leaching, but only 33% by microprobe (Fig. 6). This may be due to the presence of rare higher-As pyrite grains that were not detected and analyzed in the microprobe studies. Because of the As-heterogeneity in the Southern Appalachian pyrites, no attempt was made to calculate an average pyrite As concentration from the microprobe analyses. No mass-balance calculations were done for pyrites in the fines because pyritic sulfur determinations are not available.

Mass-balance calculations for Ni in pyrite are shown in Figure 7 for each of the as-mined coals. Agreement between microprobe and leaching results is generally good, with all results in the 10-30% range.

2 - Microprobe Analysis of Clay Minerals

Microprobe analyses of the clay minerals, illite and kaolinite, are given in Appendix 2. The average Cr_2O_3 concentration of illites in Appendix 2 (270 ppm) is marginally above the detection limit (about 200 ppm), and is therefore subject to a large uncertainty (about 150-200 ppm). These analyses reveal however, that some illites contain Cr at measurable levels of about twice the detection limit. Mass balances for Cr in illite were calculated using the average Cr_2O_3 value (equivalent to about 180 ppm Cr), and estimates of the illite content of project coals based on XRD results for low temperature ash described in the next section. Comparison of the fraction of Cr in illite, as calculated from electron microprobe data, vs. results for Cr in the silicate fraction, from leaching studies (HF leachable), is given in Figure 8. The microprobe (60 %) and leaching data (75%) are similar for the Southern Appalachian as-mined sample, but for the Northern Appalachian and Eastern Interior as-mined samples, the microprobe (25-30%) appears to underestimate the fraction of Cr in illite indicated by leaching studies (75-80%). This tendency is even more pronounced in mass-balance calculations for the fines (Fig. 9). While the microprobe data confirm the presence of Cr in illite, the mass-balance calculations appear to be less reliable than those for trace elements in pyrite, for reasons that may include: 1) a much smaller population of grains was used to calculate average compositions; 2) the proportion of illite in each coal, as determined by XRD, is only semi-quantitative; 3) detection limits for trace elements in illite are higher, about 200 ppm; and 4) chromium may be present in silicates other than illite; 5) some Cr may exist as Cr oxyhydroxides (Huggins and Huffman, 1996) which we did not detect nor analyze. The illites also show large major-element variations, likely due to the presence of mixed-layer clays and finely disseminated quartz, making it difficult to analyze a truly representative illite. No Powder River illites were analyzed, so this coal was excluded from the Cr mass-balance calculations.

Unlike illites, kaolinites show little chemical variation, all were essentially stoichiometric $Al_2Si_2O_5(OH_4)$, with minor substitution by Fe, and K, possibly from adjacent illites (Appendix 2). Some kaolinites give a response for Cr_2O_3, but all such cases are near or at the detection limit, and the presence of Cr in kaolinite is questionable. Kaolinite and illite analyses in Appendix 2 have sums that are less than 100 percent because of structural water in these clays.

Examination of Maceral Separates

Vitrinite and inertinite separates from the Northern Appalachian coal and the Eastern Interior coals were prepared by J. Crelling (Southern Illinois University) to help us assess the contribution of coal macerals to the trace element mass balance. Ash yields of the vitrinites were 2.1 weight percent (Eastern Interior) and 5.7 weight percent (Northern Appalachian). For the inertinites, ash yields were 39.8 weight percent (Northern Appalachian) and 15.1 weight percent (Eastern Interior). These high ash yields indicate that the separates were not sufficiently pure to be representative of the macerals. For example, Lyons et al. (1989) found that ash contents of hand-picked vitrinite were less than 1 weight percent. The Northern Appalachian and Eastern Interior vitrinites and inertinites were examined under SEM and significant mineral matter was found. In addition, stainless steel contamination (Fe, Cr, Ni) was found in both Northern Appalachian separates. As a follow-up, an improved 2 gram separate of the Northern Appalachian vitrinite containing about 2 weight percent ash was obtained. Elemental maps of Se, Ni, As, and Cr all show that these elements are below the limit of detection of the electron microprobe (100 ppm).

There was not sufficient sample to determine mode of occurrence on the improved 2 gram Northern Appalachian vitrinite, but we did leaching experiments on the original separates. For several elements it is likely that we are determining the modes of occurrence of contaminates rather than elements truly associated with the vitrinite or inertinites. For example, a density separation using CsCl was part of the separation procedure for these macerals. This resulted in one weight percent Cs in the Eastern Interior vitrinite separate and 3 weight percent Cs in the Eastern Interior inertinite separate. The Northern Appalachian vitrinite separate contained 0.27 weight percent Cs and the inertinite separate from that coal contained 0.86 weight percent Cs. This compares to 4.33 ppm Cs in the as-mined Eastern Interior coal and 2.92 ppm in the Northern Appalachian as-mined coal. Eighty to ninety-four percent of the Cs is leached by the ammonium acetate, up to 17 percent is leached by HCl indicating that this contaminant is not being completely removed by ammonium acetate. Most of the other elements are in lower concentrations in the maceral separates than in the original coal. The exceptions are Cr, U, and As in the inertinite separates. The high Cr concentrations are probably due to clay embedded in these separates. Twenty-six to forty-nine percent of the Cr and 25 to 56 percent of the U was leached by HF indicating significant quantities of silicates in the vitrinites and inertinites. Mercury and arsenic were higher in both the inertinite and vitrinite separates but leaching results (the high proportion of these elements in the ammonium acetate leaches) suggest that this might be due to contamination. Despite the possibility of contamination we feel that this procedure has potential to determine the amounts of elements that are organically bound, and we recommend another attempt to gain this insight from the vitrinite concentrates.

Heating Experiments

All samples were heated to 200° C, 550° C and 1000° C. Preliminary results indicate that there is little or no volatilization of any elements for the Southern or Northern Appalachian coals or cleaned fines or for As, Be, Mn, Th, U, Ni or Co in the other program coals. Significant reductions in the concentrations of Sb, Pb, Cd, and Zn in the 1000° C split indicates volatilization of these elements in the Eastern Interior coal and cleaned fines for Pb and Cd only. Pb and Zn were also partially volatilized from the Powder River Basin coal; Pb is volatilized in the Northern Appalachian as-mined and cleaned coals. The reason for this is still under investigation. Because mercury is such a volatile element all samples were heated to 102° C and the concentration of mercury was determined. Table 1 shows the results. The Eastern Interior as-mined coal was the only coal to show a significant loss in mercury. It should be noted that the concentrations of Hg determined for the unheated coal samples in this experiment coals were generally lower that those determined for similar samples in our leaching study. The reasons for this are still being investigated.

Semi-Quantitative Mineralogy of Low-Temperature Ash

Table 2 gives semi-quantitative estimates of mineralogy based on X-ray diffraction analysis (XRD) of low-temperature ash (LTA) of all as-mined and cleaned or natural fines. About 15 to 35 percent of the ash of each of the LTA consists of quartz, 15 to 55 percent of the LTA is kaolinite and 5 to 35 percent of the LTA is illite. The Eastern Interior LTA contains 5 to 10 percent pyrite. Other coals all contained less than 5 percent pyrite. LTA for the Powder River Basin coal and natural fines containes about 20 percent calcite and about 5 to 10 percent calcium sulfate (gypsum; bassanite- a dehydrated mineral usually assumed to be formed from gypsum during the low temperature ashing process). Calcium sulfate was also observed by SEM. Some gypsum/bassanite may form in the ashing process from organic sulfur and calcite. The LTA of Southern Appalachian as-mined and cleaned fines containes 20 to 40 percent chlorite. Chlorite was not identified by SEM (Appendix 1). It is possible that some of this chlorite is produced during the ashing process from illite and mixed layered clays. Feldspar, siderite, sphalerite and apatite were found in minor and trace quantities in several coals. Although feldspar was not observed by SEM, they could likely be mistaken for clays when analyzed by SEM.

Elemental Analysis— Quality Control

All of the samples were analyzed by several techniques. Table 3 shows the data for the unleached as-mined coals and cleaned and natural fines. Elemental composition of each of the as-mined coals and cleaned or natural fines were determined in duplicate by instrumental neutron activation analysis (INAA) except for the Eastern Interior cleaned fines which was determined in triplicate, and the as-mined Eastern Interior coal which was analyzed only once . The as-mined Eastern Interior sample was also analyzed in duplicate by inductively coupled atomic emission analysis (ICP-AES), and inductively coupled plasma mass spectrometry (ICP-MS), and in triplicate by cold vapor atomic absorption (CVAA) for Hg, and by hydride generation atomic absorption (HGAA) for Se. All other program coals were analyzed by these techniques only once. Comparison of results from different independent procedures can provide information on reliability of data.

With each group of about 30 samples, the USGS standard coal CLB-1 was analyzed by INAA as a control sample. Table 4a shows eight different analyses of CLB-1 by INAA. The mean and standard deviation of the values, and the mean of the reported errors are also given with the accepted values. There was excellent agreement between the measured values and the "accepted" values. Four control standards were used for ICP-AES. Determined values and accepted values are given in Table 4b. Several control samples were used for ICP-MS. These control samples, along with accepted values, are given in Table 4c. In addition, several control samples were determined with cold-vapor atomic absorption (Hg) and with hydride generation atomic absorption (Se). These control samples are included in Table 4d. Duplicate analysis of leached samples by INAA and ICP-AES are also given in Table 5a and 5b.

Leaching Experiments

Leaching experiments were completed for the four program coals and the resulting leachate solutions and solid residues were submitted for chemical analysis. ICP-AES and ICP-MS analyses (for leachate solutions) and INAA analyses (for solid residues) have been obtained. Chemical data for the leachates have been processed to derive the mean percentages of each element leached by each of the four leaching agents. The calculated percentages were then used as an indirect estimate of the mode of occurrence of specific trace elements in the coals. We estimate an error of up to $\pm$25 percent for these data.

The data for the as-mined coals (referred to as "Raw" in figures to follow) were determined in quadruplet leaching experiments. The cleaned fines (-28 mesh) of the Southern Appalachian, Eastern Interior and Northern Appalachian coals, and the natural fines of the Powder River Basin coal, were determined in duplicate. The clean and natural fines are all referred to as "Fines" in the figures that follow. Figure 10 shows a complete data set for arsenic. Reproducibility is generally within the expected analytical errors and is generally better within a technique than between techniques. The instrumental neutron activation analysis (NA in figures) values were determined on the solid residues whereas the ICP-MS values (ICP in figures) were determined on the solutions. Values over 100 percent in some of the ICP-MS fractions may indicate contamination of the solutions, or analytical error. We are currently examining each of these possibilities. The average of percent leached values for each technique is shown in Figure 11. The total stacked bar height of the NA is determined by subtracting the concentration of a given element in the HNO_3 solid residue (in this case As) from 100 percent leached. The difference of the total stacked bar height and 100 percent leached represents a fifth fraction, an unleached fraction, of each element. By considering the potential uncertainty of each technique, and constraining each leach to five fractions and no more than 100% leached a single value for the percent leached for each element in each of the fractions of each sample was derived (Figure 12). This procedure was followed for all of the data presented, except for elements that were determined only on either the solid or the liquid.

1 - Arsenic

The bulk of the arsenic in the bituminous coals is in pyrite. Forty-five to seventy-five percent of the As in these coals is leached by HNO_3. It is possible that micro-fine pyrite grains are encapsulated (shielded) within the coal. When this material is considered, sixty to eighty percent of the arsenic is associated with the pyrite in the bituminous coals (See Table 6). In contrast, only 25 to 30 percent of the arsenic in the subbituminous Powder River Basin coal and natural fines are associated with pyrite. The remaining arsenic is distributed among arsenates and silicates (probably clays). This does not rule out small amounts (5 to 10%) which may be organically associated. No pyrite was found in HNO_3 leached residues of Powder River Basinand the Southern Appalachian coals, and only very minor amounts of pyrite remained in the residue of in the other two program coals.

Arsenic in all samples were partially soluble (5 to 40%) in HCl. HCl soluble arsenic may be due arsenates in the coal or due to the formation of arsenates by oxidation of pyrite. It also possible that there are HCl-soluble arsenic-bearing sulfides present. In the Powder River Basin coals and natural fines, 40% of the arsenic is HCl soluble and 25% is HF soluble (silicates). Microprobe data confirms the presence of arsenic in pyrite but also shows its distribution to be very heterogeneous. Mass balance calculations based on microprobe data suggest that roughly 30 to 70 percent of arsenic is in pyrite, similar to results obtained by the leaching determinations.

2- Mercury

The behavior of mercury is somewhat similar than that of arsenic. However, the low concentrations and the volatility of mercury make it much more difficult to measure. Mercury was determined by cold vapor atomic absorption on a split of the solid residue, and in solution by ICP-MS. Mercury has two dominant modes of occurrence. Figure 13 shows that 25 to 65 percent of the Hg is leached by HNO_3 indicating 25 to 65 % of the Hg is in pyrite (Table 6). In addition, 5 to 35 percent is leached by HCl. The HCl-leachable Hg may be associated with oxidized pyrite or HCl soluble sulfides. The residual Hg (<35 percent) may be due to shielded pyrite and/or organic association. It is likely that some of the Hg is in shielded pyrite, however, there are some indications that Hg behaves slightly differently than other elements associated with pyrite and some Hg may be absorbed to the surfaces of organic rich particles (Table 6). Further work is needed to resolve this issue.

3 - Chromium

Unlike As and Hg, Cr is mostly associated with silicates. Figure 14 shows that 35 to 65 percent of the Cr is leached by HF. Microprobe analysis of illite grains in the study coals indicates Cr concentrations of 0 to 0.05 weight percent. Mass balance calculations suggest that average concentrations in illite account for up to 60 percent of the total Cr. Minor amounts of Cr were also leached by HCl and HNO_3. HNO_3 leached Cr is probably associated with sulfides. Ten to forty-five percent of the Cr remained in the HNO_3-leached residue. This is most likely due to insoluble spinels such as chromite. However we cannot rule out organically associated Cr or shielded illite grains. The 5 to 25 percent HCl soluble Cr may be due to oxyhydroxides as reported by Huggins and Huffman (1996).

4 - Selenium

As much as 75 percent may be organically associated (Table 6). Figure 15 shows 20 to 75 percent of the Se remains in the HNO_3-leached residue. Work is continuing to verify the organic Se association. Some of the Se is also associated with the sulfides. Although 25 to 55 percent of the Se is leached by HNO_3, oxidation of organics by HNO_3 may release some organically-bound Se, which would then be indistinguishable from Se released from the sulfides. Microprobe data of individual pyrite grains indicates an average Se concentration at or below the detection limit of 0.01 weight percent. Some Se was leached by ammonium acetate, possibly indicating the presence of exchangeable-or water-soluble Se compounds, especially in the Powder River Basin coal. Lead selenide (PbSe) was detected by microprobe in the Southern Appalachian coal, which may account for some of the HCl-leachable Se in that sample.

5 - Nickel

Finkelman (1994) reports a large uncertainty in the mode of occurrence of Ni in coal. Although the results for each of the coals are generally consistent (Figure 16), there seems to be no single mode of occurrence for Ni. Table 6 suggests four modes of occurrence for Ni. Significant amounts of Ni are leached by HCl, HF, and HNO_3. Ni is commonly present at the 0.02 to 0.03 weight percent level in pyrite, but has not yet been detected in other forms. Because up to about 37 percent of the Ni remains unleached by HNO_3, some of the Ni may be organically associated, or in small particles shielded by organics. The relatively high analytical errors of Ni values determined by INAA at or near their detection limits, especially in the solid residues, contributes to the overall uncertainty.

6 - Cobalt

The behavior of cobalt is similar to that of Ni (Figure 17; Table 6) and Co may have similar modes of occurrence. HCl was the most effective solvent, but like Ni, substantial amounts of Co were leached by HF and HNO_3 . Co has also been detected in some pyrite grains. Because up to 38 percent of the Co remains unleached by HNO_3, some of the Co may be organically associated, or in small particles shielded by organics. Although the concentration of Co is lower than that of Ni, INAA is much more sensitive for Co than that for Ni, making the analytical uncertainties significantly lower for Co than for Ni.

7 - Antimony

The behavior of antimony (Sb), is also similar to Co and Ni (Figure 18; Table 6). In addition to a sulfide association, some Sb is associated with the silicates, as evidenced by significant amounts of Sb leached by HF, especially in the Powder River Basin coal; some Sb probably exists as oxides as well (Table 2; HCl leachable) More than 35 percent of the Sb remains in the HNO_3 leached solid residue suggesting that some of the Sb may be organically associated or in small mineral grains shielded by organics.

8 - Lead

Lead is leached by HCl and HNO_3 (Figure 19), consistent with galena as the major source of Pb in these coals. Although galena was not detected by SEM in any of these coals (Appendix I), galena is a common accessory mineral in coals (Finkelman, 1994). The wide range of unleached Pb may be due to shielding of submicroscopic galena grains. Pb was not determined in the residues because Pb cannot be determined using the INAA procedures employed in this study. Lead selenide (PbSe) was detected by microprobe in the Southern Appalachian which may account for HCl leachable Pb in that sample.

9 - Cadmium

Data for Cd are very limited. Cd concentrations are below the detection limit in all of the solid residues. Cd concentrations were also below the detection limit in the cleaned fines of the Southern Appalachian coal and all the as-mined coals, except the Eastern Interior sample, and in all of the pyrites analyzed by microprobe. Cd is primarily soluble in HCl (Figure 20). These data are consistent with a sphalerite (ZnS) association. Zn (Figure 21) which is in much higher concentrations, has similar leaching patterns as Cd, and the Zn/Cd ratio is relatively constant in most coal samples.

10 - Beryllium

Beryllium is generally associated with the silicates. Be is not easily detectable by microprobe or SEM due to its low atomic number. It is also not detected in the residues (by INAA). Data plotted in Figure 22 shows that 37 to 100 percent is leached by HF and 71 to 100+ percent is leached by all solvents. In addition to HF, some Be is HCl leachable (0-70 percent).

11 - Manganese

Manganese is primarily HCl-leachable, and is mostly associated with carbonates (Figure 23). Small amounts of Mn are leachable by all other solvents suggesting several minor associations. Mn was not determined in the residues by INAA due to its relatively short half-life (2.5 hours). Mass balances are between 80 and 130 percent suggesting that there is little or no organic association. Concentrations of Mn are relatively high, which should provide precise analysis.

12 - Radioactive Elements (U and Th)

Uranium (Figure 24) and thorium (Figure 25) have slightly differing modes of occurrence. Uranium is mostly leached by HF and HCl, whereas HNO_3 is the most effective solvent for Th. The HNO_3-leached thorium may be attributable to phosphates (e.g. monazite). Less than 55 percent of the U in the Northern Appalachian samples and less than 50 percent of the Th in the as-mined Powder River Basin sample were leached, suggesting a possible organic associations for these elements in those samples. In general, less U was leached than Th, indicating a higher organic association for U than Th. Both elements are present in insoluble phases such as zircon (Appendix 1).

13 - Iron

Iron is a major element in pyrite which contains many of the HAP's elements. We have therefore included iron in many of our tables. Figure 26 shows the modes of occurrence of iron in the various coal samples. Although pyrite is the major mode of occurrence for Fe in the Eastern Interior, Northern Appalachian, and Powder River Basin coals and cleaned and natural fines, the Southern Appalachian coal has significant amounts of HCl and HF leachable material indicating that iron in this coal is also in the silicates and oxides and/or carbonates.

SEM Examination of Leached Residues

To assess the effectiveness of the leaching process, each of the solid residues remaining after leaching were examined by SEM. All of the residues contain only traces of mineral matter. In each of the residues, a TiO_2 phase, probably rutile, is the most common mineral. TiO_2 was originally present as a minor or trace phase in each of the samples, and is relatively insoluble. Isolated grains of other insoluble minerals, such as zircon, are also present. SEM study confirms that nitric acid leaching effectively removes pyrite. For example, in the Eastern Interior samples (originally the most pyrite-rich), pyrite was not observed in the as-mined residue, and only several very isolated pyrites were found in the fines. The largest of the remaining pyrites (~5 microns) appear to be encapsulated by organic material. In several samples, isolated silicates (quartz, illite), up to about 10 microns in length, survived the leaching process due to similar encapsulation.

Semi-quantitative Modes of Occurrence

Combining all of the information from the leaching experiments, the microprobe and scanning electron microscopy, X-ray diffraction analysis and the various chemical analyses, as well as geochemical characteristics of each element, semi-quantitative assessments of each trace-element's modes of occurrence have been determined. Table 6 includes determinations of the percent of each element present partitioned among three or four major phases or minerals, for 15 elements. In cases where there is significant direct evidence, the exact form of the mineral is given, such as arsenic in pyrite or Cr in illite. In cases where there was strong geochemical evidence and strong indirect evidence, classes of minerals are given, such as sulfides, silicates, oxides or arsenates. In some cases a descriptor is used, such as HCl-soluble sulfides to distinguish these species from other sulfides, such as pyrite. All organic-element species were grouped into headings called organics.

Conclusion

Work on Phase I is complete. The USGS has analyzed the four program coals (Southern Appalachian, Eastern Interior, Northern Appalachian and the Powder River Basin) and cleaned fines from the first three and natural fines from the latter. Modes of occurrence of trace elements in coal were determined by: (1) trace element analysis (ICP-AES, ICP-MS, cold vapor atomic absorption, hydride generation), (2) leaching experiments, (3) SEM analysis, and (4) microprobe analysis. We have provided semi-quantitative results for modes of occurrence of fifteen elements. We have provided chemical analysis of whole coals by several techniques. We have provided chemical analysis by microprobe of individual pyrite and illite grains. Finally, mineralogical information on each of the as-mined coals and cleaned or natural fines has been provided.

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Table 1. Hg retention when samples are heated to 102^{o} C

	Hg concentration (ppm)		Ratio
	unheated	heated	heated/unheated
Whole coal			
Southern App 1	0.06	0.06	1.0
Southern App 2	0.06	0.07	1.2
Southern App Avg	0.06	0.06	1.1
Whole coal			
Eastern Int. 1	0.15	0.06	0.4
Eastern Int. 2	0.09	0.05	0.6
Eastern Int. Avg	0.12	0.06	0.5
Whole coal			
Northern App 1	0.06	0.06	1.0
Northern App 2	0.08	0.07	0.9
Northern App Avg	0.07	0.06	0.9
Whole coal			
Powder River B 1	<0.02	0.02	---
Powder River B 2	0.02	0.02	1.0
Powder River B Avg	0.02	0.02	1.0

Table 2. Semi-Quantitative Ash Mineralogy*

Sample	% LTA**	Quartz	Feldspar	Calcite	Siderite	Illite	Kaolinite	Chlorite	Pyrite	Gypsum + Bassanite	Sphalerite	Apatite
N. Appalachian As-Mined	25.9	25	trace	trace		20	50		trace	trace		trace
N. Appalachian Fines	12.0	20	trace			20	55		minor		trace	trace
S. Appalachian As-Mined	60.4	35	trace	trace		35	10	20	trace	trace		
S. Appalachian Fines	11.0	15	trace	trace	trace	20	20	40	trace	trace		trace
Eastern Interior As-Mined	38.0	30	trace	minor	trace	35	15	trace	10	trace		trace
Eastern Interior Fines	27.4	25	minor	trace		30	25		5	trace	trace	
Powder River As-Mined	10.5	15		20	trace	5	40		trace	10	trace	
Powder River Fines	22.0	20		20		10	40		trace	5	trace	5

*analyst F. Dulong, USGS-Reston
**percent low-temperature ash

Table 3. Analytical Values for Whole Coals

Element units:	As ug/g	Hg ug/g	Cr ug/g	Se ug/g	Ni ug/g	Co ug/g	Sb ug/g	Pb ug/g	Cd ug/g	Zn ug/g	Be ug/g	Mn ug/g	Th ug/g	U ug/g	Fe %
Southern Appalachian															
As-mined INAA1	17.1	ND	61	< 0.5	34	10.6	1.47	ND	ND	54	ND	ND	10.0	2.8	2.51
As-mined INAA2	19.9	ND	58	< 0.9	33	12.1	1.56	ND	ND	60	ND	ND	9.9	2.9	2.46
As-mined Denver Techniques	23	0.10	63	2.4	29	13	2	24	< 0.8	63	2	230	7.5	2.9	2.6
Cleaned Fines INAA1	14.8	ND	32	1.4	29	9.8	1.60	ND	ND	30	ND	ND	3.3	1.6	0.61
Cleaned Fines INAA2	14.0	ND	31.7	1.3	29	8.7	1.61	ND	ND	36	ND	ND	3.11	1.5	0.60
Cleaned Fines Denver Techniques	20	0.06	39	1.9	21	11	2.0	11	< 0.8	36	1.1	35	3.5	1.7	0.62
Northern Appalachian															
As-mined INAA1	7.6	ND	30	1.7	23	5.4	0.73	ND	ND	20	ND	ND	4.78	1.5	0.74
As-mined INAA2	7.5	ND	30.5	1.8	18	5.9	0.71	ND	ND	22	ND	ND	4.6	1.4	0.75
As-mined Denver Techniques	7.1	0.09	31	2.9	21	6.7	0.7	15	0.2	29	1	29	3.8	1.4	0.77
Cleaned Fines INAA1	6.0	ND	25.0	1.2	21	4.6	0.68	ND	ND	23	ND	ND	2.63	0.79	0.60
Cleaned Fines INAA2	6.4	ND	27.2	1.6	17	4.8	0.77	ND	ND	25	ND	ND	2.66	0.9	0.62
Cleaned Fines Denver Techniques	8.2	0.06	29	1.9	15	4.7	0.90	8.9	0.1	25	1	17	2.7	0.86	0.67
Eastern Interior															
As-mined INAA1	6.9	ND	95	9.2	51	6.3	1.1	ND	ND	185	ND	ND	4.4	5.7	2.7
As-mined Denver Techniques 1	7.3	0.09	91	13.6	40	6.2	1	20	2.6	190	1	91	4.7	6.2	ND
As-mined Denver Techniques 2	7.0	0.09	110	12.1	49	6.6	1	17	2.5	190	1	98	4.5	5.9	2.2
As-mined Denver Techniques 3	ND	0.09	ND	13.6	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Cleaned Fines INAA1	4.2	ND	84	2.6	31	6.1	0.64	ND	ND	139	ND	ND	4.1	2.1	1.62
Cleaned Fines INAA2	4.2	ND	83	2.5	36	6.1	0.65	ND	ND	142	ND	ND	4.1	2.2	1.61
Cleaned Fines INAA3	4.3	ND	84	2.9	31	6.2	0.67	ND	ND	147	ND	ND	4.1	2.1	1.62
Cleaned Fines Denver Techniques	5.1	0.06	91	4.7	30	6.3	0.8	14	0.5	150	1	86	3.0	2.2	1.7
Powder River Basin															
As-mined INAA1	1.5	ND	4.4	0.43	< 7	1.77	0.52	ND	ND	5.4	ND	ND	2.13	1.1	0.47
As-mined INAA2	1.3	ND	4.9	0.63	< 10	1.36	0.54	ND	ND	5.5	ND	ND	2.23	1.1	0.49
As-mined Denver Techniques 1	0.8	0.07	5.9	0.70	5.7	2.0	0.3	3.0	< 1.1	9.0	0.5	150	2.4	1.4	0.50
As-mined Denver Techniques 2	ND	0.07	ND	0.65	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Natural Fines INAA1	1.62	ND	24.1	0.6	< 9	1.58	0.54	ND	ND	16	ND	ND	2.10	0.98	0.78
Natural Fines INAA2	1.72	ND	23.5	0.5	< 9	1.63	0.60	ND	ND	20	ND	ND	2.11	1.08	0.78
Natural Fines Denver Techniques	1.9	0.06	31	0.69	8.1	2.1	0.77	< 8	0.2	21	0.4	130	2.3	0.98	0.75

Denver Techniques: ICP:Be, Co, Cr, Mn, Ni, Th, Zn, Fe.
ICP-MS: As,Cd, Pb, Sb, U.
Hydride Generation Atomic Absorption: Se
Cold Vapor Atomic Absorption: Hg
ND = not determined

Table 4. Control Samples

A. INAA

Element	As	Hg	Cr	Se	Ni	Co	Sb	Pb	Cd	Zn	Be	Mn	Th	U	Fe
units:	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	%
CLB Split 1	15.8	ND	10.0	1.8	19	6.39	1.6	ND	ND	50	ND	ND	1.43	0.53	0.91
Error:	0.47	ND	0.74	0.11	3.3	0.091	0.11	ND	ND	2.1	ND	ND	0.065	0.088	0.048
CLB Split 2	12.9	ND	10.3	2.4	18	7.1	1.5	ND	ND	47	ND	ND	1.4	0.6	0.88
Error:	0.46	ND	0.78	0.17	5.0	0.14	0.12	ND	ND	1.9	ND	ND	0.12	0.12	0.014
CLB Split 3	20.4	ND	10.3	2.7	30	6.2	1.66	ND	ND	57	ND	ND	1.44	0.46	0.88
Error:	0.61	ND	0.69	0.20	4.5	0.13	0.084	ND	ND	2.5	ND	ND	0.042	0.070	0.017
CLB Split 4	13.0	ND	11.0	2.8	21	7	1.5	ND	ND	54	ND	ND	1.47	0.54	0.94
Error:	0.28	ND	0.66	0.15	4.5	1.3	0.10	ND	ND	1.7	ND	ND	0.063	0.043	0.013
CLB Split 5	14.5	ND	10.2	1.8	20	7.1	1.59	ND	ND	55	ND	ND	1.42	0.62	0.91
Error:	0.44	ND	0.50	0.15	3.0	0.14	0.097	ND	ND	2.0	ND	ND	0.050	0.091	0.015
CLB Split 6	15.7	ND	10.1	1.7	21	6.7	1.7	ND	ND	51	ND	ND	1.37	0.49	0.91
Error:	0.48	ND	0.39	0.18	2.7	0.20	0.12	ND	ND	2.0	ND	ND	0.076	0.060	0.017
CLB Split 7	14.1	ND	10.1	1.9	17	7.0	1.51	ND	ND	48	ND	ND	1.38	0.49	0.90
Error:	0.42	ND	0.60	0.16	3.7	0.19	0.048	ND	ND	2.2	ND	ND	0.077	0.064	0.021
CLB Split 8	14.4	ND	9.6	1.9	19	7.0	1.52	ND	ND	50	ND	ND	1.37	0.52	0.89
Error:	0.44	ND	0.38	0.17	2.9	0.16	0.073	ND	ND	5.1	ND	ND	0.077	0.073	0.020
Mean:	15.1	ND	10.2	2.1	20	6.8	1.6	ND	ND	51	ND	ND	1.41	0.53	0.90
Standard Deviation:	2.4	ND	0.40	0.45	4.1	0.35	0.085	ND	ND	3.5	ND	ND	0.037	0.054	0.022
Mean Error:	0.45	ND	0.59	0.16	3.7	0.29	0.095	ND	ND	2.4	ND	ND	0.071	0.076	0.021
Acccepted Value:	13		9.7	2	18	7	1.5			48			1.4	0.55	1.1
Error:	IV		1.2	IV	2	0.7	IV			4			IV	IV	0.44

ND = not determined
IV = Information values

Table 4. Control Samples

B. ICP-AES

Element:	As	Hg	Cr	Se	Ni	Co	Sb	Pb	Cd	Zn	Be	Mn	U	Th	Fe
units:	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	%
Ref 65-1	ND	ND	0.02	ND	0.02	ND	ND	ND	ND	0.66	0.1	0.31	ND	ND	ND
Ref 65-2	ND	ND	0.03	ND	0.02	ND	ND	ND	ND	0.86	0.12	0.41	ND	ND	ND
Ref 65-3	ND	ND	0.02	ND	0.01	ND	ND	ND	ND	0.62	0.09	0.33	ND	ND	ND
Ref 65-4	ND	ND	0.02	ND	0.03	ND	ND	ND	ND	0.59	0.09	0.3	ND	ND	ND
Ref 65-5	ND	ND	0.02	ND	0.03	ND	ND	ND	ND	0.59	0.09	0.33	ND	ND	ND
Ref 65-6	ND	ND	0.03	ND	0.01	ND	ND	ND	ND	0.78	0.11	0.37	ND	ND	ND
Ref 65-7	ND	ND	0.02	ND	0.02	ND	ND	ND	ND	0.77	0.11	0.32	ND	ND	ND
Ref 65-8	ND	ND	0.02	ND	0.02	ND	ND	ND	ND	0.62	0.0056	0.32	ND	ND	ND
Mean			0.02		0.0202					0.69	0.09	0.34			
Standard Deviation			0.005		0.008					0.10	0.036	0.036			
Accepted Value (1)			0.019		0.017					0.489	0.185	0.279			
NIST 1632b (2)	ND	ND	11	ND	6.1	2.2	ND	ND	ND	11.7	0.7	12.2	ND	1.5	1.04
NIST 1632b (2)	ND	ND	13	ND	7.4	2.6	ND	ND	ND	ND	0.7	15.8	ND	2.7	0.884
Mean	ND	ND	12	ND	6.7	2.4	ND	ND	ND	ND	0.7	14.0	ND	2.1	0.962
Accepted Value (3)			11		6.1	2.29				11.89	NR	12.4		1.342	0.759
PPG-1	ND	ND	10.8	ND	3.44	5.62	ND	ND	ND	115	4.41	726	ND	30.1	3
PPG-2	ND	ND	9.68	ND	3.05	5.16	ND	ND	ND	104	3.83	653	ND	30.4	2.71
Mean			10.2		3.24	5.39				110	4.12	690		30.2	2.9
Accepted Value (4)			10		4	6				110	4	670		31	2.7
CLB-1 (5)	ND	ND	13	ND	17	5	ND	ND	ND	53	ND	12	ND	3.5	1.01
Accepted Value (6)			9.7		18	7				48		8		1.4	1.25

(1) U.S. Geological Survey Water Standard Certificate of Anaysis
(2) Values based on dissolution of NIST 1632b coal ash reported on a whole coal basis
(3) Values taken from National Bureau of Standards Certificate of Analysis for 1632b
(4) U.S. Geological Survey Internal Standard Certificate of Anaysis
(5) Values based on acid digest of CLB-1 ash recalulated to a whole coal basis
(6) U.S. Geological Survey Certificate of Anaysis
ND = Not determined
NR = Not reported

Table 4. Control Samples

C. ICP-MS

Element:	As	Hg	Cr	Se	Ni	Co	Sb	Pb	Cd	Zn	Be	Mn	U	Th	Fe
units:	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	%
NIST 1632b (1)	4.23	ND	ND	ND	ND	ND	0.28	4.79	0.072	ND	ND	ND	0.428	ND	ND
NIST 1632b (1)	4.77	ND	ND	ND	ND	ND	0.27	4.46	0.071	ND	ND	ND	0.454	ND	ND
Mean	4.50						0.27	4.63	0.071				0.441		
1632b Acc Value (2)	3.72						0.24	3.67	0.058				0.436		
NIST 1633a	158	ND	ND	ND	ND	ND	5.35	88.4	1.21	ND	ND	ND	11.39	ND	ND
NIST 1633a	176	ND	ND	ND	ND	ND	7.54	77.5	1.28	ND	ND	ND	11.15	ND	ND
Mean	167						6.44	82.9	1.25				11.27		
1633a Acc. Value (3)	145						6.8	72.4	0.98				10.2		
NIST 1633b	191.6	ND	ND	ND	ND	ND	6.53	83.4	1.09	ND	ND	ND	10.7	ND	ND
NIST 1633b	164.5	ND	ND	ND	ND	ND	6.37	77.6	0.92	ND	ND	ND	9.52	ND	ND
Mean	178.0						6.45	80.5	1.01				10.1		
1633b Acc. Value (4)	136.2						6	68.2	1				8.79		
CLB-1 (100) -1 (5)	14	ND	ND	ND	ND	ND	1.26	4.35	0.11	ND	ND	ND	0.45	ND	ND
CLB-1 (100) -2 (5)	12	ND	ND	ND	ND	ND	1.35	4.47	0.13	ND	ND	ND	0.45	ND	ND
CLB-1 (100) -3 (5)	13	ND	ND	ND	ND	ND	1.32	3.28	0.12	ND	ND	ND	0.35	ND	ND
CLB-1 (400) -1 (6)	17	ND	ND	ND	ND	ND	1.34	4.03	0.12	ND	ND	ND	0.43	ND	ND
CLB-1 (400) -2 (6)	15	ND	ND	ND	ND	ND	1.41	4.28	0.13	ND	ND	ND	0.43	ND	ND
CLB-1 (400) -3 (6)	14	ND	ND	ND	ND	ND	1.39	3.53	0.12	ND	ND	ND	0.33	ND	ND
CLB-1 (S)-1	11	ND	ND	ND	ND	ND	0.59	5.94	0.05	ND	ND	ND	0.56	ND	ND
CLB-1 (S)-2	12	ND	ND	ND	ND	ND	0.66	5.86	0.07	ND	ND	ND	0.54	ND	ND
CLB-1 (S)-3	18	ND	ND	ND	ND	ND	1.76	6.39	0.11	ND	ND	ND	0.55	ND	ND
CLB-1 (S)-4	19	ND	ND	ND	ND	ND	1.88	6.02	0.11	ND	ND	ND	0.54	ND	ND
Mean	15						1.30	4.81	0.11				0.46		
Standard Deviation	3						0.41	1.13	0.02				0.09		
CLB-1 Acc. Value (7)	13						1.5	5	NR				0.55		
Ref 65-1	14	ND	ND	ND	ND	ND	10	38	11	ND	ND	ND	2.4	ND	ND
Ref 65-2	12	ND	ND	ND	ND	ND	11	40	12	ND	ND	ND	2.5	ND	ND
Ref 65-3	12	ND	ND	ND	ND	ND	10	40	11	ND	ND	ND	2.1	ND	ND
Mean	12						10	39	11				2.3		
Standard Deviation	1						1	1	1				0.21		
Accepted Value (8)	8.2						10	38	12				NR		

(1) Values based on dissolution of NIST 1632b coal ash reported on a whole coal basis
(2) Values taken from National Bureau of Standards Certificate of Analysis for 1632b
(3) Values taken from National Bureau of Standards Certificate of Analysis for 1633a
(4) Values taken from National Bureau of Standards Certificate of Analysis for 1633b
(5) Values based on acid digest of CLB-1 ash diluted 100x; recalulated to a whole coal basis
(6) Values based on acid digest of CLB-1 ash diluted 400x; recalulated to a whole coal basis
(7) U.S. Geological Survey Certificate of Anaysis
(8) U.S. Geological Survey Water Standard Certificate of Anaysis
ND = Not determined
NR = Not reported

Table 4. Control Samples

D. Cold Vapor (Hg) and Hydride Generation (Se) Atomic Absorption

Element:	Hg	Se
units:	ug/g	ug/g
CLB-1	0.19	3.04
CLB-1	0.12	2.86
CLB-1	0.12	2.85
CLB-1	0.13	1.77
Mean	0.14	2.63
Standard Deviation	0.029	0.502
Accepted Value (1)	0.15	2.5
1632-b	0.08	1.51
1632-b	0.05	1.24
1632-b	0.06	0.96
1632-b	0.05	1.19
Mean	0.06	1.23
Standard Deviation	0.01	0.20
Accepted Value (2)	0.068	1.29
1633-b	0.17	ND
1633-b	0.15	ND
1633-b	0.16	ND
1633-b	0.17	ND
Mean	0.16	
Standard Deviation	0.0083	
Accepted Value (3)	0.14	
1635-b	ND	1.1
1635-b	ND	0.81
1635-b	ND	0.74
1635-b	ND	0.86
Mean		0.87
Standard Deviation		0.13
Accepted Value (4)		0.9

(1) U.S. Geological Survey Certificate of Anaysis
(2) Values taken from National Bureau of Standards Certificate of Analysis for 1632b
(3) Values taken from National Bureau of Standards Certificate of Analysis for 1633b
(4) Values taken from National Bureau of Standards Certificate of Analysis for 1635b
ND = not determined

Table 5. Replicate Analyses of Selected Samples

A. INAA

Element	As	Hg	Cr	Se	Ni	Co	Sb	Pb	Cd	Zn	Be	Mn	Th	U	Fe
units:	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	%
NAPHNO3-1	0.50	ND	12.7	0.85	10	1.54	0.28	ND	ND	2.3	ND	ND	1.47	0.45	0.0142
ERROR:	0.070	ND	0.77	0.083	1.7	0.036	0.039	ND	ND	0.55	ND	ND	0.074	0.063	0.00084
NAPHNO3-1 Dup	0.52	ND	12.6	0.9	8	1.62	0.30	ND	ND	1.8	ND	ND	1.42	0.57	0.0164
ERROR:	0.079	ND	0.52	0.15	2.4	0.077	0.017	ND	ND	0.44	ND	ND	0.035	0.069	0.00078
NAPHNO3-2	0.49	ND	12.3	< 1.2	12	1.84	0.34	ND	ND	< 2.6	ND	ND	1.46	0.61	0.019
ERROR:	0.080	ND	0.63	0.00	3.7	0.079	0.027	ND	ND	0.00	ND	ND	0.051	0.092	0.0027
NAPHNO3-2 Dup	0.5	ND	13.0	< 1.3	< 24	1.9	0.34	ND	ND	< 5.0	ND	ND	1.43	0.35	0.015
ERROR:	0.10	ND	0.72	0.00	0.0	0.10	0.035	ND	ND	0.00	ND	ND	0.055	0.087	0.0018
SAPHNO3-2	2.0	ND	20.6	< 0.8	< 30	4.8	1.47	ND	ND	6	ND	ND	4.6	1.2	0.027
ERROR:	0.15	ND	0.81	0.00	0.0	0.22	0.054	ND	ND	1.6	ND	ND	0.19	0.14	0.0025
SAPHNO3-2 Dup	1.7	ND	20.1	< 0.8	< 15	4.7	1.5	ND	ND	6	ND	ND	4.3	1.1	0.030
ERROR:	0.16	ND	0.77	0.00	0.0	0.22	0.10	ND	ND	1.5	ND	ND	0.30	0.15	0.0020
EIHNO3-2	0.39	ND	28	2.7	19	0.80	0.49	ND	ND	< 5.0	ND	ND	1.08	1.3	0.052
ERROR:	0.061	ND	1.3	0.22	3.6	0.063	0.035	ND	ND	0.00	ND	ND	0.052	0.13	0.0046
EIHNO3-2 Dup	0.44	ND	27	3.0	23	0.82	0.54	ND	ND	3.4	ND	ND	1.03	1.5	0.047
ERROR:	0.068	ND	1.3	0.21	4.9	0.090	0.062	ND	ND	0.92	ND	ND	0.042	0.14	0.0025
PRHNO3-2	0.40	ND	1.6	0.4	< 7.0	0.23	0.21	ND	ND	< 2.8	ND	ND	0.76	0.20	0.021
ERROR:	0.049	ND	0.21	0.11	0.00	0.018	0.016	ND	ND	0.00	ND	ND	0.030	0.051	0.0015
PRHNO3-2 Dup	0.39	ND	1.2	0.4	< 15	0.21	0.18	ND	ND	< 2.0	ND	ND	0.81	0.17	0.015
ERROR:	0.052	ND	0.34	0.13	0.0	0.019	0.013	ND	ND	0.00	ND	ND	0.029	0.039	0.0011
CLBHNO3-2	0.61	ND	9.1	1.2	< 25	1.42	0.54	ND	ND	4.8	ND	ND	0.98	0.27	0.051
ERROR:	0.088	ND	0.74	0.14	0.0	0.070	0.047	ND	ND	0.85	ND	ND	0.065	0.068	0.0022
CLBHNO3-2 Dup	0.7	ND	7.6	1.2	< 30	1.48	0.60	ND	ND	4	ND	ND	1.03	0.4	0.060
ERROR:	0.12	ND	0.56	0.16	0.0	0.069	0.034	ND	ND	1.0	ND	ND	0.096	0.10	0.0042

SAP = Southern Appalachian
NAP = Northern Appalachian
EI = Eastern Interior
PR = Powder River Basin
ND = Not Determined

Table 5. Replicate Analyses of Selected Samples

B. ICP

Element	As	Hg	Cr	Se	Ni	Co	Sb	Pb	Cd	Zn	Be	Mn	Th	U	Fe
units:	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g
SAPHNO1	ND	ND	0.83	ND	0.33	0.15	ND	ND	ND	0.61	0.0058	1.2	0.33	ND	180
SAPHNO1Dup	ND	ND	1.2	ND	0.50	0.22	ND	ND	ND	0.90	0.0017	1.2	0.66	ND	250
SAPHNO2	ND	ND	0.83	ND	0.32	0.15	ND	ND	ND	0.64	0.0058	1	0.3	ND	180
SAPHNO2 Dup	ND	ND	0.88	ND	0.35	0.16	ND	ND	ND	0.69	0.0007	0.9	0.4	ND	190
SAPHF1	ND	ND	3	ND	0.7	0.2	ND	ND	ND	1.2	0.1	4	0.2	ND	600
SAPHF1 Dup	ND	ND	3	ND	0.7	0.2	ND	ND	ND	1.2	0.1	3	0.2	ND	570
SAPHF2	ND	ND	7	ND	18	3	ND	ND	ND	38	1	8	0.2	ND	7700
SAPHF2 Dup	ND	ND	7	ND	19	3	ND	ND	ND	40	1	10	0.2	ND	8100
EICl1	ND	ND	1.4	ND	0.38	0.095	ND	ND	ND	3.3	< 0.02	0.65	< 0.08	ND	170
EICl1 Dup	ND	ND	1.2	ND	0.38	0.090	ND	ND	ND	3.7	0.0053	0.65	0.14	ND	160
EICl2	ND	ND	0.087	ND	0.20	0.056	ND	ND	ND	1.5	< 0.02	0.57	< 0.08	ND	42
EICl2 Dup	ND	ND	0.088	ND	0.23	0.062	ND	ND	ND	1.6	0.0025	0.62	0.08	ND	43
SAPCl1	ND	ND	1.4	ND	1.4	0.57	ND	ND	ND	4.0	0.037	13	0.15	ND	1200
SAPCl1 Dup	ND	ND	1.2	ND	1.20	0.50	ND	ND	ND	3.6	0.034	12	0.17	ND	1100
SAPCl2	ND	ND	0.67	ND	0.30	0.14	ND	ND	ND	1.3	< 0.02	1.4	< 0.08	ND	220
SAPCl2 Dup	ND	ND	0.62	ND	0.28	0.14	ND	ND	ND	1.4	0.0088	1.3	0.30	ND	200

SAP = Southern Appalachian
EI = Eastern Interior
ND = Not Determined

Note: Values given are concentrations in solution

Table 6. Modes of Occurrence of Trace Elements in As-Mined Coals and Fines

Arsenic

	Pyrite	Arsenates	Silicates
Southern Appalachian As-Mined	75%	5%	20%
Southern Appalachian Cleaned Fines	80%	15%	5%
Eastern Interior As-Mined	60%	20%	15%
Eastern Interior Cleaned Fines	70%	25%	5%
Northern Appalachian As-Mined	70%	20%	5%
Northern Appalachian Cleaned Fines	70%	30%	0%
Powder River Basin As-Mined	25%	40%	25%
Powder River Basin Natural Fines	30%	40%	25%

Mercury

	Pyrite	Organic	HCl Soluble Sulfides
Southern Appalachian As-Mined	35%	35%	30%
Southern Appalachian Cleaned Fines	30%	36%	35%
Eastern Interior As-Mined	60%	25%	15%
Eastern Interior Cleaned Fines	65%	25%	10%
Northern Appalachian As-Mined	60%	35%	5%
Northern Appalachian Cleaned Fines	45%	25%	30%
Powder River Basin As-Mined	25%	40%	30%
Powder River Basin Natural Fines	55%	10%	30%

Chromium

	Illite	Sulfides	Oxyhydroxides(?)
Southern Appalachian As-Mined	75%	10%	10%
Southern Appalachian Cleaned Fines	50%	45%	5%
Eastern Interior As-Mined	80%	15%	
Eastern Interior Cleaned Fines	60%	15%	20%
Northern Appalachian As-Mined	75%	15%	10%
Northern Appalachian Cleaned Fines	60%	20%	25%
Powder River Basin As-Mined	70%	20%	10%
Powder River Basin Natural Fines	70%	20%	10%

Table 6. Modes of Occurrence of Trace Elements in As-Mined Coals and Fines (Continued)

Selenium

	Pyrite	Organic	Silicates	Accessory and Mono-Sulfides
Southern Appalachian As-Mined	55%	20%	15%	10%
Southern Appalachian Cleaned Fines	35%	35%	5%	30%
Eastern Interior As-Mined	55%	45%		
Eastern Interior Cleaned Fines	25%	15%	10%	50%
Northern Appalachian As-Mined	35%	65%		
Northern Appalachian Cleaned Fines	30%	45%		25%
Powder River Basin As-Mined	10%	75%		15%
Powder River Basin Natural Fines	25%	20%		55%

Nickel

	Sulfides	Ni oxides	Silicates	Organics
Southern Appalachian As-Mined	15%	20%	40%	20%
Southern Appalachian Cleaned Fines	15%	30%	20%	25%
Eastern Interior As-Mined	25%	15%	20%	40%
Eastern Interior Cleaned Fines	40%	10%	15%	35%
Northern Appalachian As-Mined	15%	25%	20%	40%
Northern Appalachian Cleaned Fines	30%	15%	20%	35%
Powder River Basin As-Mined	15%	40%	30%	15%
Powder River Basin Natural Fines	15%	40%	30%	15%

Cobalt

	Sulfides	HCl Soluble	Silicates	Organics
Southern Appalachian As-Mined	20%	30%	20%	30%
Southern Appalachian Cleaned Fines	15%	25%	5%	55%
Eastern Interior As-Mined	20%	35%	25%	20%
Eastern Interior Cleaned Fines	25%	25%	10%	40%
Northern Appalachian As-Mined	25%	30%	20%	25%
Northern Appalachian Cleaned Fines	20%	35%	20%	30%
Powder River Basin As-Mined	5%	60%	15%	20%
Powder River Basin Natural Fines	5%	65%	15%	15%

Table 6. Modes of Occurrence of Trace Elements in As-Mined Coals and Fines (Continued)

Antimony

	Sulfides	Silicates	Oxides	Organic
Southern Appalachian As-Mined	20%	15%	15%	50%
Southern Appalachian Cleaned Fines	25%	10%	25%	40%
Eastern Interior As-Mined	40%	15%	0%	40%
Eastern Interior Cleaned Fines	50%	10%	0%	40%
Northern Appalachian As-Mined	10%	15%	10%	60%
Northern Appalachian Cleaned Fines	25%	15%	10%	55%
Powder River Basin As-Mined	15%	30%	0%	50%
Powder River Basin Natural Fines	20%	25%	0%	50%

Lead

	Galena	Organics	Silicates
Southern Appalachian As-Mined	90%	5%	5%
Southern Appalachian Cleaned Fines	85%	5%	10%
Eastern Interior As-Mined	100%		
Eastern Interior Cleaned Fines	70%	30%	
Northern Appalachian As-Mined	100%		
Northern Appalachian Cleaned Fines	90%		10%
Powder River Basin As-Mined	35%	65%	
Powder River Basin Natural Fines	ND	ND	ND

Cadmium

	Sphalerite	Silicates	Organic
Southern Appalachian As-Mined	ND	ND	ND
Southern Appalachian Cleaned Fines	ND	ND	ND
Eastern Interior As-Mined	85%	10%	5%
Eastern Interior Cleaned Fines	70%	20%	10%
Northern Appalachian As-Mined	ND	ND	ND
Northern Appalachian Cleaned Fines	50%	30%	20%
Powder River Basin As-Mined	ND	ND	ND
Powder River Basin Natural Fines	80%	15%	5%

Table 6. Modes of Occurrence of Trace Elements in As-Mined Coals and Fines (Continued)

Zinc

	Sphalerite	Silicates	Organic
Southern Appalachian As-Mined	65%	30%	5%
Southern Appalachian Cleaned Fines	80%	10%	10%
Eastern Interior As-Mined	85%	5%	10%
Eastern Interior Cleaned Fines	65%	10%	25%
Northern Appalachian As-Mined	65%	20%	15%
Northern Appalachian Cleaned Fines	50%	15%	35%
Powder River Basin As-Mined	40%	35%	25%
Powder River Basin Natural Fines	85%	5%	10%

Manganese

	Carbonates	Sulfides	Silicates	Organics
Southern Appalachian As-Mined	65%	10%	25%	0%
Southern Appalachian Cleaned Fines	60%	0%	20%	20%
Eastern Interior As-Mined	40%	20%	15%	25%
Eastern Interior Cleaned Fines	15%	35%	15%	35%
Northern Appalachian As-Mined	50%	15%	15%	20%
Northern Appalachian Cleaned Fines	30%	10%	35%	25%
Powder River Basin As-Mined	85%	0%	5%	10%
Powder River Basin Natural Fines	70%	0%	5%	25%

Beryllium

	Silicates	Oxides	Sulfides	Organics
Southern Appalachian As-Mined	70%	20%	10%	
Southern Appalachian Cleaned Fines	60%	40%	0%	
Eastern Interior As-Mined	70%	20%	10%	
Eastern Interior Cleaned Fines	85%	0%	0%	15%
Northern Appalachian As-Mined	70%	20%	10%	
Northern Appalachian Cleaned Fines	100%	0%	0%	
Powder River Basin As-Mined	25%	50%	25%	
Powder River Basin Natural Fines	100%	0%	0%	

Table 6. Modes of Occurrence of Trace Elements in As-Mined Coals and Fines (Continued).

Thorium

	Phosphates	Insoluble	Silicates	HCl Soluble
Southern Appalachian As-Mined	30%	30%	25%	15%
Southern Appalachian Cleaned Fines	45%	30%	5%	20%
Eastern Interior As-Mined	20%	30%	15%	35%
Eastern Interior Cleaned Fines	40%	20%	0%	40%
Northern Appalachian As-Mined	40%	30%	0%	30%
Northern Appalachian Cleaned Fines	45%	30%	0%	25%
Powder River Basin As-Mined	10%	50%	35%	0%
Powder River Basin Natural Fines	35%	30%	20%	15%

Uranium

	Silicates	HCl Soluble	Oxides	Organics
Southern Appalachian As-Mined	45%	10%	15%	30%
Southern Appalachian Cleaned Fines	20%	10%	10%	60%
Eastern Interior As-Mined	20%	30%	0%	50%
Eastern Interior Cleaned Fines	30%	10%	15%	45%
Northern Appalachian As-Mined	30%	10%	5%	55%
Northern Appalachian Cleaned Fines	20%	10%	20%	50%
Powder River Basin As-Mined	35%	35%	5%	20%
Powder River Basin Natural Fines	40%	45%	5%	10%

Iron

	Pyrite	Silicates	Carbonates and Sulfates
Southern Appalachian As-Mined	20%	35%	45%
Southern Appalachian Cleaned Fines	25%	25%	50%
Eastern Interior As-Mined	60%	20%	15%
Eastern Interior Cleaned Fines	60%	15%	20%
Northern Appalachian As-Mined	60%	25%	10%
Northern Appalachian Cleaned Fines	55%	25%	20%
Powder River Basin As-Mined	75%	10%	10%
Powder River Basin Natural Fines	65%	15%	20%

Figure 1

SEM Comparison of as-mined coal samples and cleaned fines

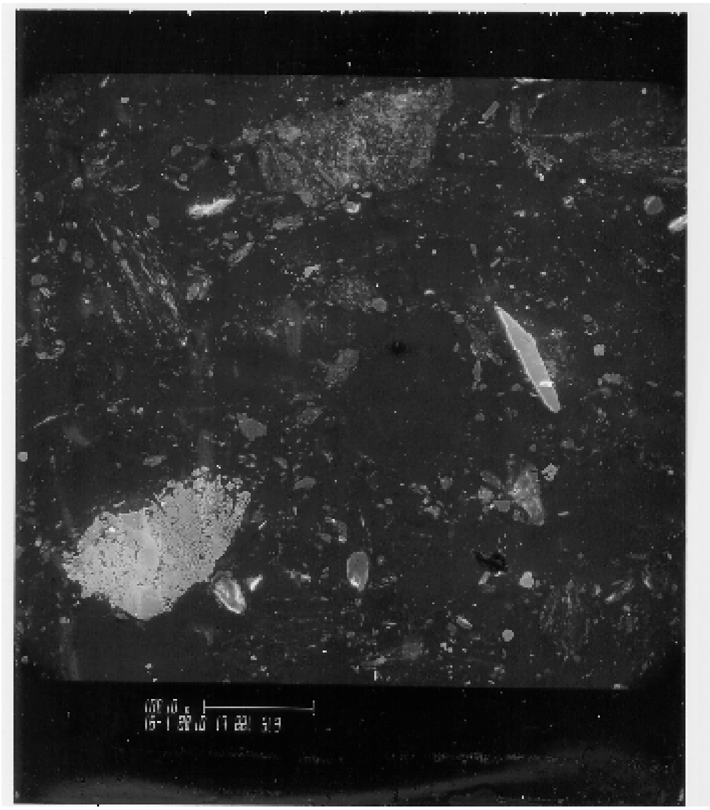


Figure 1a. As-Mined Eastern Interior Basin Coal Sample

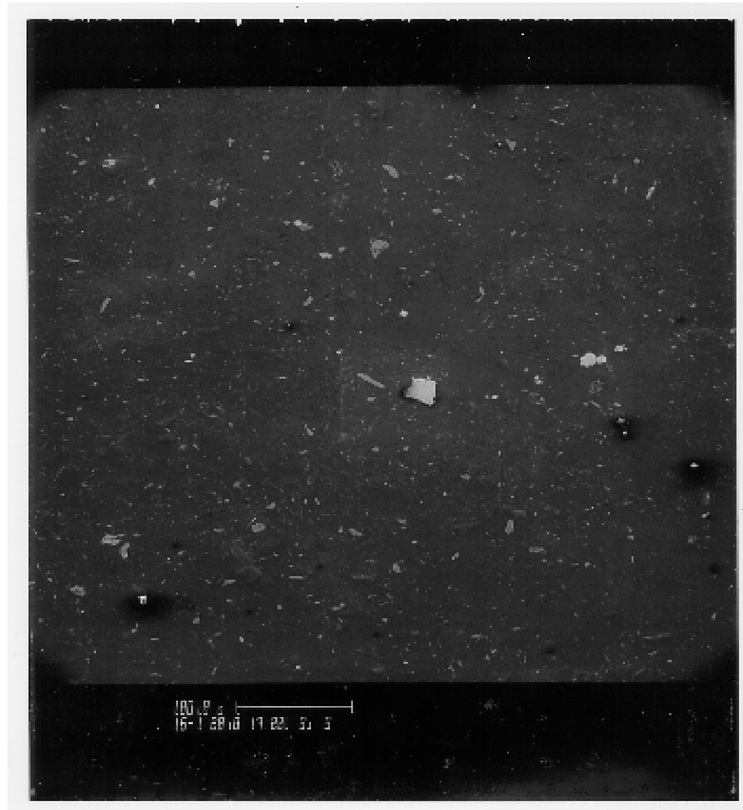


Figure 1b. Cleaned Fines--Eastern Interior Basin

Figure 2a

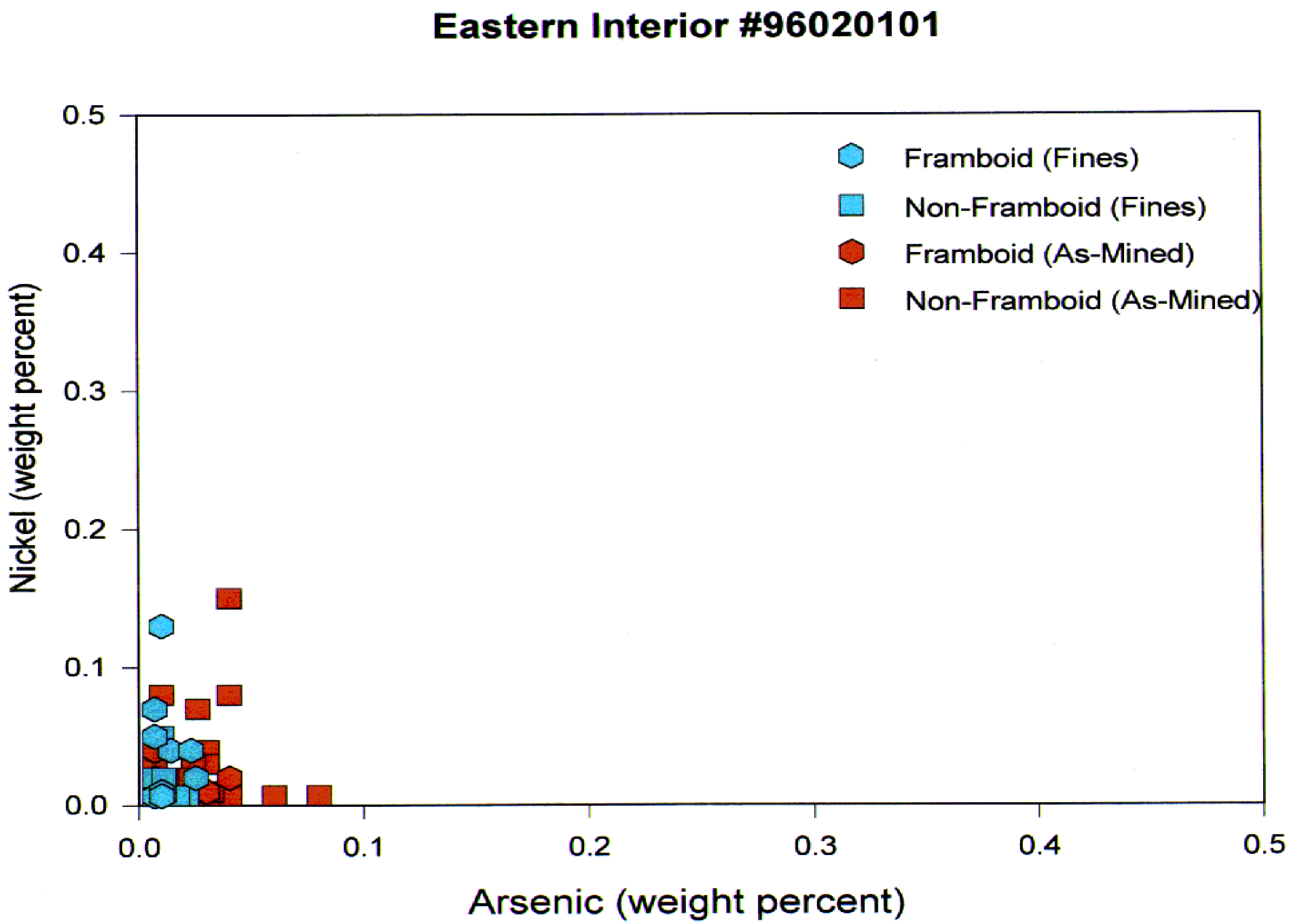

Eastern Interior #96020101
Framboid (Fines)
Non-Framboid (Fines)
Framboid (As-Mined)
Non-Framboid (As-Mined)
Nickel (weight percent)
Arsenic (weight percent)
0.0
0.1
0.2
0.3
0.4
0.5
0.0
0.1
0.2
0.3
0.4
0.5

Figure 2b

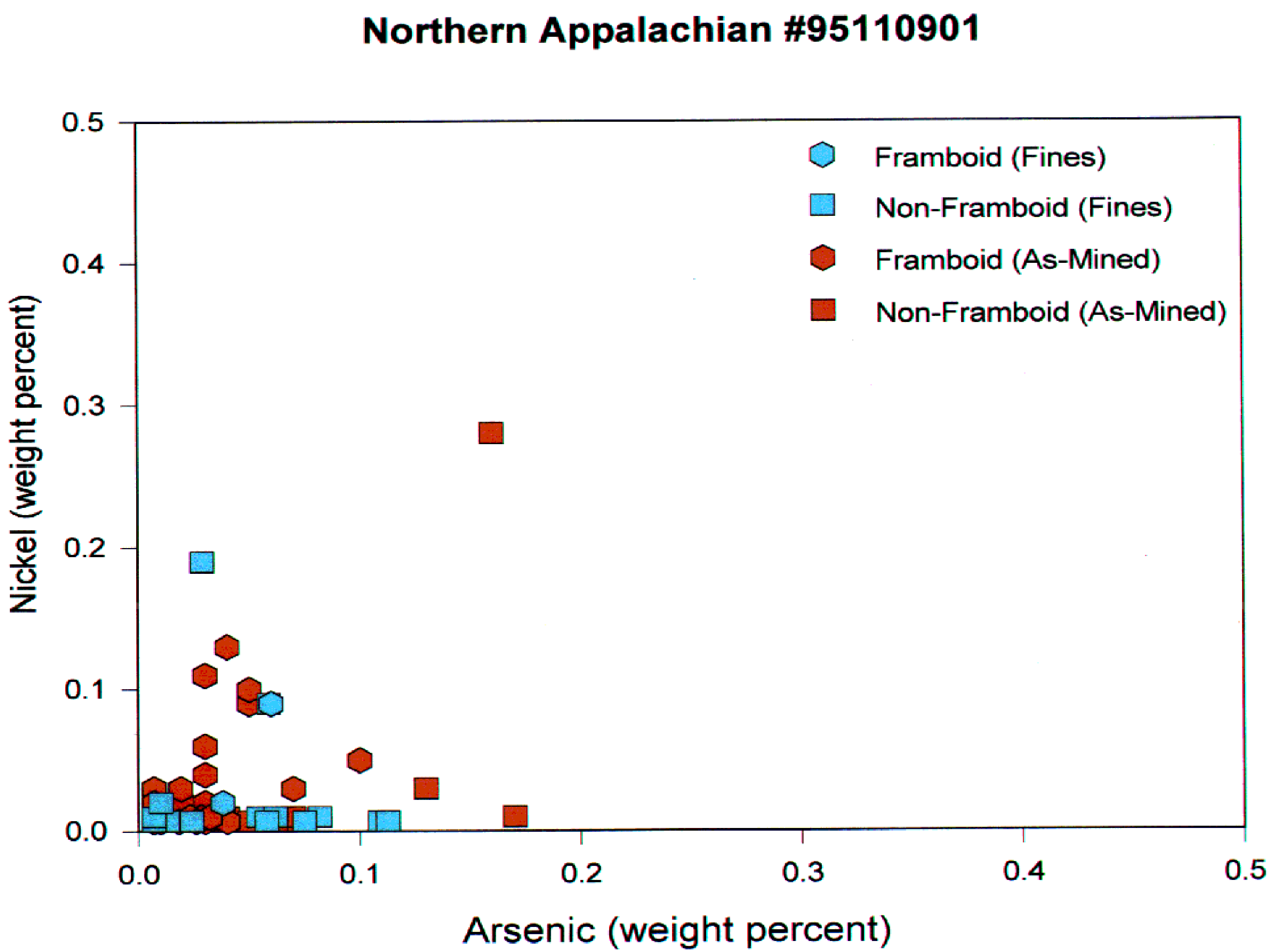

Northern Appalachian #95110901
Framboid (Fines)
Non-Framboid (Fines)
Framboid (As-Mined)
Non-Framboid (As-Mined)
Nickel (weight percent)
0.0
0.1
0.2
0.3
0.4
0.5
Arsenic (weight percent)
0.0
0.1
0.2
0.3
0.4
0.5

Figure 2c

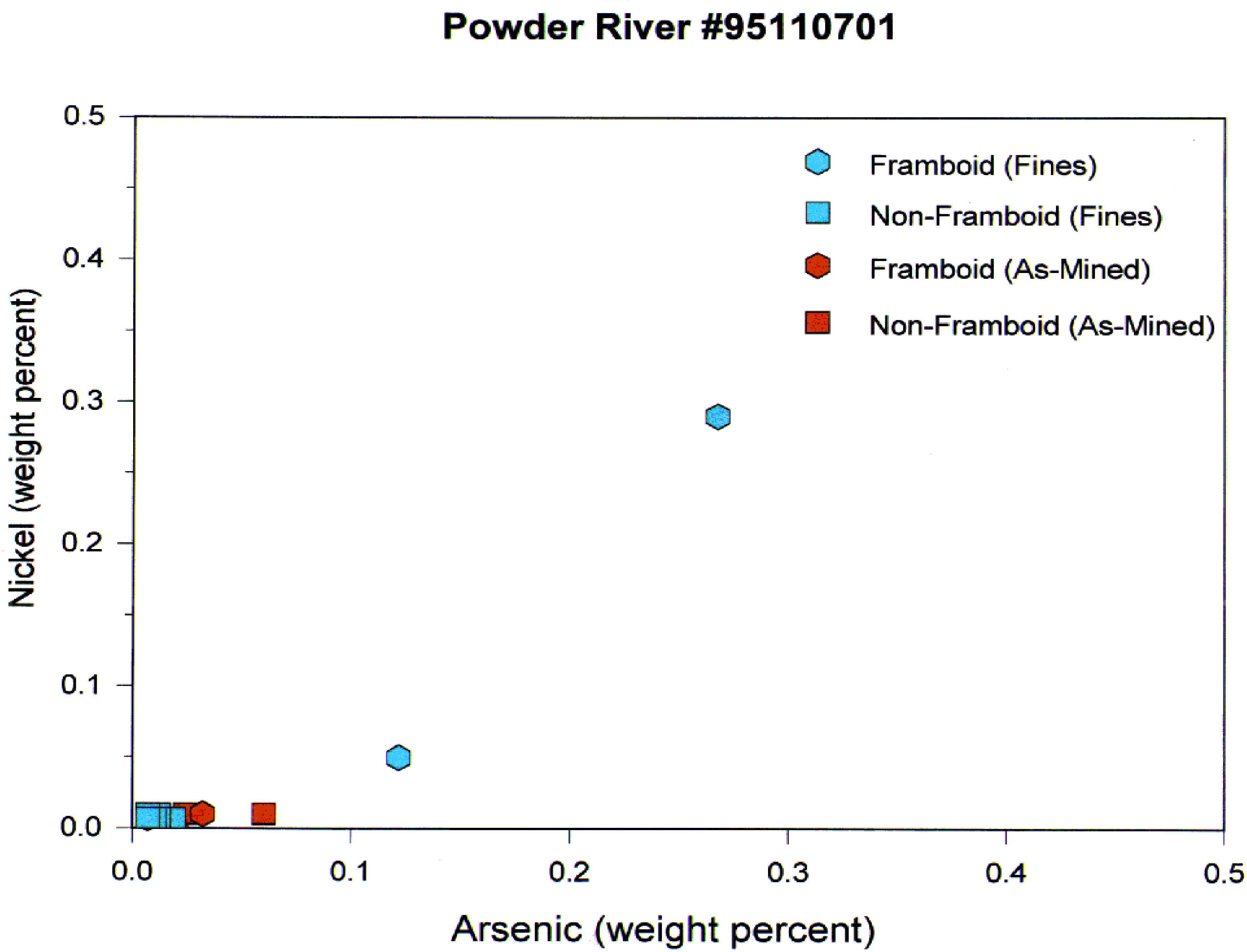
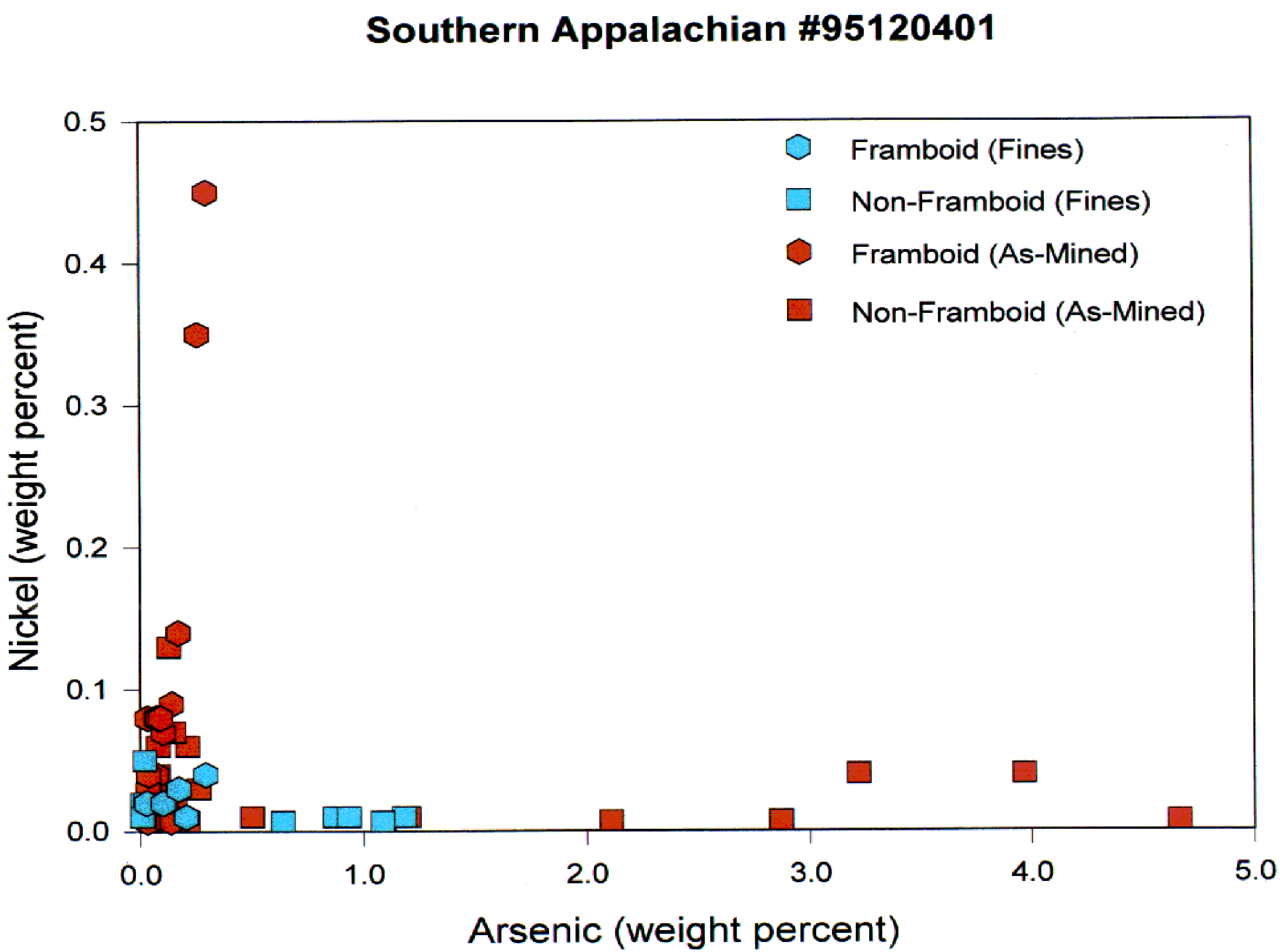

Powder River #95110701
Framboid (Fines)
Non-Framboid (Fines)
Framboid (As-Mined)
Non-Framboid (As-Mined)
Nickel (weight percent)
Arsenic (weight percent)
0.0
0.1
0.2
0.3
0.4
0.5
0.0
0.1
0.2
0.3
0.4
0.5

Figure 2d



Southern Appalachian #95120401
Framboid (Fines)
Non-Framboid (Fines)
Framboid (As-Mined)
Non-Framboid (As-Mined)
Nickel (weight percent)
Arsenic (weight percent)
0.0
0.1
0.2
0.3
0.4
0.5
0.0
1.0
2.0
3.0
4.0
5.0

Figure 3

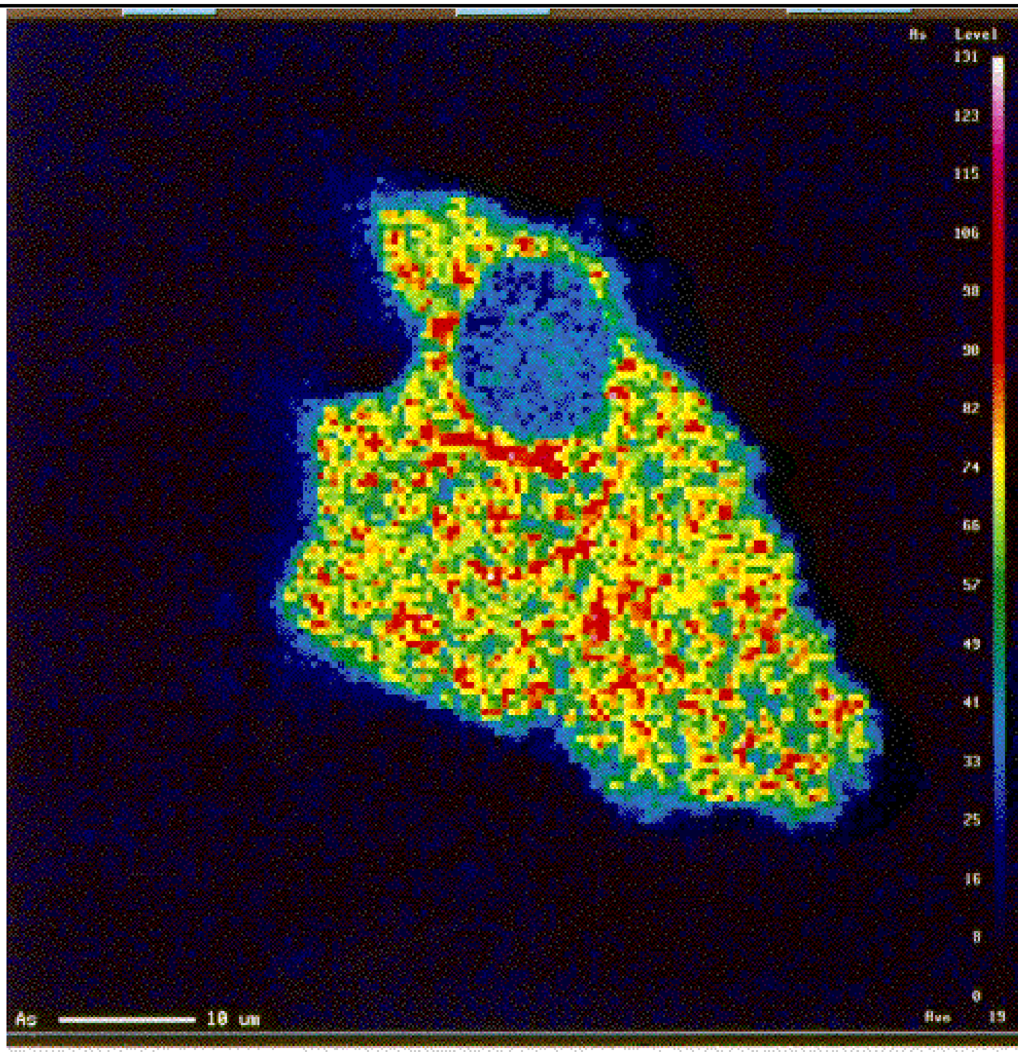

As
Level
131
123
115
106
98
90
82
74
66
57
49
41
33
25
16
8
0
As
10 um
Ave
19

Figure 4a

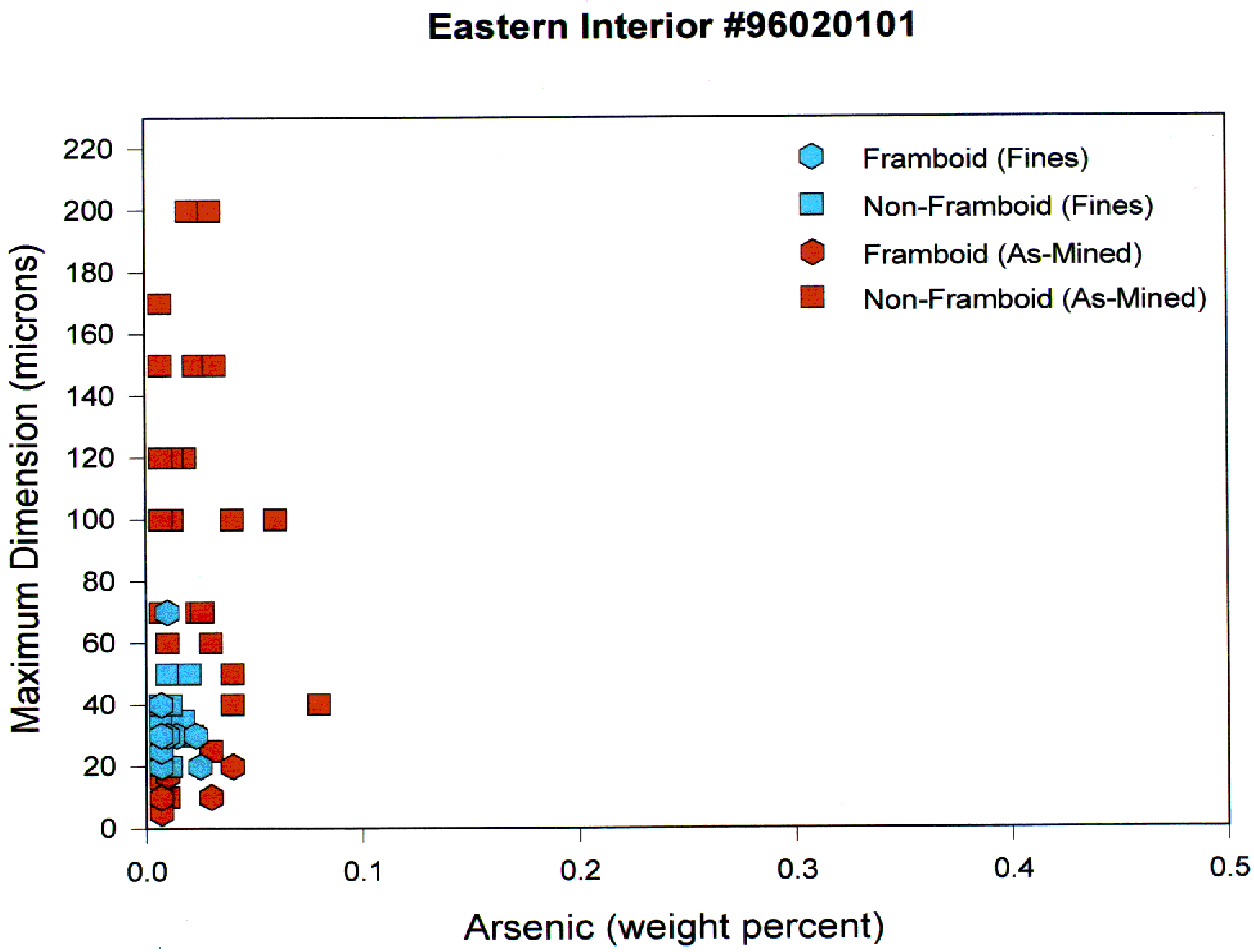
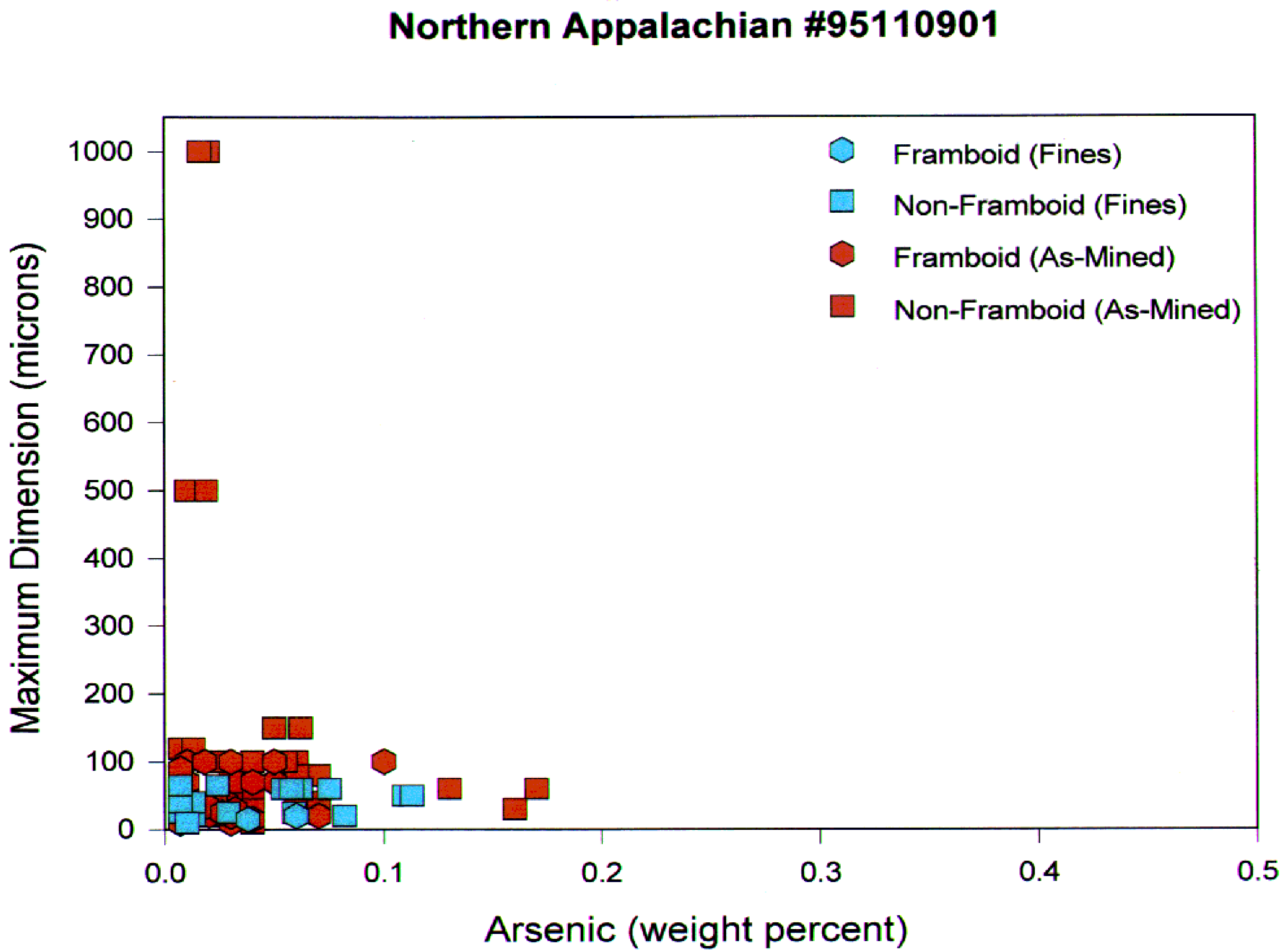
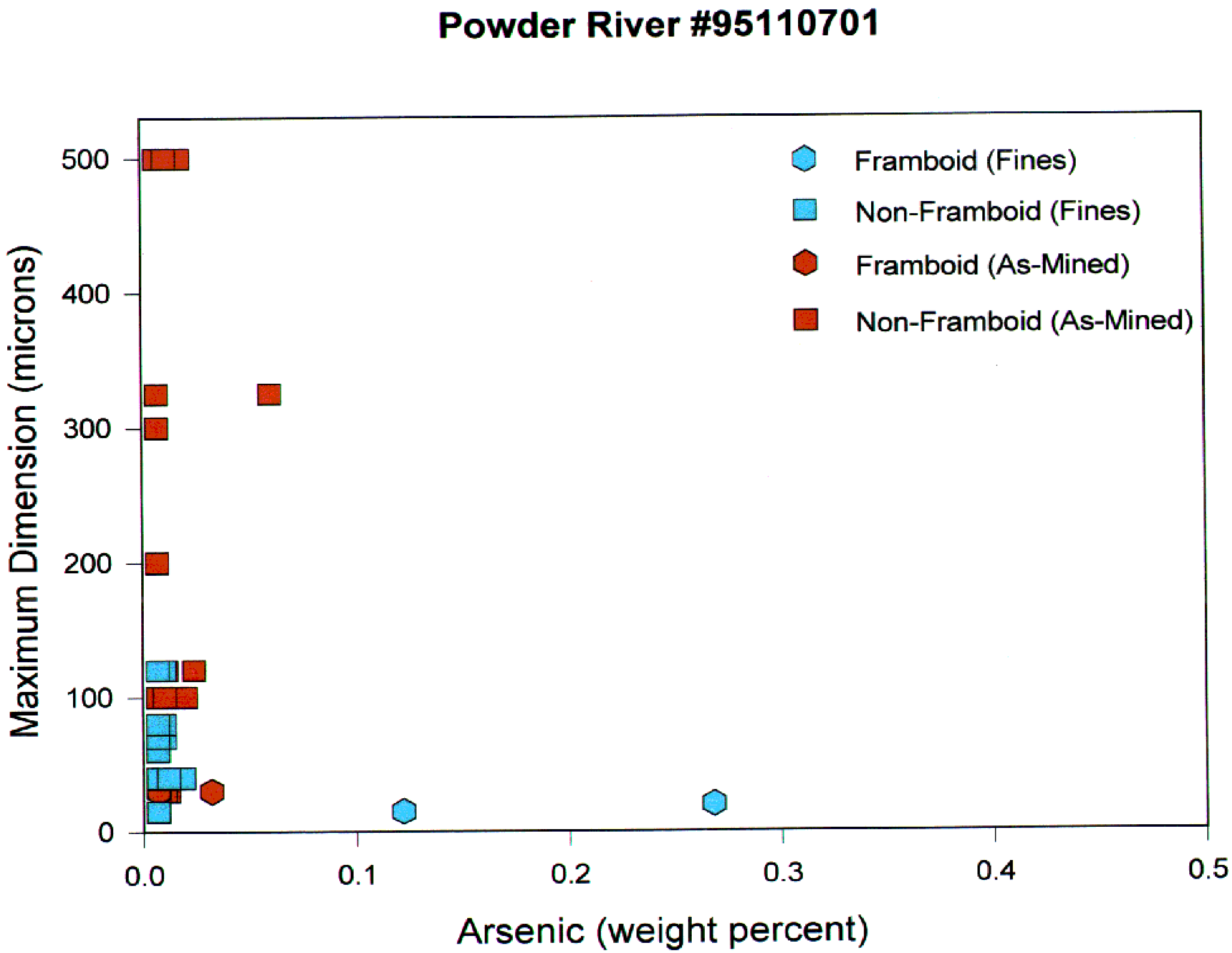

Eastern Interior #96020101
Framboid (Fines)
Non-Framboid (Fines)
Framboid (As-Mined)
Non-Framboid (As-Mined)
Maximum Dimension (microns)
0
20
40
60
80
100
120
140
160
180
200
220
0.0
0.1
0.2
0.3
0.4
0.5
Arsenic (weight percent)

Figure 4b



Northern Appalachian #95110901
Framboid (Fines)
Non-Framboid (Fines)
Framboid (As-Mined)
Non-Framboid (As-Mined)
Maximum Dimension (microns)
0
100
200
300
400
500
600
700
800
900
1000
0.0
0.1
0.2
0.3
0.4
0.5
Arsenic (weight percent)

Figure 4c



Powder River #95110701
Framboid (Fines)
Non-Framboid (Fines)
Framboid (As-Mined)
Non-Framboid (As-Mined)
Maximum Dimension (microns)
500
400
300
200
100
0
0.0
0.1
0.2
0.3
0.4
0.5
Arsenic (weight percent)

Figure 4d

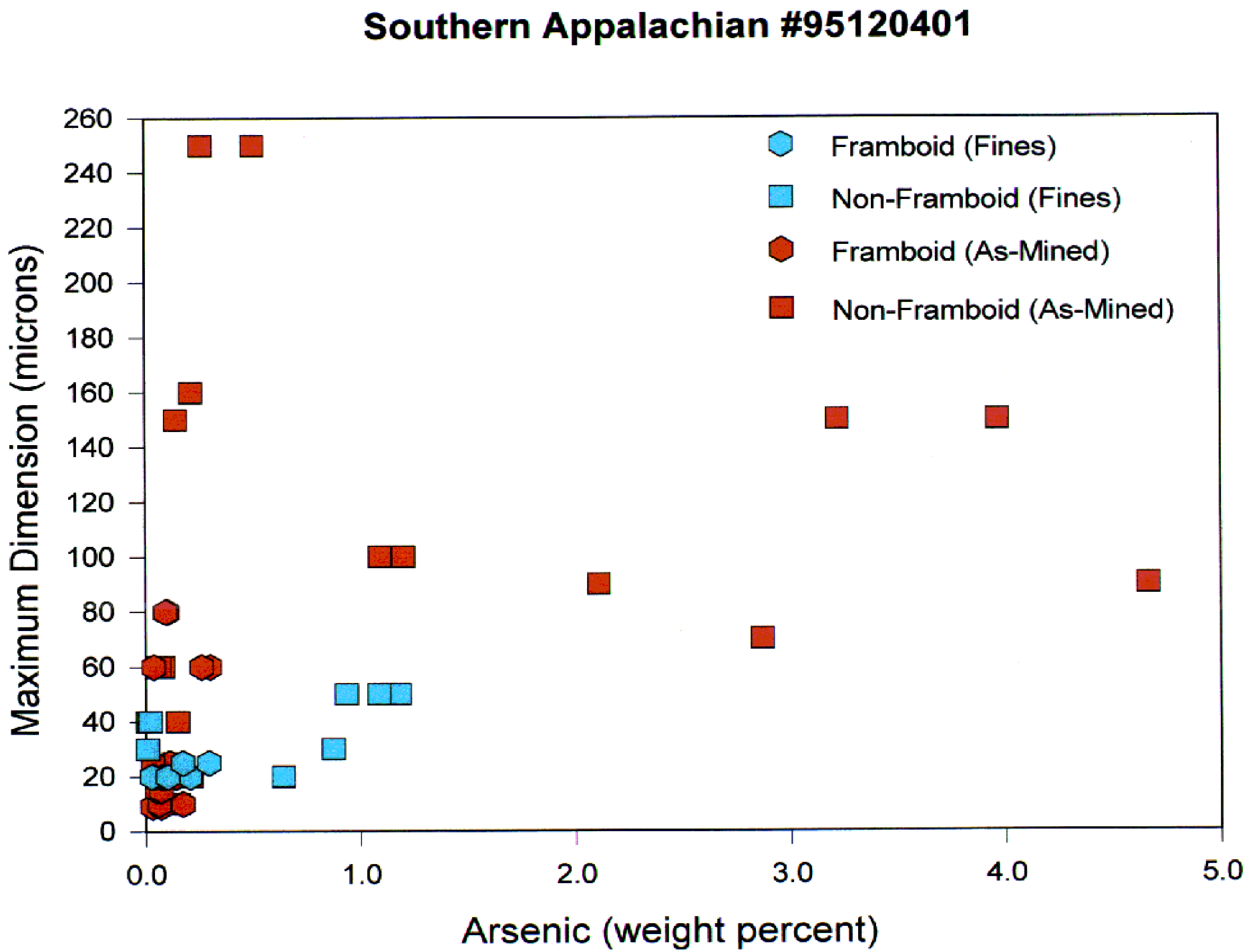
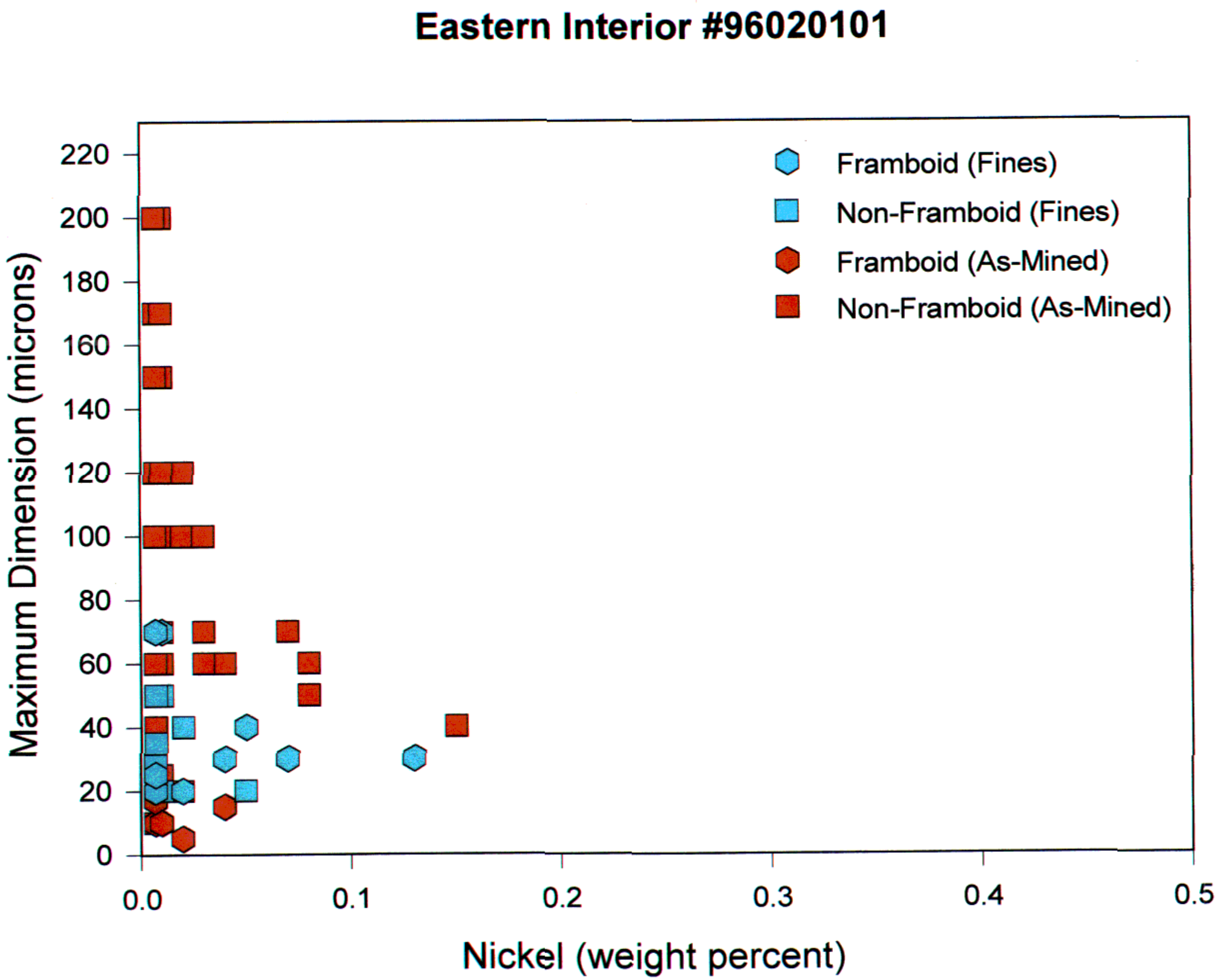
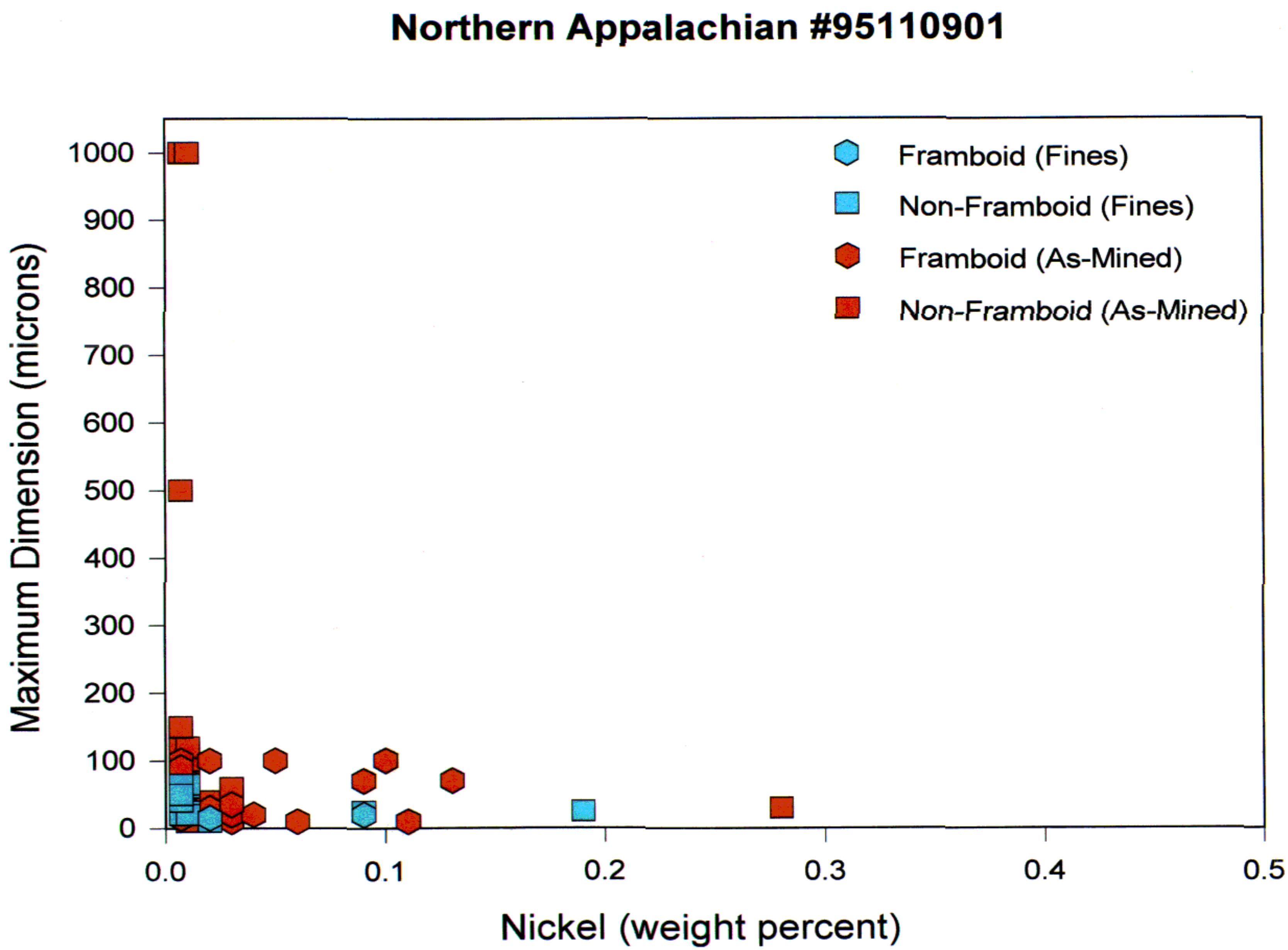

Southern Appalachian #95120401
Framboid (Fines)
Non-Framboid (Fines)
Framboid (As-Mined)
Non-Framboid (As-Mined)
Maximum Dimension (microns)
0
20
40
60
80
100
120
140
160
180
200
220
240
260
Arsenic (weight percent)
0.0
1.0
2.0
3.0
4.0
5.0

Figure 5a



Eastern Interior #96020101
Framboid (Fines)
Non-Framboid (Fines)
Framboid (As-Mined)
Non-Framboid (As-Mined)
Maximum Dimension (microns)
0
20
40
60
80
100
120
140
160
180
200
220
0.0
0.1
0.2
0.3
0.4
0.5
Nickel (weight percent)

Figure 5b



Northern Appalachian #95110901
Framboid (Fines)
Non-Framboid (Fines)
Framboid (As-Mined)
Non-Framboid (As-Mined)
Maximum Dimension (microns)
1000
900
800
700
600
500
400
300
200
100
0
0.0
0.1
0.2
0.3
0.4
0.5
Nickel (weight percent)

Figure 5c

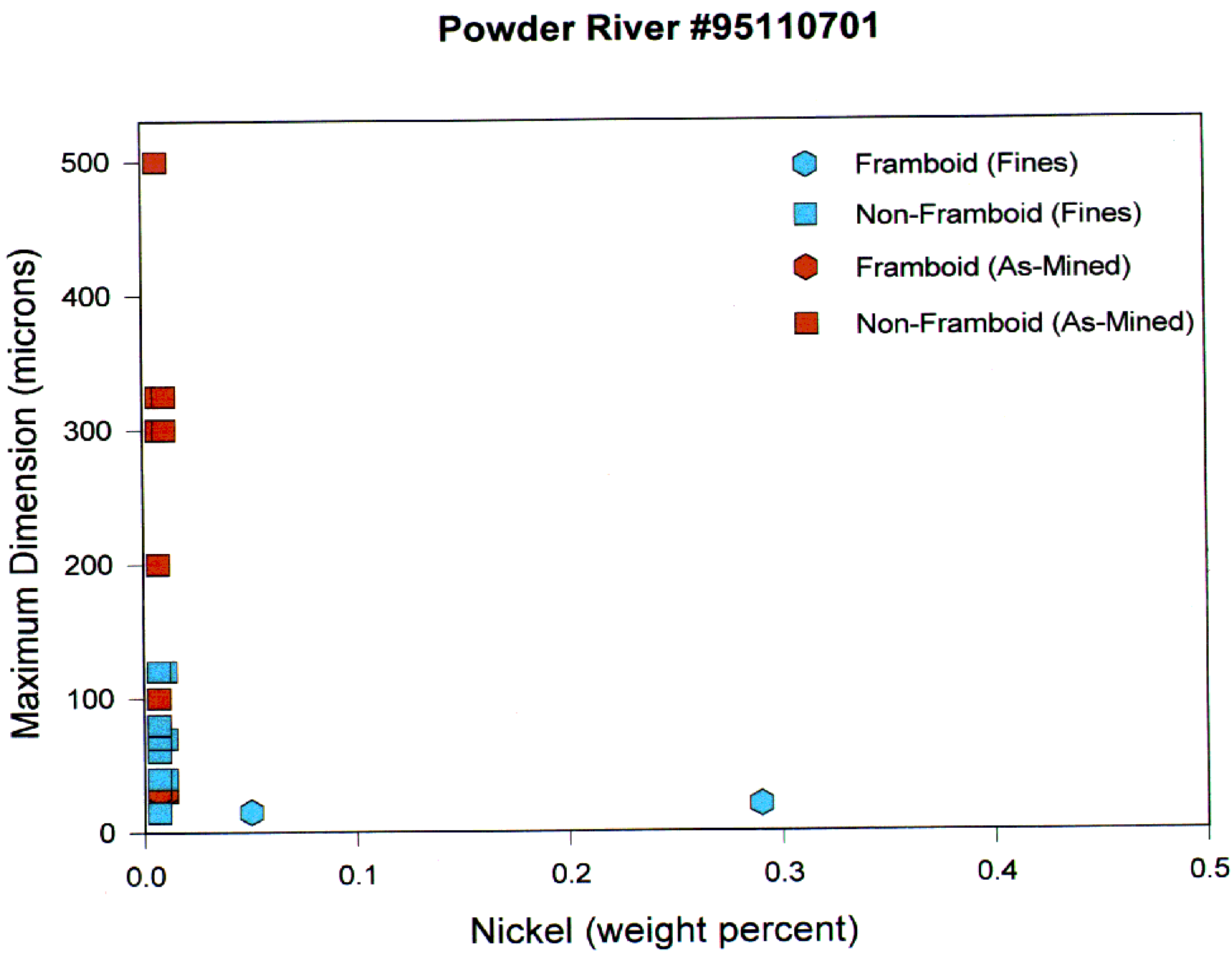

Powder River #95110701
Framboid (Fines)
Non-Framboid (Fines)
Framboid (As-Mined)
Non-Framboid (As-Mined)
Maximum Dimension (microns)
500
400
300
200
100
0
0.0
0.1
0.2
0.3
0.4
0.5
Nickel (weight percent)

Figure 5d

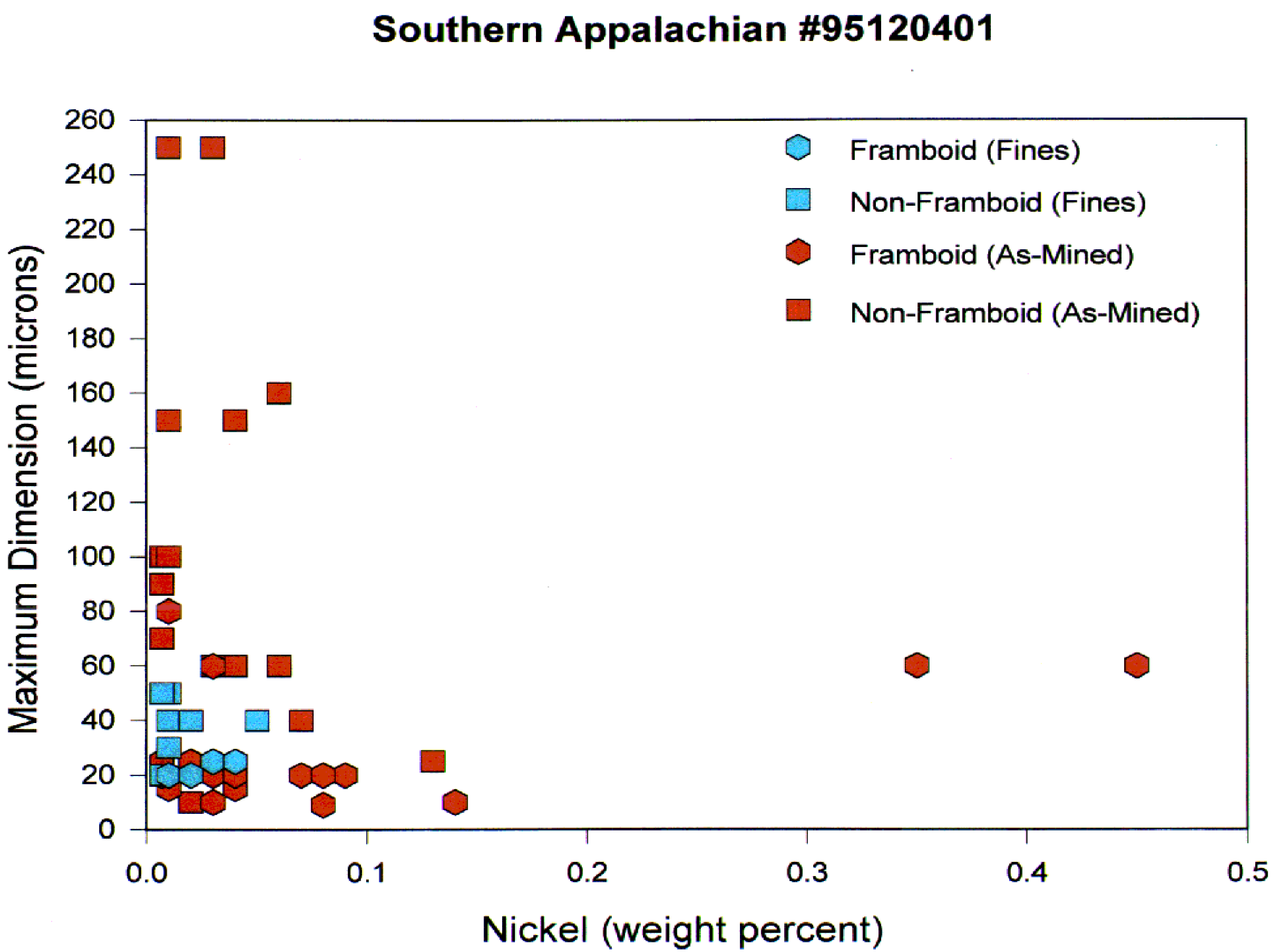

Southern Appalachian #95120401
Framboid (Fines)
Non-Framboid (Fines)
Framboid (As-Mined)
Non-Framboid (As-Mined)
Maximum Dimension (microns)
260
240
220
200
180
160
140
120
100
80
60
40
20
0
0.0
0.1
0.2
0.3
0.4
0.5
Nickel (weight percent)

Figure 6

Arsenic Mass Balance (As-Mined)

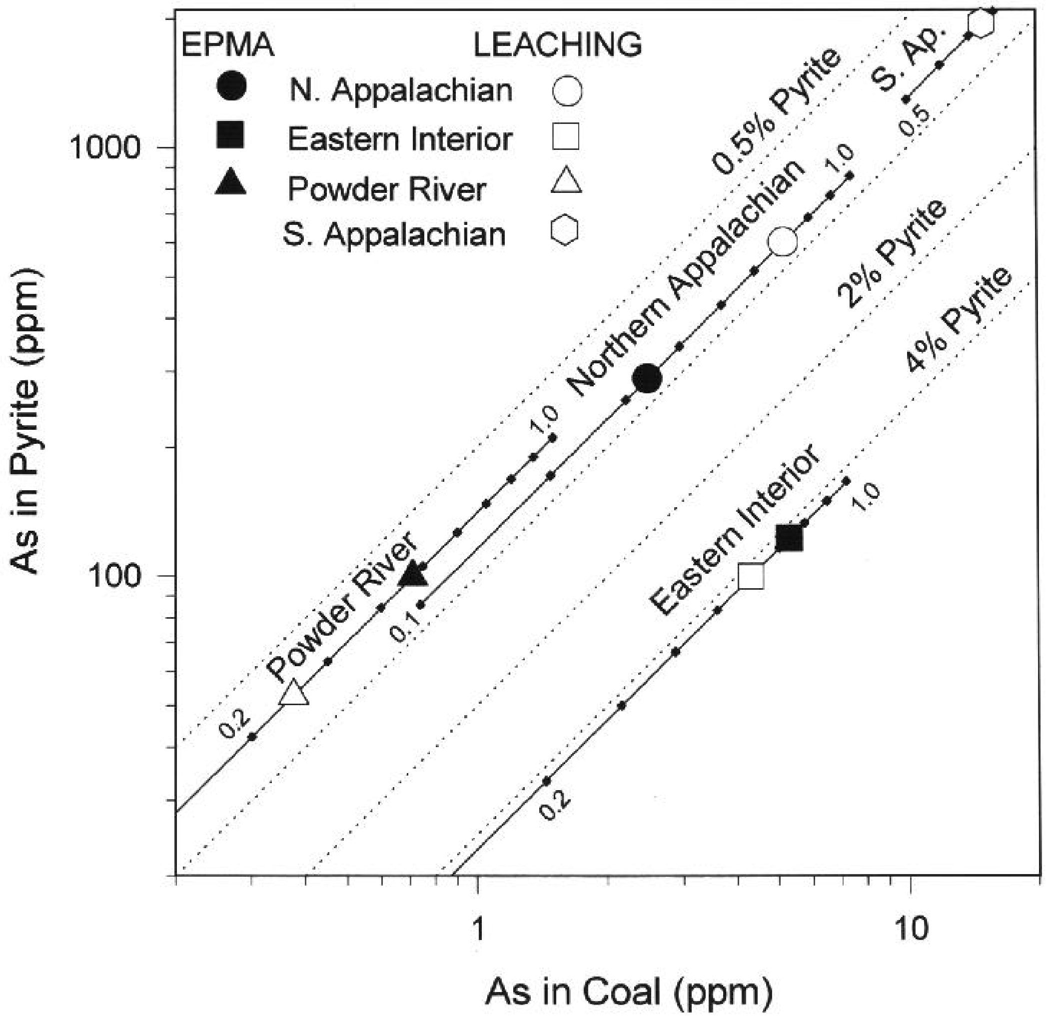


Figure 7

Nickel Mass Balance (As-Mined)

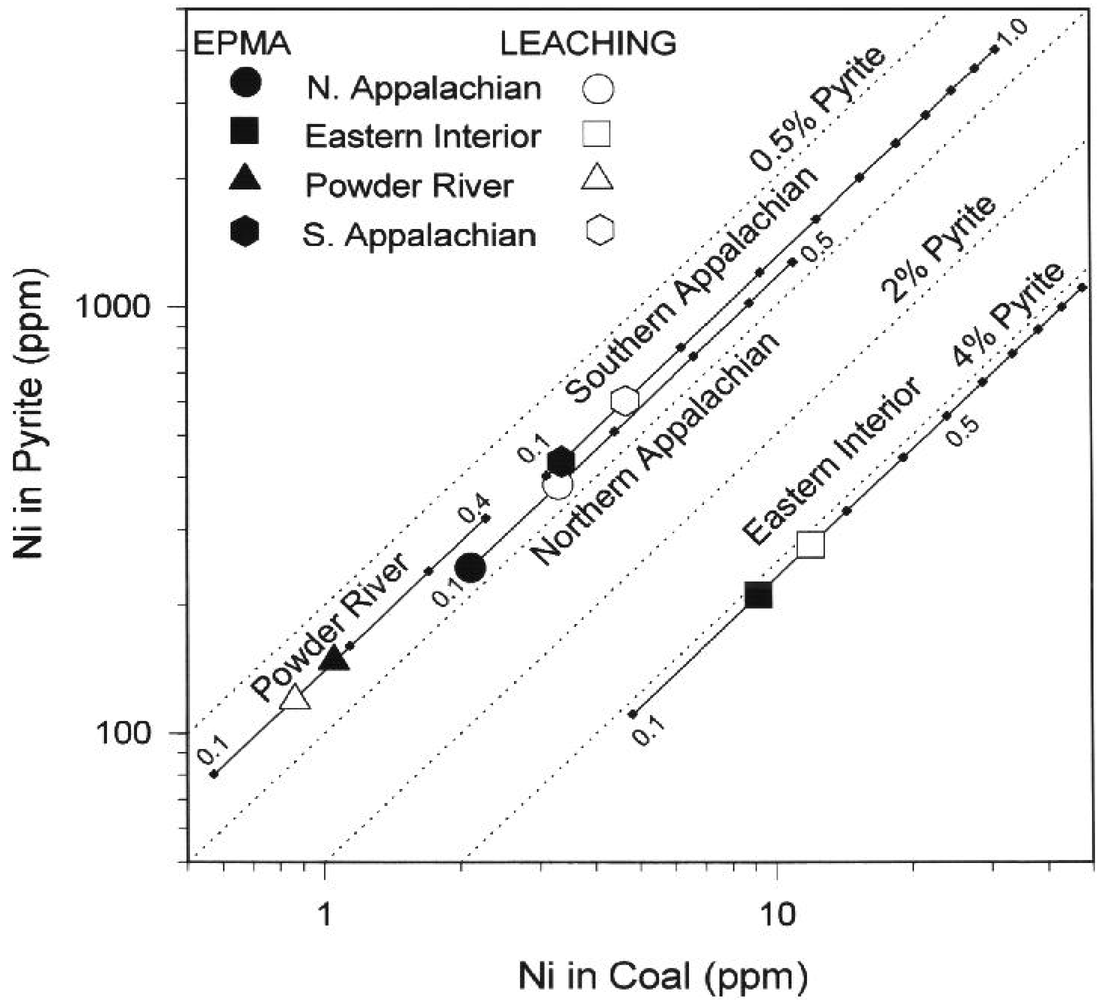


Figure 8

Cr Mass Balance (As-Mined)

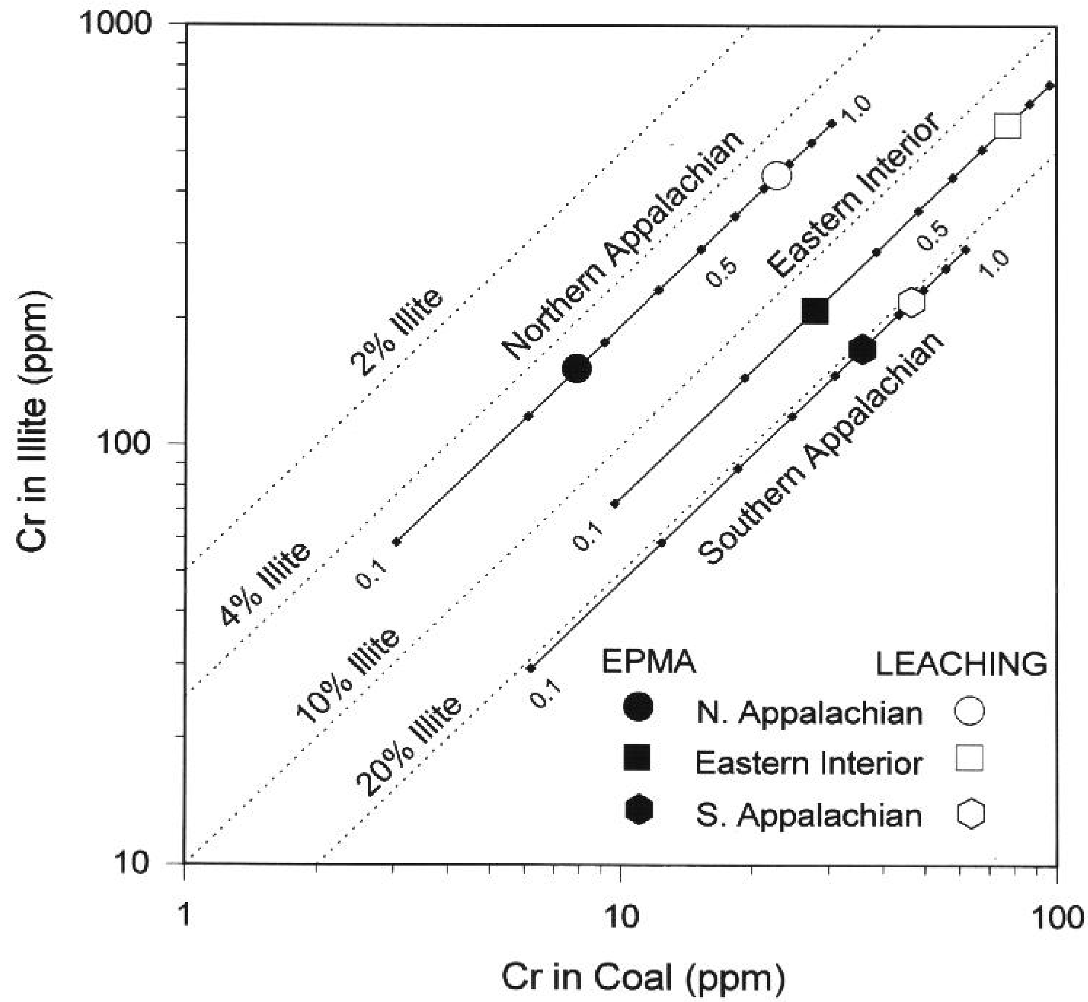


Figure 9

Cr Mass Balance (Fines)

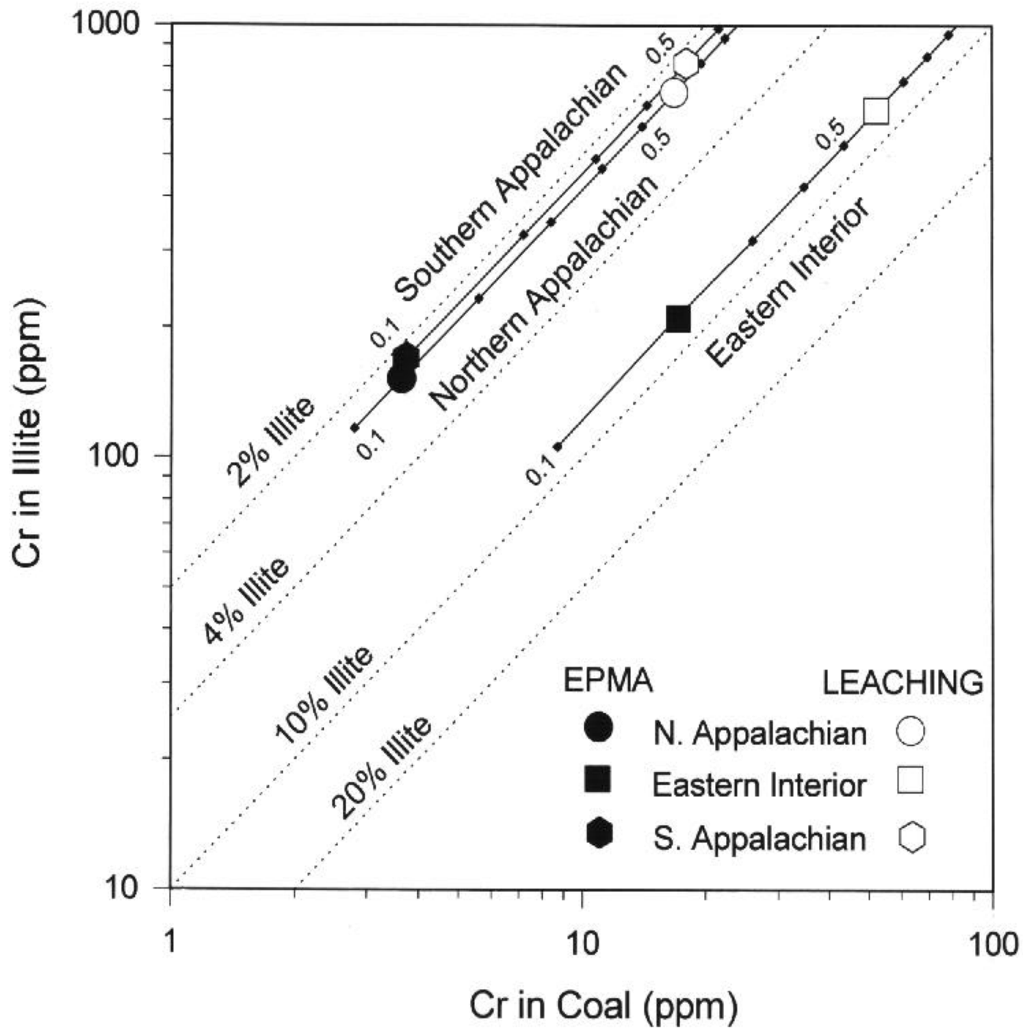


Figure 10

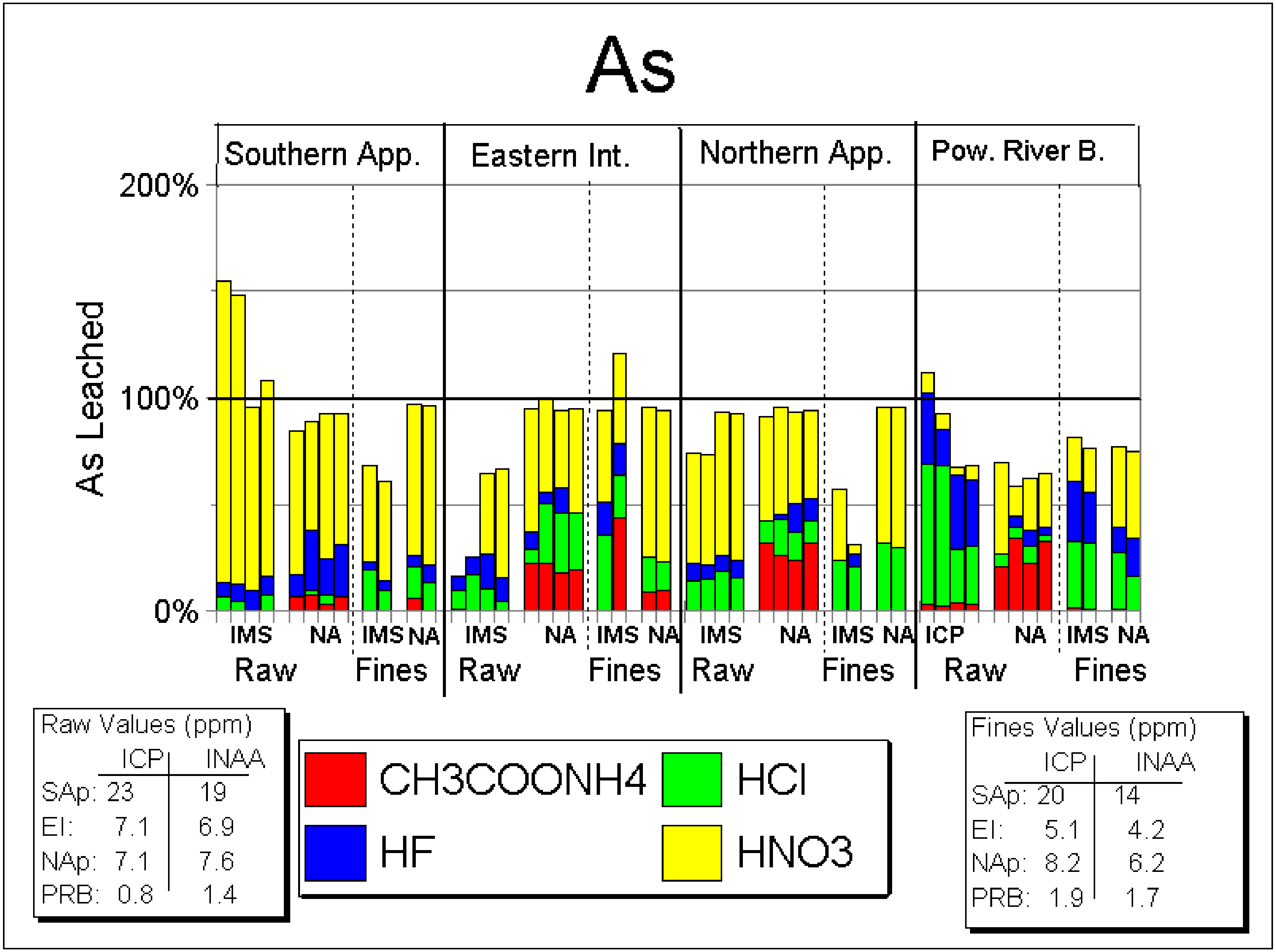

As
Southern App.
Eastern Int.
Northern App.
Pow. River B.
200%
100%
0%
As Leached
IMS
NA
IMS
NA
IMS
NA
IMS
NA
IMS
NA
IMS
NA
ICP
NA
IMS
NA
Raw
Fines
Raw
Fines
Raw
Fines
Raw
Fines
Raw Values (ppm)
ICP INAA
SAp: 23 19
EI: 7.1 6.9
NAp: 7.1 7.6
PRB: 0.8 1.4
CH3COONH4
HCl
HF
HNO3
Fines Values (ppm)
ICP INAA
SAp: 20 14
EI: 5.1 4.2
NAp: 8.2 6.2
PRB: 1.9 1.7

Figure 11

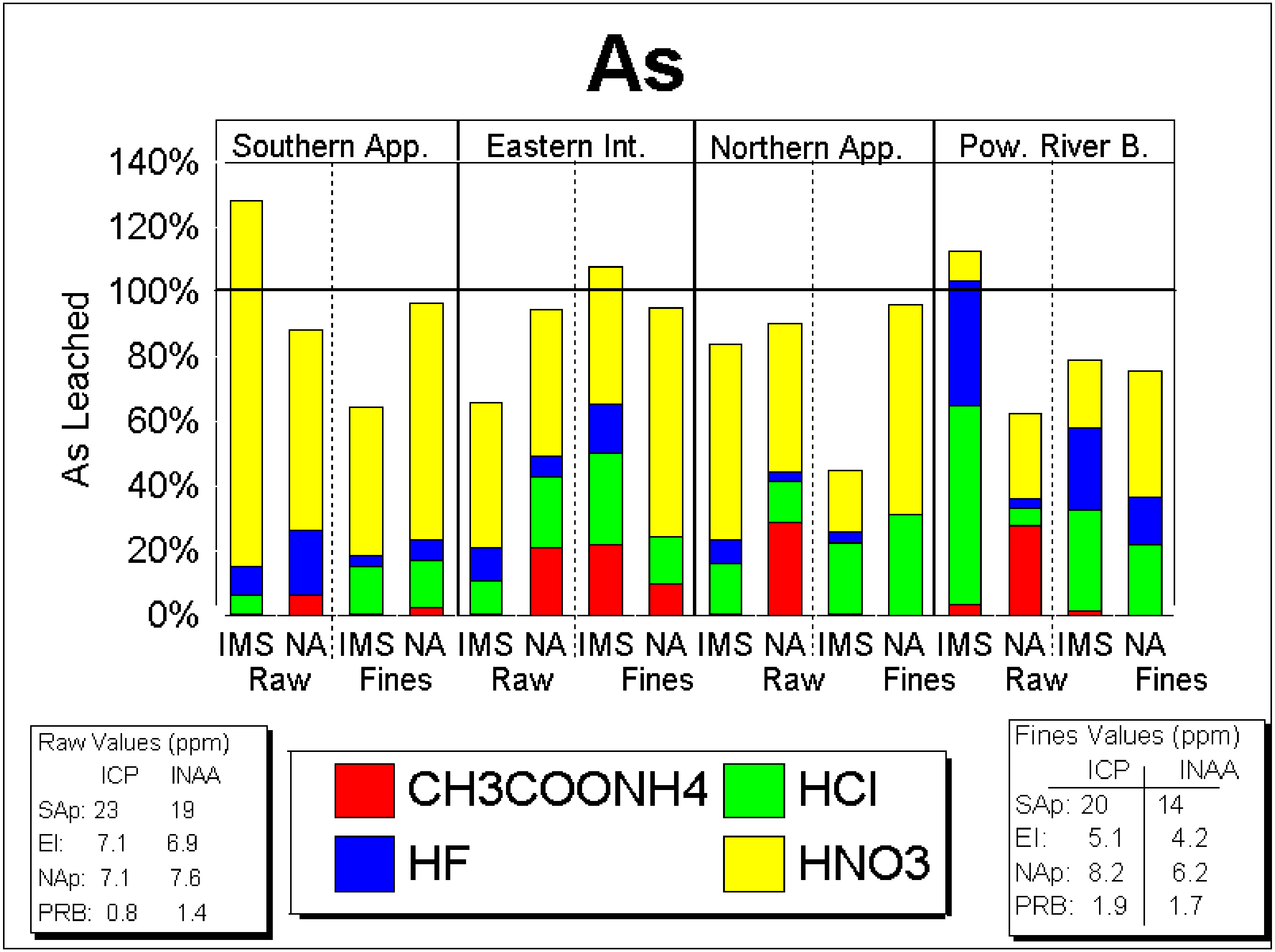

As
As Leached
140%
120%
100%
80%
60%
40%
20%
0%
Southern App.
Eastern Int.
Northern App.
Pow. River B.
IMS NA IMS NA IMS NA IMS NA IMS NA IMS NA IMS NA IMS NA
Raw Fines Raw Fines Raw Fines Raw Fines
Raw Values (ppm)
ICP INAA
SAp: 23 19
EI: 7.1 6.9
NAp: 7.1 7.6
PRB: 0.8 1.4
CH3COONH4
HCl
HF
HNO3
Fines Values (ppm)
ICP INAA
SAp: 20 14
EI: 5.1 4.2
NAp: 8.2 6.2
PRB: 1.9 1.7

Figure 12

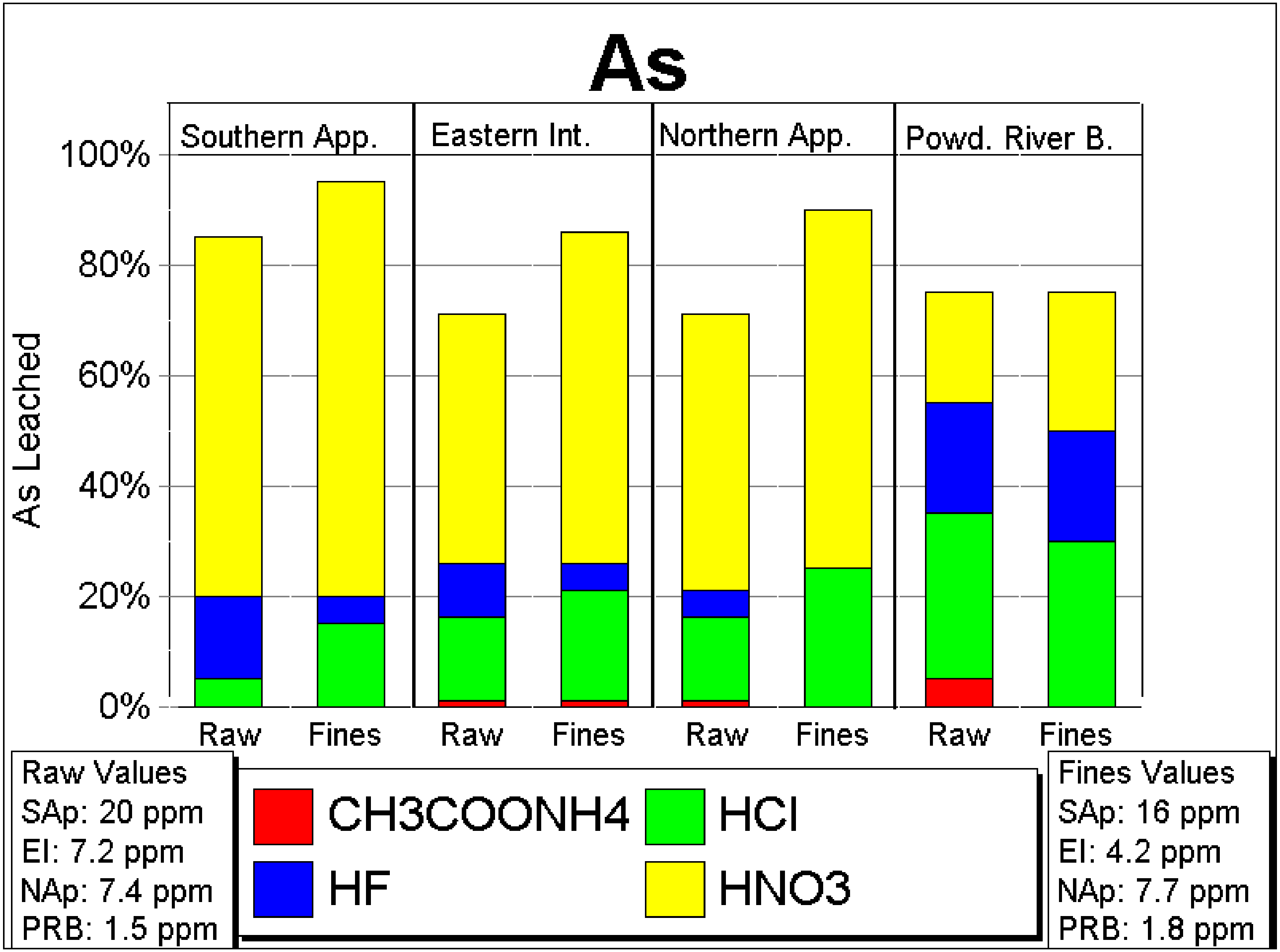
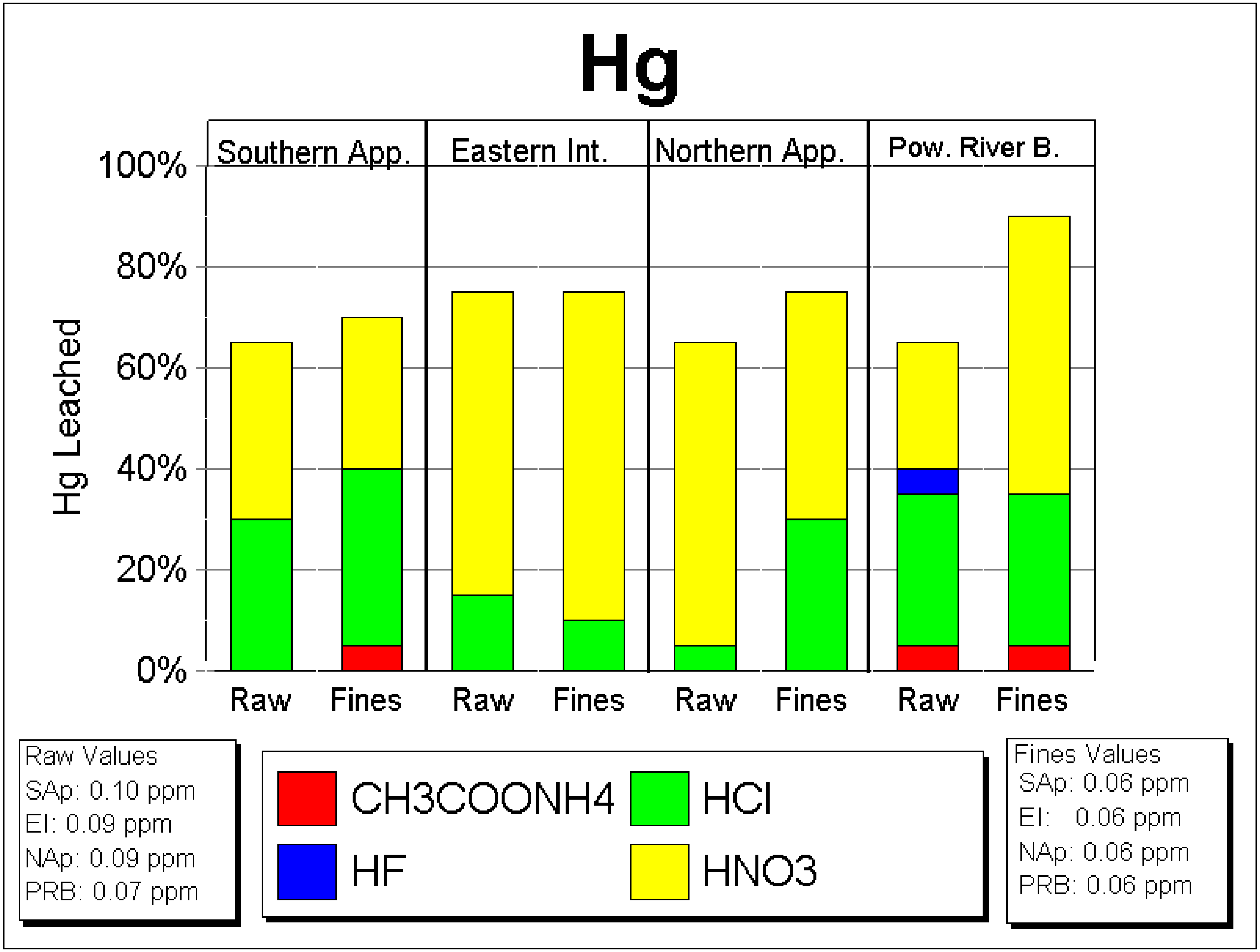

As
Southern App.
Eastern Int.
Northern App.
Powd. River B.
100%
80%
60%
40%
20%
0%
As Leached
Raw
Fines
Raw
Fines
Raw
Fines
Raw
Fines
Raw Values
SAp: 20 ppm
EI: 7.2 ppm
NAp: 7.4 ppm
PRB: 1.5 ppm
CH3COONH4
HCl
HF
HNO3
Fines Values
SAp: 16 ppm
EI: 4.2 ppm
NAp: 7.7 ppm
PRB: 1.8 ppm

Figure 13



Hg
Southern App.
Eastern Int.
Northern App.
Pow. River B.
100%
80%
60%
40%
20%
0%
Hg Leached
Raw
Fines
Raw
Fines
Raw
Fines
Raw
Fines
Raw Values
SAp: 0.10 ppm
EI: 0.09 ppm
NAp: 0.09 ppm
PRB: 0.07 ppm
CH3COONH4
HCl
HF
HNO3
Fines Values
SAp: 0.06 ppm
EI: 0.06 ppm
NAp: 0.06 ppm
PRB: 0.06 ppm

Figure 14

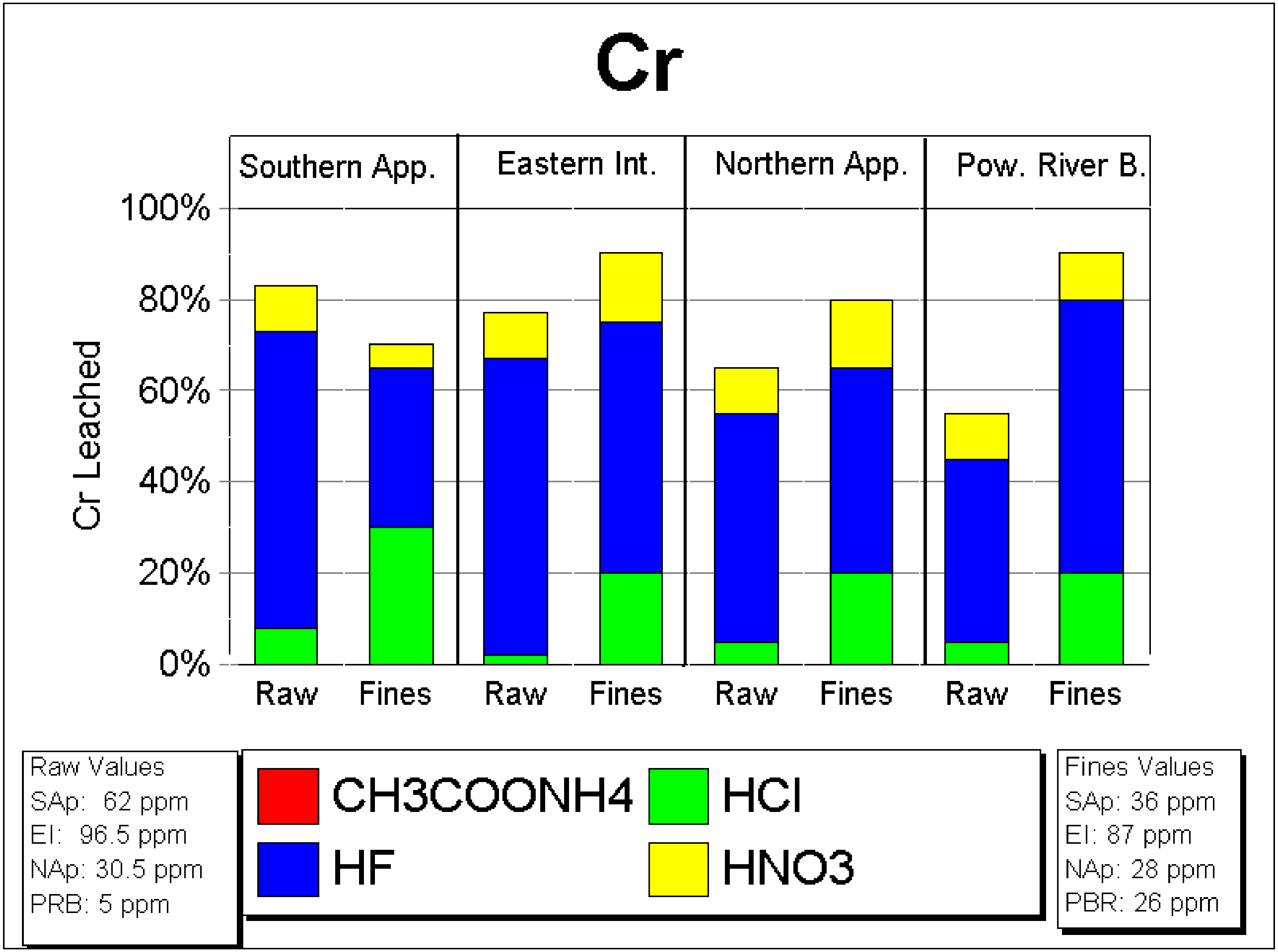

Cr
Southern App.
Eastern Int.
Northern App.
Pow. River B.
100%
80%
60%
40%
20%
0%
Cr Leached
Raw
Fines
Raw
Fines
Raw
Fines
Raw
Fines
Raw Values
SAp: 62 ppm
EI: 96.5 ppm
NAp: 30.5 ppm
PRB: 5 ppm
CH3COONH4
HCl
HF
HNO3
Fines Values
SAp: 36 ppm
EI: 87 ppm
NAp: 28 ppm
PBR: 26 ppm

Figure 15

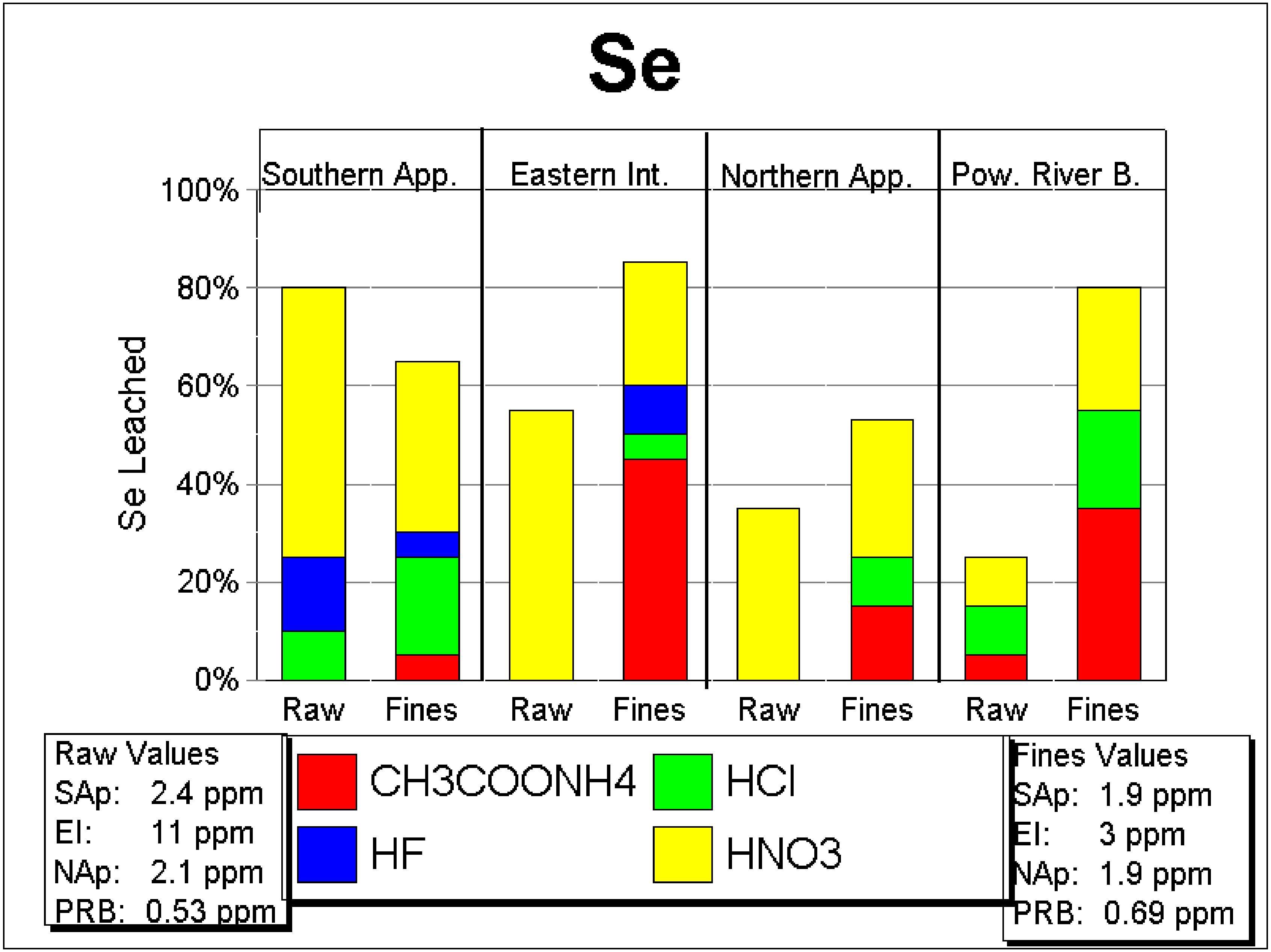

Se
Southern App.
Eastern Int.
Northern App.
Pow. River B.
Se Leached
100%
80%
60%
40%
20%
0%
Raw
Fines
Raw
Fines
Raw
Fines
Raw
Fines
Raw Values
SAp: 2.4 ppm
EI: 11 ppm
NAp: 2.1 ppm
PRB: 0.53 ppm
CH3COONH4
HCl
HF
HNO3
Fines Values
SAp: 1.9 ppm
EI: 3 ppm
NAp: 1.9 ppm
PRB: 0.69 ppm

Figure 16

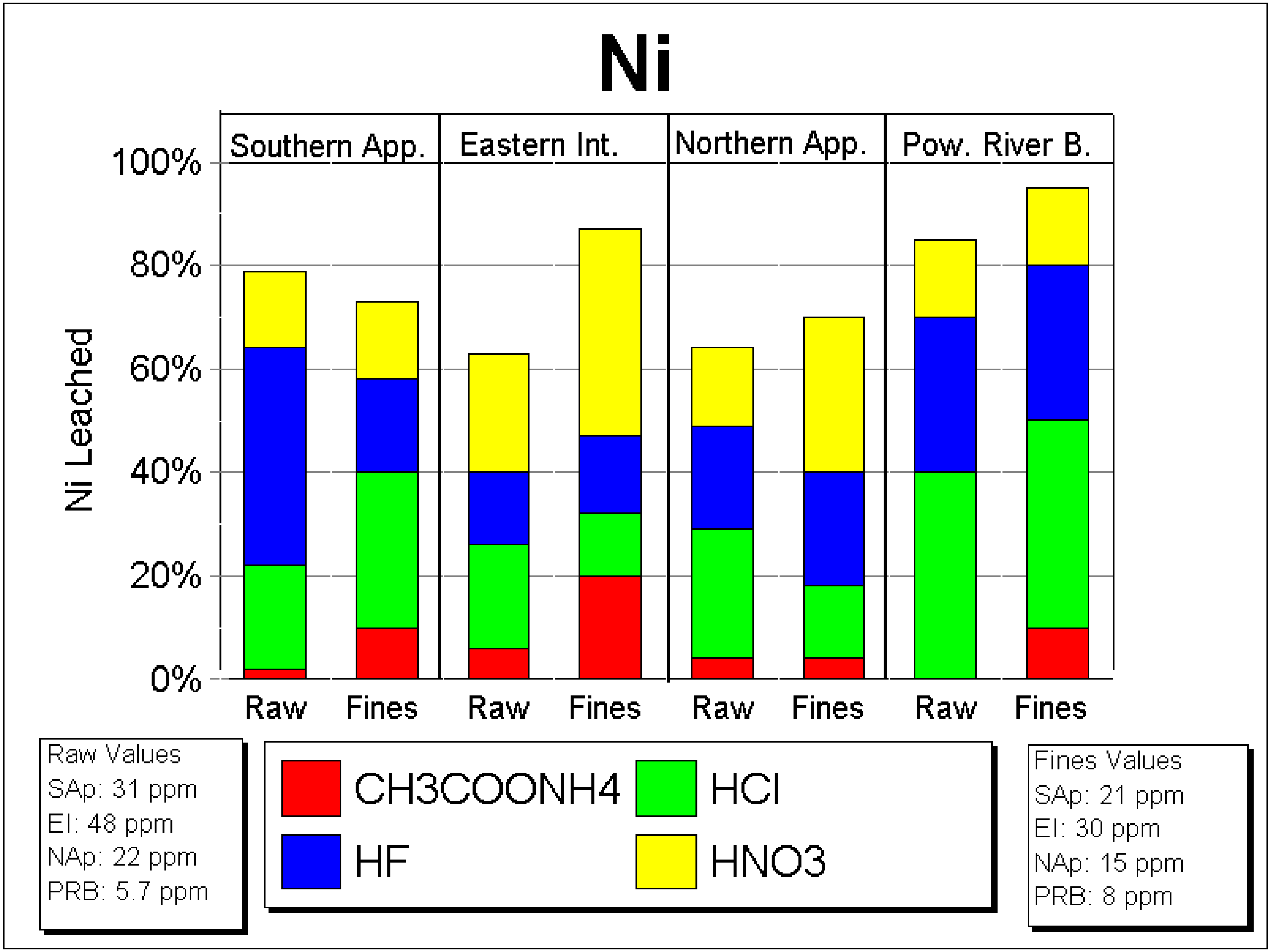

Ni
Southern App.
Eastern Int.
Northern App.
Pow. River B.
100%
80%
60%
40%
20%
0%
Ni Leached
Raw
Fines
Raw
Fines
Raw
Fines
Raw
Fines
Raw Values
SAp: 31 ppm
EI: 48 ppm
NAp: 22 ppm
PRB: 5.7 ppm
CH3COONH4
HCl
HF
HNO3
Fines Values
SAp: 21 ppm
EI: 30 ppm
NAp: 15 ppm
PRB: 8 ppm

Figure 17

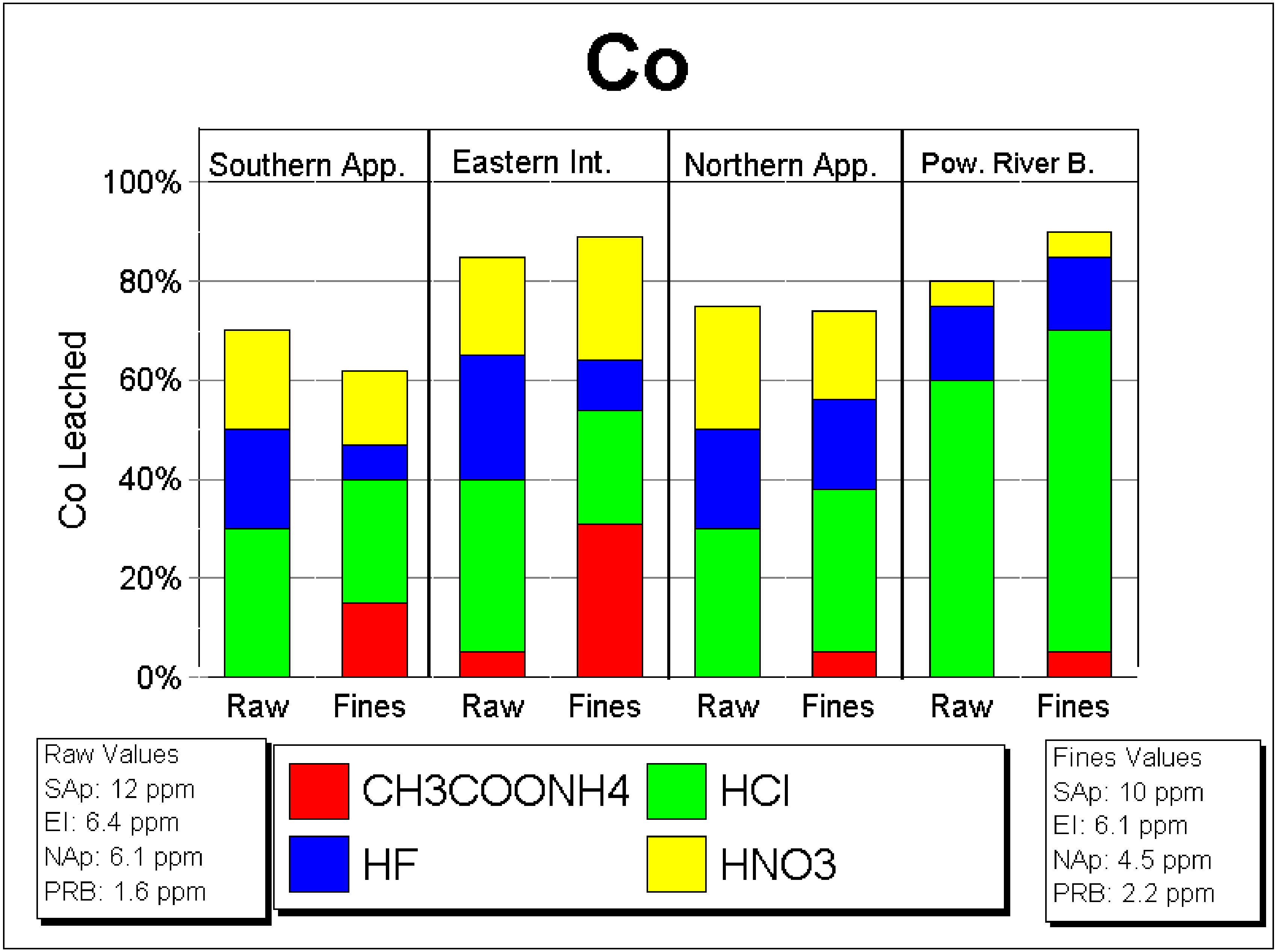

Co
Southern App.
Eastern Int.
Northern App.
Pow. River B.
100%
80%
60%
40%
20%
0%
Co Leached
Raw
Fines
Raw
Fines
Raw
Fines
Raw
Fines
Raw Values
SAp: 12 ppm
EI: 6.4 ppm
NAp: 6.1 ppm
PRB: 1.6 ppm
CH3COONH4
HCl
HF
HNO3
Fines Values
SAp: 10 ppm
EI: 6.1 ppm
NAp: 4.5 ppm
PRB: 2.2 ppm

Figure 18

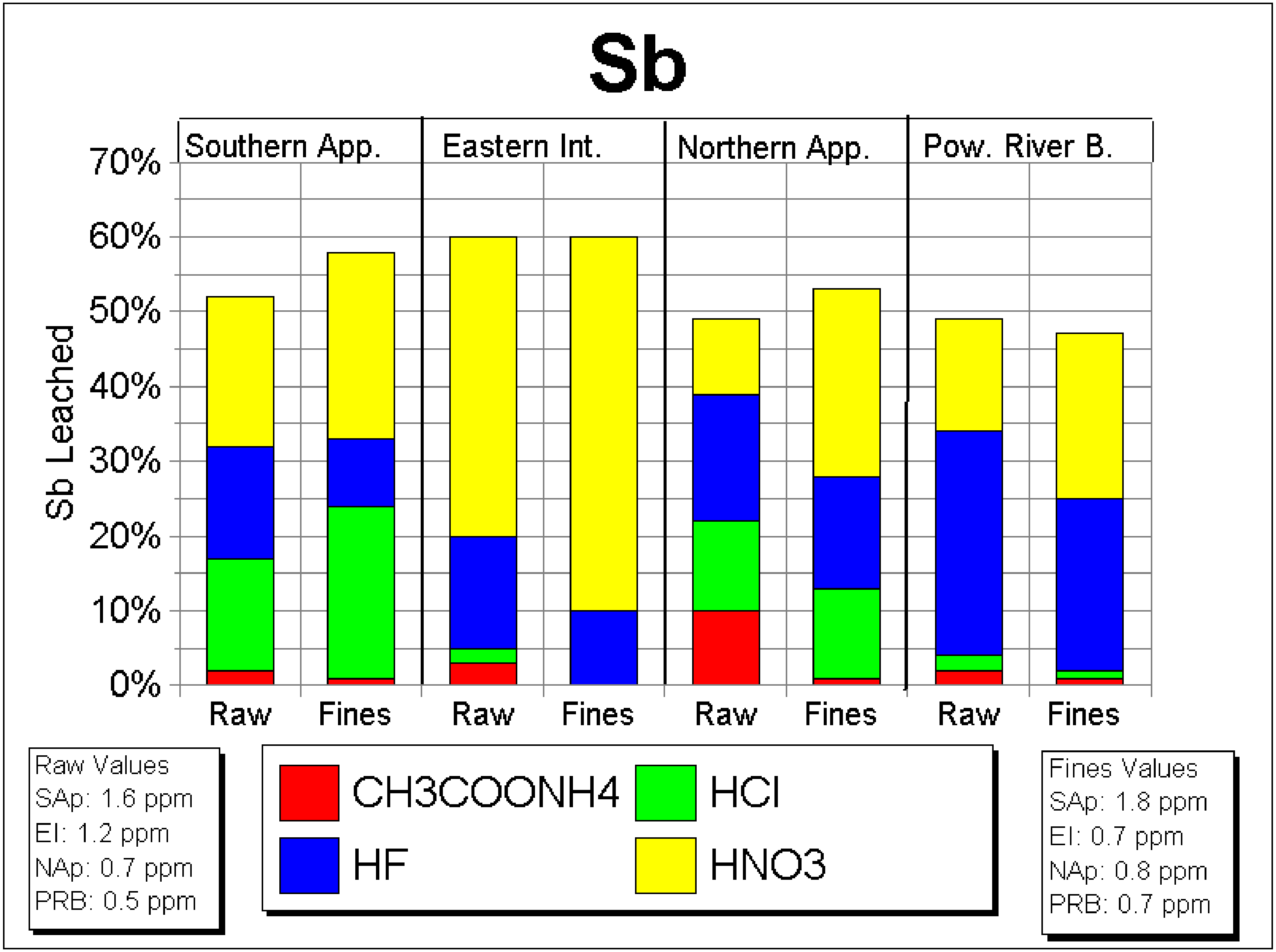
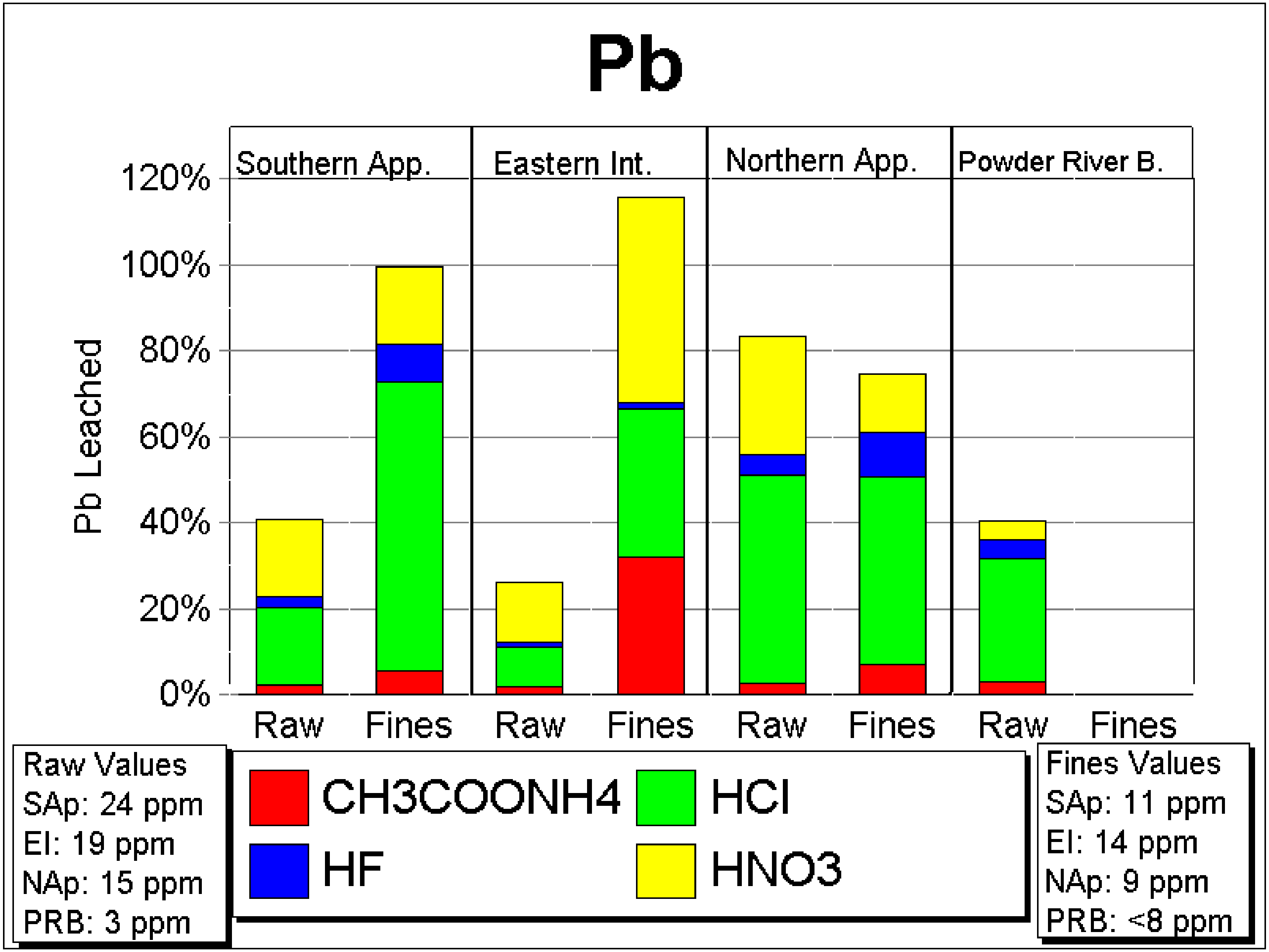

Sb
Southern App.
Eastern Int.
Northern App.
Pow. River B.
70%
60%
50%
40%
30%
20%
10%
0%
Sb Leached
Raw
Fines
Raw
Fines
Raw
Fines
Raw
Fines
Raw Values
SAp: 1.6 ppm
EI: 1.2 ppm
NAp: 0.7 ppm
PRB: 0.5 ppm
CH3COONH4
HCl
HF
HNO3
Fines Values
SAp: 1.8 ppm
EI: 0.7 ppm
NAp: 0.8 ppm
PRB: 0.7 ppm

Figure 19



Pb
Pb Leached
120%
100%
80%
60%
40%
20%
0%
Southern App.
Eastern Int.
Northern App.
Powder River B.
Raw
Fines
Raw
Fines
Raw
Fines
Raw
Fines
Raw Values
SAp: 24 ppm
EI: 19 ppm
NAp: 15 ppm
PRB: 3 ppm
CH3COONH4
HCl
HF
HNO3
Fines Values
SAp: 11 ppm
EI: 14 ppm
NAp: 9 ppm
PRB: <8 ppm

Figure 20

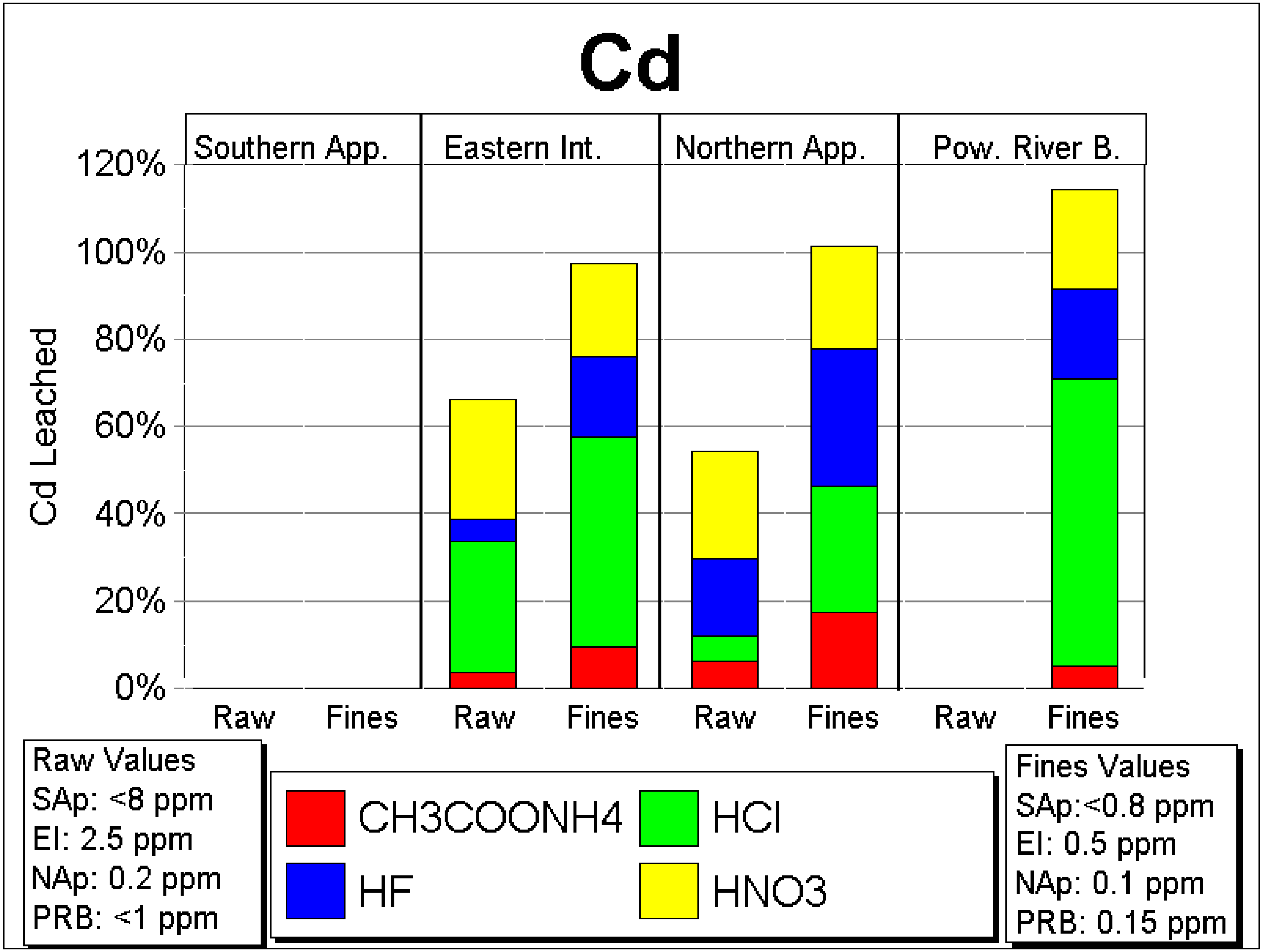

Cd
Southern App.
Eastern Int.
Northern App.
Pow. River B.
Cd Leached
120%
100%
80%
60%
40%
20%
0%
Raw
Fines
Raw
Fines
Raw
Fines
Raw
Fines
Raw Values
SAp: <8 ppm
EI: 2.5 ppm
NAp: 0.2 ppm
PRB: <1 ppm
CH3COONH4
HCl
HF
HNO3
Fines Values
SAp:<0.8 ppm
EI: 0.5 ppm
NAp: 0.1 ppm
PRB: 0.15 ppm

Figure 21

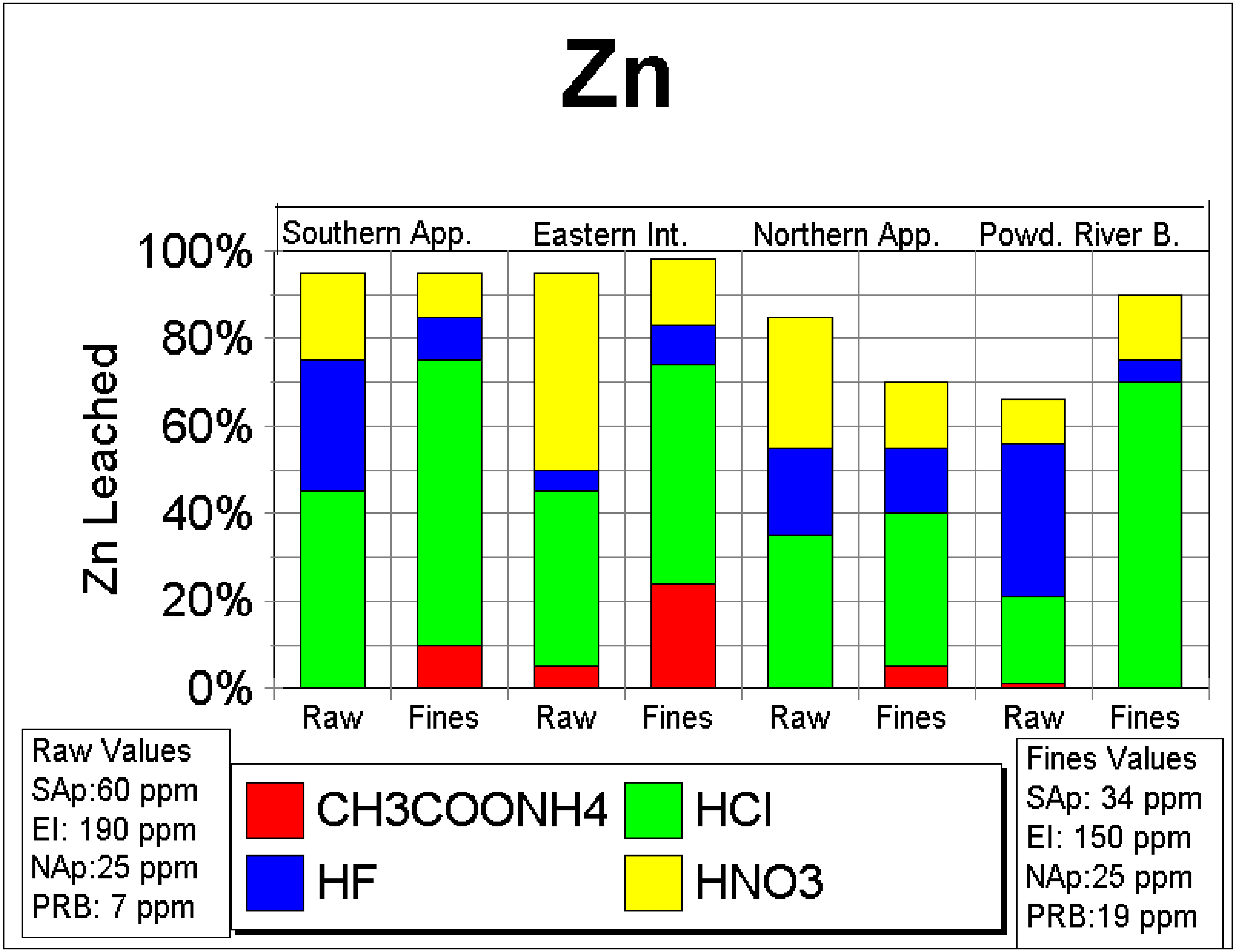
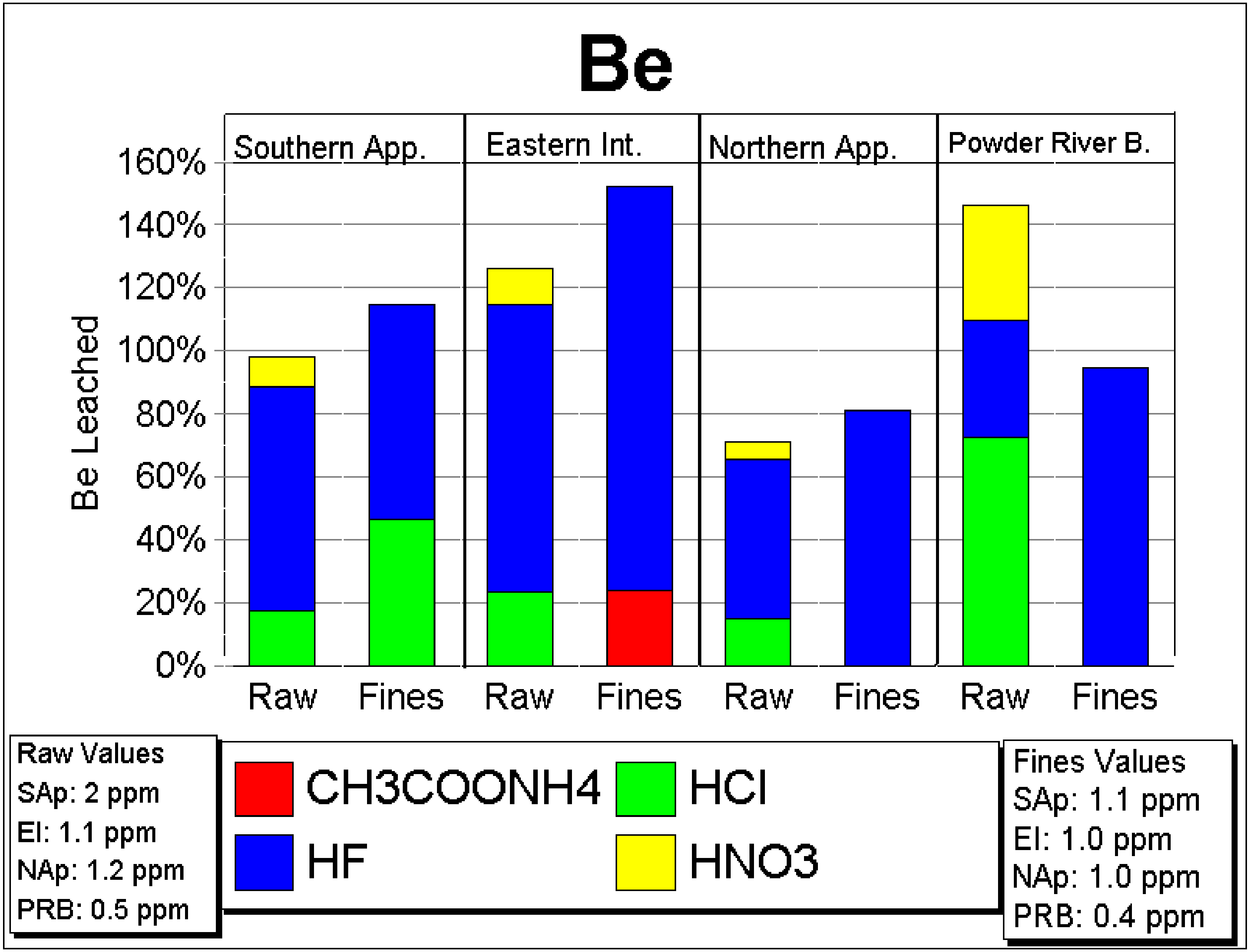
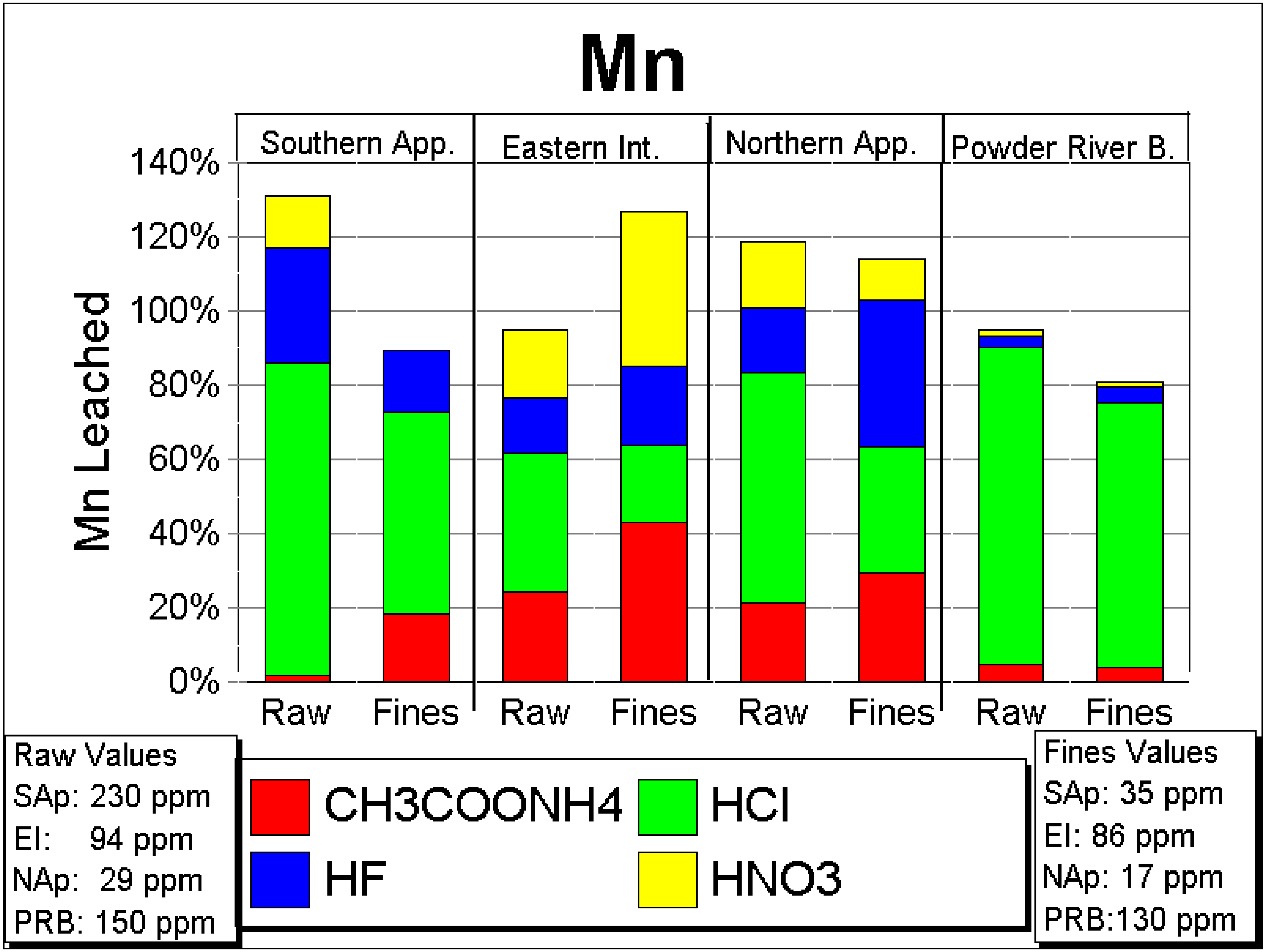

Zn
Zn Leached
100%
80%
60%
40%
20%
0%
Southern App.
Eastern Int.
Northern App.
Powd. River B.
Raw
Fines
Raw
Fines
Raw
Fines
Raw
Fines
Raw Values
SAp:60 ppm
EI: 190 ppm
NAp:25 ppm
PRB: 7 ppm
CH3COONH4
HCl
HF
HNO3
Fines Values
SAp: 34 ppm
EI: 150 ppm
NAp:25 ppm
PRB:19 ppm

Figure 22



Be
Southern App.
Eastern Int.
Northern App.
Powder River B.
Be Leached
160%
140%
120%
100%
80%
60%
40%
20%
0%
Raw
Fines
Raw
Fines
Raw
Fines
Raw
Fines
Raw Values
SAp: 2 ppm
EI: 1.1 ppm
NAp: 1.2 ppm
PRB: 0.5 ppm
CH3COONH4
HCl
HF
HNO3
Fines Values
SAp: 1.1 ppm
EI: 1.0 ppm
NAp: 1.0 ppm
PRB: 0.4 ppm

Figure 23



Mn
Southern App.
Eastern Int.
Northern App.
Powder River B.
Mn Leached
140%
120%
100%
80%
60%
40%
20%
0%
Raw
Fines
Raw
Fines
Raw
Fines
Raw
Fines
Raw Values
SAp: 230 ppm
EI: 94 ppm
NAp: 29 ppm
PRB: 150 ppm
CH3COONH4
HCl
HF
HNO3
Fines Values
SAp: 35 ppm
EI: 86 ppm
NAp: 17 ppm
PRB:130 ppm

Figure 24

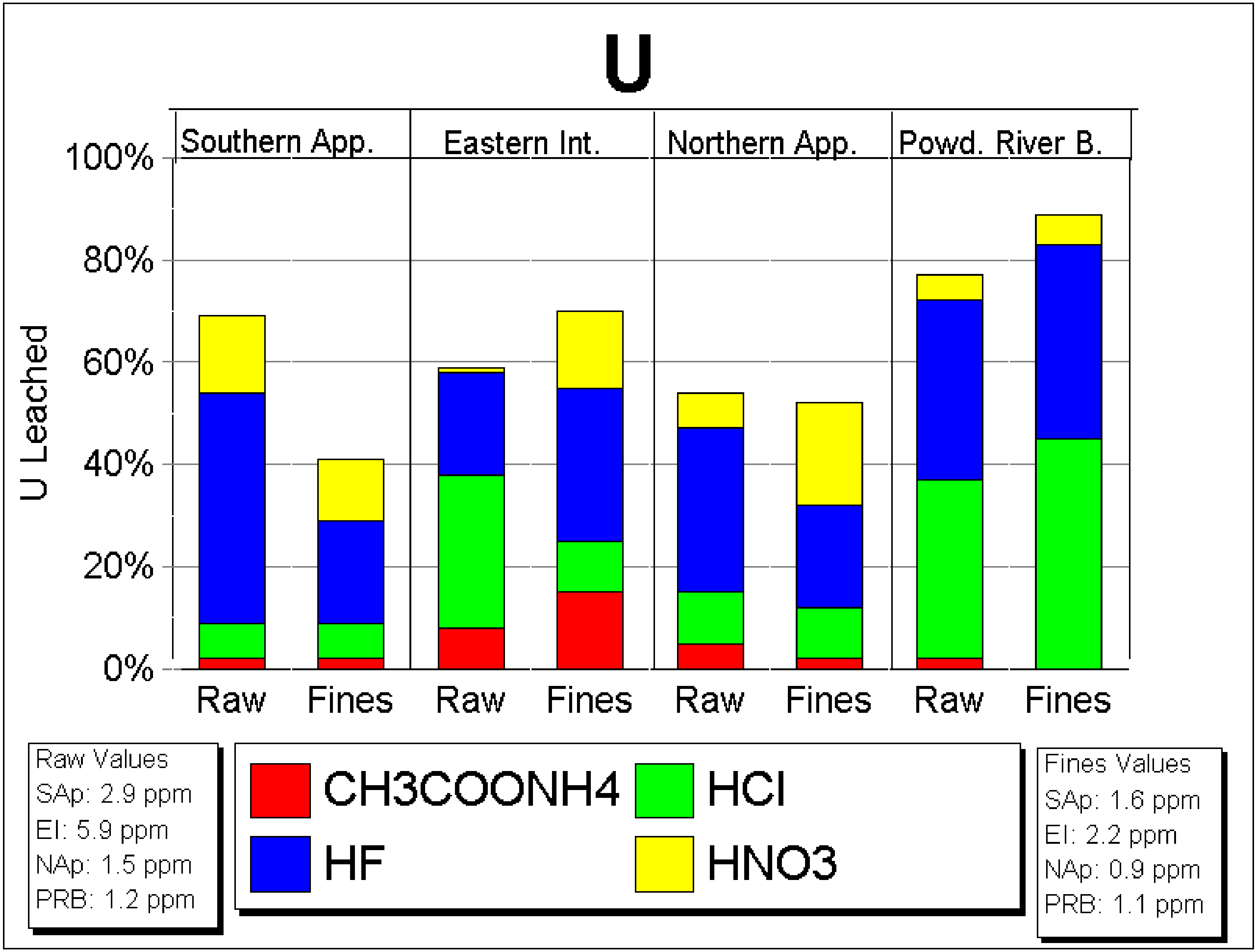
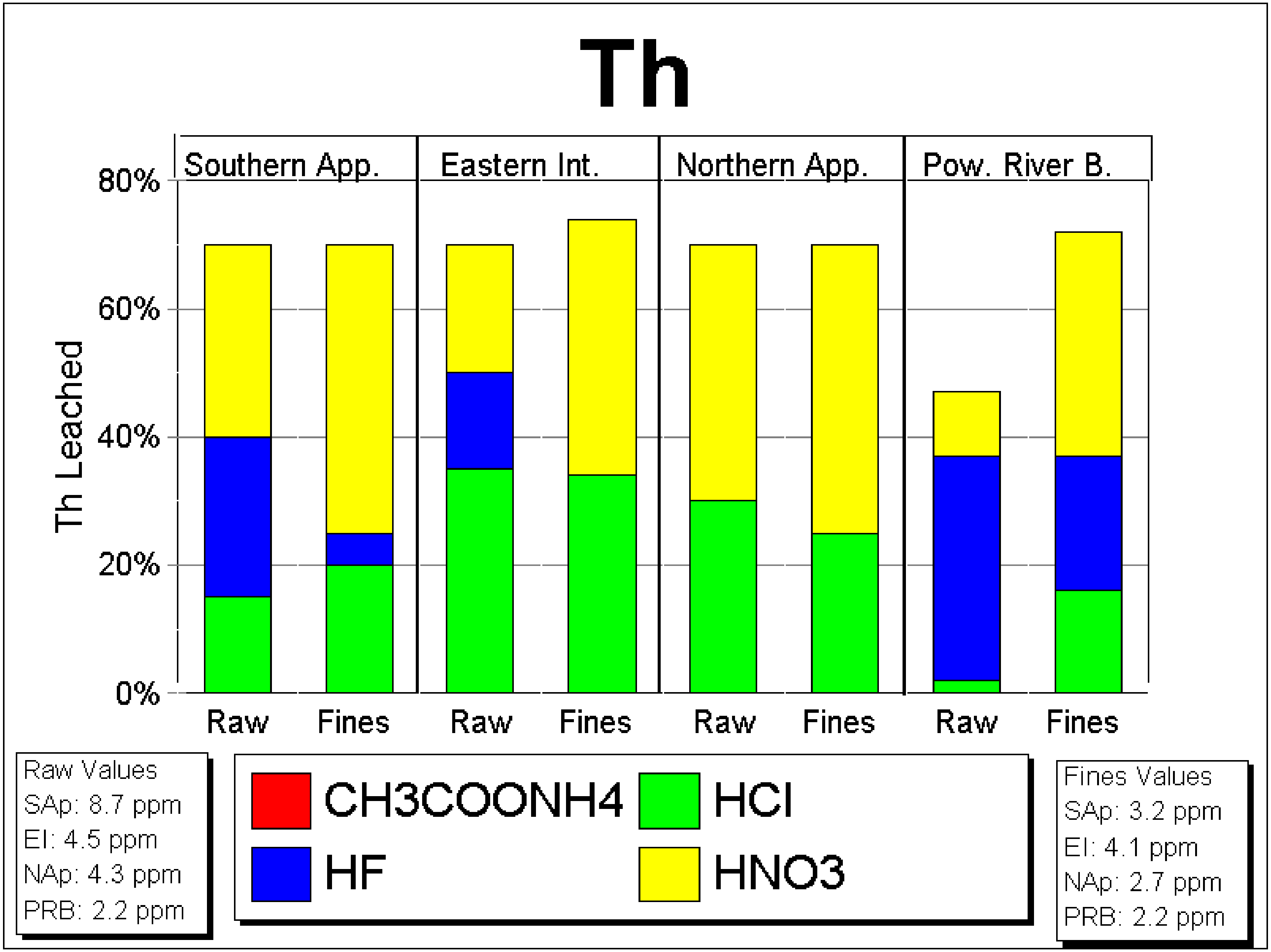
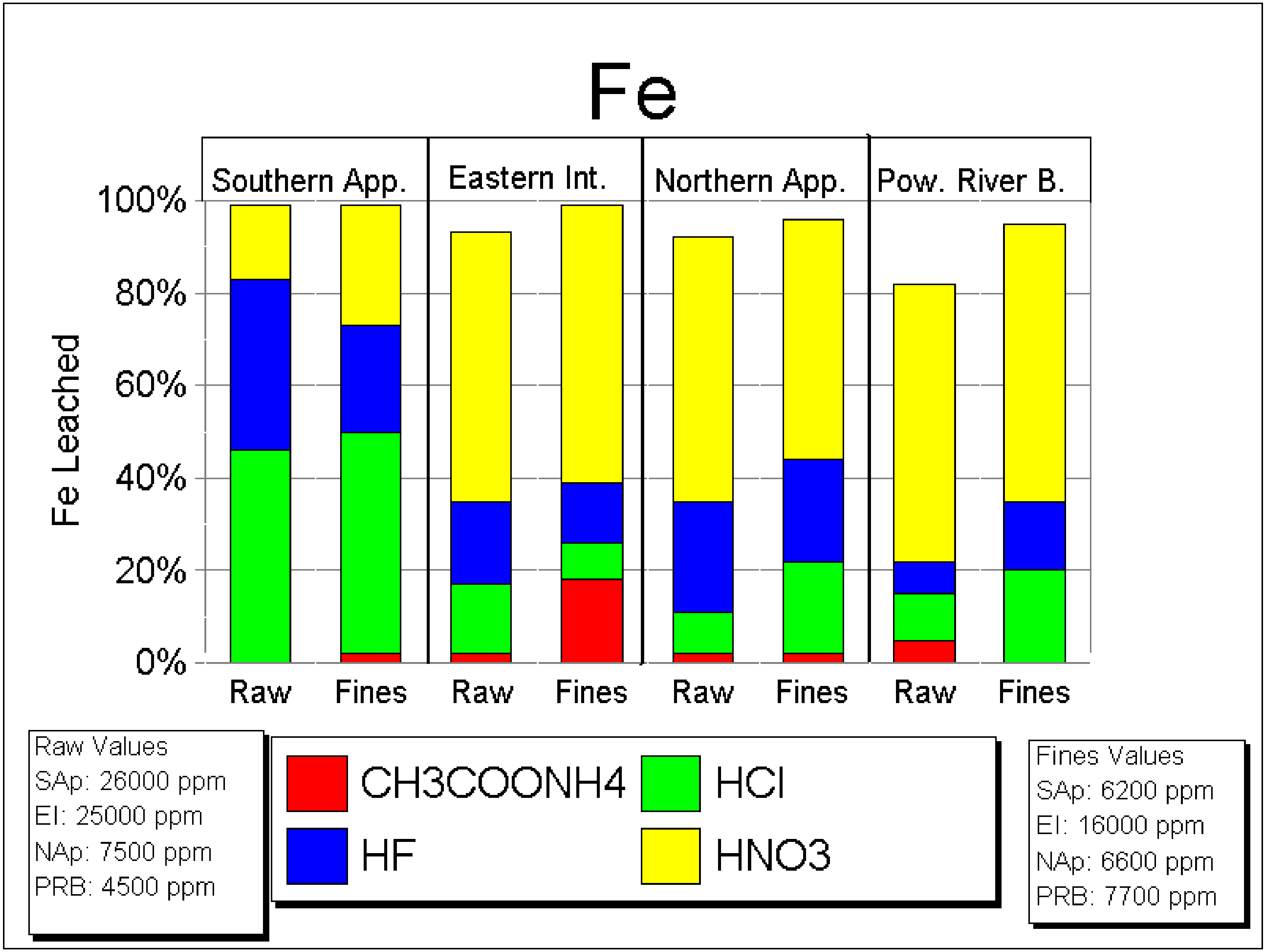

U
U Leached
100%
80%
60%
40%
20%
0%
Southern App.
Eastern Int.
Northern App.
Powd. River B.
Raw
Fines
Raw
Fines
Raw
Fines
Raw
Fines
Raw Values
SAp: 2.9 ppm
EI: 5.9 ppm
NAp: 1.5 ppm
PRB: 1.2 ppm
CH3COONH4
HCl
HF
HNO3
Fines Values
SAp: 1.6 ppm
EI: 2.2 ppm
NAp: 0.9 ppm
PRB: 1.1 ppm

Figure 25



Th
Southern App.
Eastern Int.
Northern App.
Pow. River B.
80%
60%
40%
20%
0%
Th Leached
Raw
Fines
Raw
Fines
Raw
Fines
Raw
Fines
Raw Values
SAp: 8.7 ppm
EI: 4.5 ppm
NAp: 4.3 ppm
PRB: 2.2 ppm
CH3COONH4
HCl
HF
HNO3
Fines Values
SAp: 3.2 ppm
EI: 4.1 ppm
NAp: 2.7 ppm
PRB: 2.2 ppm

Figure 26


Fe
Southern App.
Eastern Int.
Northern App.
Pow. River B.
100%
80%
60%
40%
20%
0%
Fe Leached
Raw
Fines
Raw
Fines
Raw
Fines
Raw
Fines
Raw Values
SAp: 26000 ppm
EI: 25000 ppm
NAp: 7500 ppm
PRB: 4500 ppm
CH3COONH4
HCl
HF
HNO3
Fines Values
SAp: 6200 ppm
EI: 16000 ppm
NAp: 6600 ppm
PRB: 7700 ppm

Appendix 1

Mineralogy of As-mined and Cleaned and Natural Fines Based on SEM Analysis

A. Major Phases

Southern Appalachian

As-Mined	Cleaned Fines
Illite	Illite
Kaolinite	Kaolinite
Quartz	Quartz
Pyrite	Pyrite
Calcite	Calcite
Iron Oxide	Iron Oxide

Eastern Interior

As-Mined	Cleaned Fines
Illite	Illite
Kaolinite	Kaolinite
Quartz	Quartz
Pyrite	Pyrite
Calcite	Calcite
Apatite	-------

Northern Appalachian

As-Mined	Cleaned Fines
Illite	Illite
Kaolinite	Kaolinite
Quartz	Quartz
Calcite	Calcite
Pyrite	Pyrite
-------	-------

Pwder River Basin

As-Mined	Natural Fines
Kaolinite	Kaolinite
Quartz	Quartz
Calcite	Calcite
Pyrite	Pyrite
Barite	Barite
Iron Oxide	---------

B. Minor or Trace Phases

Southern Appalachian

As-Mined	Cleaned Fines
Barite	Barite
Apatite	Apatite
Chalcopyrite	Chalcopyrite
Sphalerite	Sphalerite
Monazite	Monazite
Zircon	Titanium dioxide
Zirconium oxide	Siderite
Aluminum phosphate	
Xenotime (Yttrium phosphate)	
Lead Selenide	

Eastern Interior

As-Mined	Cleaned Fines
Barite	Apatite
Titanium oxide	Barite
Sphalerite	Titanium dioxide
	Sphalerite
	Iron oxide

Northern Appalachian

As-Mined	Cleaned Fines
Titanium dioxide	Titanium dioxide
Monazite (REE phosphate)	Monazite
Zircon	Zircon
Barite	

Powder River Basin

As-Mined	Natural Fines
Apatite	Iron oxide
Cassiterite (Tin Oxide)	Apatite
Titanium dioxide	Cassiterite
Monazite	Sphalerite
Calcium sulfate	

Appendix 2

Quantitative Microprobe Analyses

A. Pyrite Analyses

							POWDER RIVER AS-MINED						
Date	Anal#	Se	Cu	Ni	As	Zn	Cd	Co	Fe	S	Total	Grain	Size (microns)/Form
1/3/97	37	dl	0.01	0.00	0.03	dl	dl	nd	45.33	49.73	95.11	py1.1	30 framboidal
	38	dl	dl	dl	dl	dl	dl	nd	45.41	52.01	97.42	py1.2	
	41	dl	dl	0.01	0.01	dl	dl	nd	45.63	53.32	98.97	py4.1	20x30
	42	dl	dl	dl	0.01	dl	dl	nd	45.46	54.08	99.54	py4.2	subhedral
	43	dl	dl	0.00	0.02	dl	dl	nd	46.2	52.09	98.33	py5.1	100x120
	44	dl	dl	dl	0.01	0.01	dl	nd	46.7	49.37	96.05	py5.2	subhedral
	45	dl	dl	0.01	0.01	dl	dl	nd	45.7	53.81	99.53	py5.3	
	46	dl	dl	dl	dl	dl	dl	nd	46.5	52.51	99.06	py6.1	20x200 cleat
	48	dl	0.01	dl	0.01	dl	dl	nd	45.1	50.87	96.03	py7.1	30x70
	49	dl	dl	dl	dl	dl	dl	nd	46.4	52.98	99.36	py7.2	subhedral composite
	50	dl	0.01	dl	dl	dl	dl	nd	44.81	52.76	97.57	py8.1	10x300
	51	dl	0.01	dl	dl	dl	dl	nd	45.13	51.68	96.82	py8.2	cleat
	52	dl	0.01	0.01	dl	dl	dl	nd	45.22	51.65	96.89	py8.3	
	53	dl	dl	dl	0.02	dl	dl	nd	45.45	50.00	95.47	py9.1	large cleat
	54	dl	dl	dl	dl	dl	dl	nd	46.25	51.55	97.81	py9.2	
	55	dl	dl	dl	0.01	dl	dl	nd	46.02	51.24	97.27	py9.3	
7/4/96	9	0.03	dl	dl	**dl**	0.01	dl	**dl**	46.18	54.35	100.64	#16	large cleat
	10	0.01	dl	0.01	**0.11**	0.01	dl	**dl**	45.53	50.66	96.36	#17	
	14	dl	dl	dl	**dl**	0.02	dl	**dl**	44.99	53.31	98.37	#21	100 subhedral
	17	dl	dl	dl	**0.07**	dl	dl	**dl**	45.23	53.98	99.33	#24	100 subhedral
	18	dl	0.01	dl	**0.06**	dl	dl	**dl**	44.36	51.14	95.62	#25	
	19	dl	dl	0.01	**0.06**	0.01	dl	**dl**	44.48	54.31	98.93	#26	

POWDER RIVER FINES													
Date	Anal #	Se	Cu	Ni	As	Zn	Cd	Co	Fe	S	Total	Grain	Size (microns)/Form
1/3/97	108	0.02	dl	dl	0.00	dl	dl	nd	46.95	51.92	98.88	Py1.1	40x70 subhedral
	109	dl	dl	dl	dl	dl	dl	nd	46.57	51.98	98.55	Py1.2	
	110	dl	dl	dl	dl	dl	dl	nd	46.76	52.19	98.95	Py1.3	
	111	dl	dl	0.00	0.01	dl	dl	nd	46.73	52.05	98.79	Py2.1	25x70 subhedral
	115	dl	dl	dl	dl	dl	dl	nd	46.47	52.59	99.06	Py3.1	10x60 cleat
	116	dl	dl	dl	dl	0.01	dl	nd	46.08	51.87	97.97	Py3.2	
	117	dl	dl	dl	0.01	dl	dl	nd	46.17	51.99	98.18	Py4.1	40x70 subhedral
	118	dl	dl	dl	0.01	dl	dl	nd	46.30	52.78	99.09	Py4.2	
	119	dl	0.01	0.01	0.01	dl	dl	nd	46.58	51.88	98.49	Py5.1	30x40 subhedral
	120	dl	dl	dl	0.01	dl	dl	nd	46.00	51.65	97.67	Py5.2	
	121	dl	0.01	dl	dl	0.01	dl	nd	46.74	52.31	99.07	Py6.1	20x40 subhedral
	122	dl	dl	0.00	dl	dl	dl	nd	46.93	52.62	99.56	Py6.2	
	123	dl	dl	0.01	dl	dl	dl	nd	46.88	52.85	99.73	Py7.1	20x120 cleat
	124	dl	dl	dl	0.01	dl	dl	nd	46.59	51.55	98.15	Py7.2	
	125	0.01	dl	dl	dl	dl	dl	nd	46.40	52.73	99.14	Py7.3	
	126	0.00	dl	dl	0.01	dl	dl	nd	46.27	52.29	98.58	Py8.1	50x70 subhedral
	127	dl	dl	dl	dl	dl	dl	nd	46.17	52.45	98.62	Py8.2	
	128	0.02	0.03	dl	0.02	dl	dl	nd	46.74	52.62	99.42	Py9.1	20x40
	129	0.02	0.02	dl	0.01	0.01	dl	nd	46.44	52.10	98.59	Py9.2	subhedral/euhedral
	130	dl	dl	dl	0.01	dl	dl	nd	46.86	52.48	99.35	Py10.1	20x80 cleat
	131	0.00	dl	dl	dl	0.02	dl		46.72	52.39	99.14	Py10.2	
	133	dl	0.02	0.05	0.12	dl	dl	nd	46.59	52.21	98.98	Py12.1	15 framboidal

POWDER RIVER FINES- Continued													
Date	Anal #	Se	Cu	Ni	As	Zn	Cd	Co	Fe	S	Total	Grain	Size (microns)/Form
1/3/97	134	dl	dl	dl	dl	dl	dl	nd	46.10	51.89	98.00	Py13.1	15 euhedral
	135	0.12	0.05	0.29	0.27	dl	dl	nd	43.65	49.60	93.97	Py14.1	20 framboidal

*dl= below detection limit of 100 ± 100 ppm, except arsenic values listed in boldface (dl=500 ± 500 ppm).
nd=not determined

Values for Co include a 0.06 wt. % empirical correction factor subtracted from measured values.

						EASTERN INTERIOR AS-MINED							
Date	Anal #	Se	Cu	Ni	As	Zn	Cd	Co	Fe	S	Total	Grain	Size (microns)/form
12/23/96	37	dl	dl	dl	0.02	0.01	dl	nd	47.42	51.68	99.14	Py 1.1	150 composite
	38	dl	dl	0.00	0.03	dl	dl	nd	46.38	50.43	96.85	Py 1.2	
	39	0.01	0.01	dl	dl	0.00	dl	nd	47.38	51.74	99.14	Py 2.1	150 round
	40	dl	0.01	dl	dl	dl	dl	nd	47.60	52.08	99.69	Py 2.2	
	41	dl	dl	dl	0.01	0.02	dl	nd	46.16	50.65	96.85	Py 3.1	80x100 subhedral
	42	dl	dl	dl	0.01	dl	dl	nd	46.80	51.67	98.48	Py 3.2	
	45	dl	dl	dl	0.02	dl	dl	nd	45.93	50.08	96.02	Py 5.1	60x120 subhedral
	46	dl	dl	dl	dl	dl	dl	nd	46.69	51.01	97.70	Py 5.2	
	47	dl	0.01	0.01	dl	dl	dl	nd	45.75	50.23	96.00	Py 6.1	40x70 subhedral
	48	dl	0.05	0.03	0.02	0.01	dl	nd	45.65	50.67	96.43	Py 6.2	
	49	dl	dl	0.01	dl	0.01	dl	nd	46.64	50.84	97.49	Py 6.3	
	50	dl	dl	0.01	0.00	0.01	dl	nd	46.80	51.37	98.20	Py 7.1	100x120 subhedral
	52	dl	dl	0.02	0.02	dl	dl	nd	46.45	51.43	97.91	Py 7.3	
	53	dl	0.02	0.02	0.01	dl	dl	nd	47.00	51.62	98.67	Py 8.1	100x100 subhedral
	54	dl	dl	0.02	0.01	0.01	dl	nd	46.52	50.37	96.93	Py 8.2	
	55	dl	dl	0.03	dl	dl	dl	nd	47.08	50.72	97.83	Py 8.3	
	56	dl	dl	0.02	0.01	0.00	dl	nd	46.68	50.92	97.64	Py 9.1	30x40 subh./euhed.
	57	dl	0.01	dl	0.01	dl	dl	nd	46.97	50.23	97.22	Py 10.1	90x120 irregular
	58	dl	dl	0.01	dl	dl	dl	nd	47.17	51.74	98.92	Py 10.2	

	EASTERN INTERIOR AS-MINED (Continued)												
Date	Anal #	Se	Cu	Ni	As	Zn	Cd	Co	Fe	S	Total	Grain	Size (microns)/Form
12/23	59	dl	dl	dl	dl	dl	dl	nd	46.74	51.41	98.15	Py 11.1	20 framboidal
	60	dl	dl	dl	dl	0.01	dl	nd	46.56	51.09	97.66	Py 12.1	20 framboidal
	62	dl	0.01	dl	0.03	dl	dl	nd	46.98	51.07	98.09	Py 13.2	70x70 composite
10/31/96	8		0.01	0.02	**0.07**	0.02	dl	nd	45.23	51.68	97.03	EI1.1	5 framboidal
	10		0.01	0.02	**0.08**	dl	dl	nd	44.99	51.14	96.23	EI2.2	20 framboidal
	11		0.03	0.08	**0.14**	0.00	dl	nd	44.99	51.52	96.77	EI3.1	
	13		0.02	0.15	**0.14**	0.01	dl	nd	45.26	50.65	96.22	EI4.2	40 euhedral
	14		dl	0.02	**0.08**	dl	dl	nd	44.32	50.40	94.81	EI4.3	adj. 20 euhedral
	18		dl	0.01	**0.10**	dl	dl	nd	46.65	51.94	98.72	EI6.1	170 irregular
	19		dl	dl	**0.09**	dl	dl	nd	46.05	51.03	97.17	EI6.2	
	20		dl	dl	**0.09**	dl	dl	nd	45.49	50.70	96.28	EI6.3	
	21		dl	0.00	**0.10**	dl	dl	nd	45.31	50.44	95.85	EI6.4	
	23		0.02	0.04	**0.09**	dl	dl	nd	45.89	51.18	97.22	EI8.1	15 framboidal
	24		dl	dl	**0.09**	dl	dl	nd	46.04	51.55	97.68	EI9.1	10 framboidal
	26		0.04	0.01	**0.13**	0.01	dl	nd	45.79	51.75	97.72	EI11.1	10 framboidal
	28		dl	0.08	**0.14**	0.03	dl	nd	45.52	49.10	94.88	EI12.2	40x50 irregular

	EASTERN INTERIOR AS-MINED (Continued)												
Date	Anal #	Se	Cu	Ni	As	Zn	Cd	Co	Fe	S	Tot	Grain	Size (microns)/Form
10/31/96	30		0.03	0.02	**0.14**	dl	dl	nd	45.55	51.66	97.39	EI13.1	20 framboidal
	80		dl	dl	**0.18**	dl	dl	nd	46.26	51.71	98.15	EI14.1	15x40 cleat
	81		dl	dl	**0.08**	dl	dl	nd	46.11	51.58	97.77	EI14.2	
	82		0.02	dl	**0.11**	dl	dl	nd	45.97	51.65	97.75	EI15.1	17 framboidal
	83		dl	0.01	**0.12**	dl	dl	nd	45.22	49.26	94.60	EI16.1	120x200 irreg.
	84		dl	dl	**0.13**	dl	dl	nd	45.14	50.20	95.48	EI16.2	composite
	85		dl	0.01	**0.13**	dl	dl	nd	45.34	51.02	96.51	EI17.1	20x25 subhedral
7/4/96	21	0.01	dl	0.08	**dl**	0.02	dl	**dl**	46.2	53.72	100.10	#1	60 irregular
	22	dl	0.01	0.04	**0.06**	0.02	dl	**dl**	45.83	53.26	99.29	#2	
	23	dl	dl	0.03	**0.06**	0.03	dl	**dl**	47.07	54.11	101.41	#3	
	24	0.01	0.01	dl	**0.09**	dl	dl	**dl**	46.87	54.22	101.24	#4	100 irregular
	25	dl	dl	dl	**dl**	dl	dl	**dl**	46.78	54.12	101.00	#5	
	26	0.02	0.01	dl	**dl**	0.03	dl	**dl**	46.39	53.32	99.85	#6	
	29	dl	0.01	dl	**dl**	0.02	dl	**dl**	46.54	55.06	101.71	#9	10 euhedral
	33	0.01	0.01	dl	**dl**	dl	dl	**dl**	46.09	52.75	98.92	#13	100 irregular
	34	dl	0.01	dl	**0.07**	0.02	dl	**dl**	46.03	52.48	98.67	#14	
	35	dl	dl	0.01	**dl**	dl	dl	**dl**	46.73	53.24	100.06	#15	
	42	dl	0.01	dl	**dl**	0.01	dl	**dl**	46.27	53.23	99.61	#22	10x60 subhedral
	43	0.01	0.01	0.01	**dl**	dl	dl	**dl**	46.88	54.34	101.30	#23	
	44	0.01	0.02	dl	**dl**	0.02	dl	**dl**	46.76	53.82	100.71	#24	

Eastern Interior

EASTERN INTERIOR FINES

Date	Anal #	Se	Cu	Ni	As	Zn	Cd	Co	Fe	S	Total	Grain	Size (microns)/Form
1/3/97	83	dl	dl	dl	dl	dl	dl	nd	46.35	51.90	98.26	Py1.1	20 framboidal
	84	dl	0.00	dl	dl	0.00	dl	nd	46.68	51.92	98.61	Py1.2	25 framboidal
	85	0.01	0.01	0.01	0.01	dl	dl	nd	46.48	52.32	98.83	Py2.1	10x50 cleat
	86	dl	dl	dl	0.02	dl	dl	nd	46.70	52.31	99.04	Py2.2	
	88	dl	dl	dl	0.02	dl	dl	nd	45.75	50.92	96.69	Py4.1	20x30 subhedral
	89	dl	0.00	dl	dl	dl	dl	nd	46.40	50.78	97.19	Py5.1	35x35 irregular
	90	0.01	dl	dl	0.02	dl	dl	nd	44.98	50.34	95.35	Py5.2	
	91	dl	dl	0.05	0.01	0.00	dl	nd	46.31	52.21	98.58	Py6.1	20x20 subhedral
	92	0.01	0.02	0.04	0.01	0.01	dl	nd	45.53	51.29	96.91	Py7.1	30 framboidal
	93	dl	0.01	0.13	0.01	dl	dl	nd	45.34	51.24	96.73	Py7.2	
	94	dl	0.02	0.01	0.01	dl	dl	nd	45.82	51.49	97.36	Py8.1	20 subh./euhedral
	95	dl	dl	0.02	0.02	dl	dl	nd	46.18	51.35	97.58	Py9.1	20 framboidal
	96	0.01	0.02	dl	0.01	dl	dl	nd	46.54	51.23	97.82	Py10.1	50x70 framboidal
	97	dl	dl	0.01	0.01	dl	dl	nd	46.71	51.71	98.44	Py10.2	cluster
	98	0.02	dl	dl	0.01	dl	dl	nd	46.70	51.95	98.67	Py10.3	
	99	dl	dl	0.02	dl	dl	dl	nd	45.70	51.36	97.08	Py11.1	30x40 subhedral
	100	dl	dl	0.02	0.01	dl	dl	nd	46.33	52.30	98.66	Py11.2	
	101	dl	dl	0.04	0.02	dl	dl	nd	46.57	51.89	98.52	Py12.1	30 framboid with

	EASTERN INTERIOR FINES- Continued												
Date	Anal #	Se	Cu	Ni	As	Zn	Cd	Co	Fe	S	Total	Grain	Size (microns)/Form
1/3/97	102	dl	0.01	0.07	dl	dl	dl	nd	46.00	51.87	97.95	Py12.2	euhedral overgrowth
	106	dl	0.01	0.05	dl	dl	dl	nd	46.06	51.50	97.61	Py16.1	30x40 framb. cstr-core
	107	dl	dl	0.06	dl	dl	dl	nd	46.35	51.83	98.24	Py16.2	euhedral rim
	*dl= below detection limit of 100 ± 100 ppm, except arsenic values listed in boldface (dl=500 ± 500 ppm). nd=not detected.												
	Values for Co include a 0.06 wt. % empirical correction factor subtracted from measured values.												

						NORTHERN APPALACHIAN AS-MINED							
Date	No.	Se	Cu	Ni	As	Zn	Cd	Co	Fe	S	Total	Grain	Size (microns)/form
12/23/96	7	dl	dl	dl	0.04	dl	dl	nd	46.90	51.03	97.96	Py 1.1	3 mm composite
	8	dl	dl	dl	dl	0.01	dl	nd	46.63	50.67	97.32	Py 1.2	
	9	dl	dl	dl	0.04	dl	dl	nd	46.66	51.16	97.86	Py 1.3	
	10	0.01	dl	0.01	0.03	0.00	dl	nd	47.48	50.97	98.50	Py 1.4	
	11	dl	dl	dl	0.01	dl	dl	nd	47.23	51.63	98.87	Py 1.5	
	12	dl	0.02	dl	0.01	0.01	dl	nd	46.45	52.08	98.58	Py 2.1	500 irregular
	13	0.01	0.01	dl	0.02	dl	dl	nd	45.83	49.40	95.26	Py 2.2	
	14	0.03	0.03	dl	0.04	dl	dl	nd	47.28	47.04	94.42	Py 2.3	
	15	dl	dl	dl	0.03	dl	dl	nd	46.02	51.93	97.99	Py 3.1	15x70 cleat
	17	0.02	0.01	dl	0.02	0.02	dl	nd	45.58	49.42	95.08	Py 4.1	1 mm composite
	18	0.01	0.02	0.01	0.02	dl	dl	nd	47.08	50.17	97.31	Py 4.2	
	22	dl	dl	dl	0.06	0.01	dl	nd	46.21	51.86	98.14	Py 5.1	60x100 subhedral
	23	0.01	dl	0.00	0.03	0.02	dl	nd	47.42	51.87	99.35	Py 5.2	
	24	0.00	dl	dl	0.00	dl	dl	nd	47.66	51.72	99.40	Py 6.1	40x100 framboid
	25	0.00	dl	dl	0.02	dl	dl	nd	47.25	52.36	99.63	Py 6.2	cluster
	26	0.01	dl	dl	dl	dl	dl	nd	47.11	52.67	99.80	Py 7.1	60x90 framb. cluster
	27	dl	0.01	0.01	0.03	dl	dl	nd	46.66	50.28	96.99	Py 8.1	40 framboidal
	28	dl	dl	0.01	0.02	0.01	dl	nd	46.09	50.45	96.58	Py 8.2	rim on 8.1

NORTHERN APPALACHIAN AS MINED-Continued													
Date	Anal#	Se	Cu	Ni	As	Zn	Cd	Co	Fe	S	Total	Grain	Size (microns)/Form
12/23/96	29	dl	dl	0.03	0.02	dl	dl	nd	46.72	51.06	97.83	Py 9.1	35 framboidal
	30	dl	0.01	dl	dl	dl	dl	nd	46.47	50.90	97.39	Py 10.1	120 irregular
	31	dl	0.02	0.01	0.01	dl	dl	nd	46.25	49.82	96.11	Py 10.2	
	32	dl	dl	0.01	0.02	dl	dl	nd	46.42	52.82	99.26	Py 11.1	20 euhedral
	33	dl	dl	dl	0.03	dl	dl	nd	46.96	52.94	99.93	Py 12.1	25 framboidal
	34	dl	dl	0.01	0.03	dl	dl	nd	46.51	51.30	97.86	Py 13.1	30 framboidal
	35	dl	dl	dl	0.05	dl	dl	nd	47.27	52.54	99.87	Py 14.1	150 subhedral
	36	dl	0.01	dl	0.06	0.03	dl	nd	47.74	51.04	98.87	Py 14.2	
10/31/96	47		dl	dl	**0.12**	dl	dl	nd	46.52	51.96	98.60	NA1.1	2x3mm
	48		0.01	dl	**dl**	dl	dl	nd	46.35	52.20	98.64	NA1.2	
	49		0.01	dl	**0.09**	dl	dl	nd	46.15	51.78	98.03	NA1.3	
	50		dl	dl	**0.13**	dl	dl	nd	46.87	51.36	98.36	NA1.4	
	51		0.02	dl	**0.12**	dl	dl	nd	46.27	52.03	98.43	NA1.5	
	52		dl	dl	**0.07**	dl	dl	nd	46.60	52.15	98.82	NA1.6	
	53		dl	dl	**0.07**	0.01	dl	nd	46.06	51.77	97.91	NA1.7	
	54		dl	dl	**dl**	dl	dl	nd	46.56	51.46	98.06	NA1.8	
	55		dl	0.03	**0.13**	dl	dl	nd	45.58	51.08	96.81	NA2.1	20 framboidal
	57		dl	dl	**0.13**	dl	dl	nd	46.19	51.43	97.75	NA4.1	20x80 cleat
	58		dl	dl	**0.12**	dl	dl	nd	45.37	51.28	96.77	NA4.2	
	59		0.02	dl	**0.10**	dl	dl	nd	45.20	52.12	97.44	NA5.1	70 framboidal?

NORTHERN APPALACHIAN AS MINED-Continued													
Date	Anal #	Se	Cu	Ni	As	Zn	Cd	Co	Fe	S	Total	Grain	Size (microns)/Form
10/31/96	60		0.03	dl	**dl**	dl	dl	nd	45.72	52.41	98.19	NA5.2	
	61		0.01	0.01	**0.07**	dl	dl	nd	45.72	51.84	97.65	NA5.3	
	62		0.02	0.04	**0.09**	dl	dl	nd	45.63	50.74	96.53	NA6.1	20 framboidal
	63		dl	dl	**0.10**	0.01	dl	nd	45.84	51.43	97.38	NA7.1	15x30 subhedral
	64		0.01	dl	**0.12**	dl	dl	nd	45.46	51.14	96.74	NA8.1	20x30 subhedral/
	65		0.00	dl	**0.13**	dl	dl	nd	44.92	50.66	95.73	NA8.2	euhedral
	66		0.01	0.01	**0.13**	0.01	dl	nd	45.04	51.01	96.21	NA8.3	
	67		0.01	0.01	**0.10**	dl	dl	nd	45.91	50.90	96.92	NA9.1	10 euhedral
Jul-96	14	0.02	dl	dl	**0.13**	0.03	dl	**0.03**	46.57	51.25	98.17	#2	40 irregular
	15	0.01	0.22	0.01	**0.05**	0.14	dl	**0.03**	46.52	51.71	98.77	#3	80 irregular
	16	dl	0.18	dl	**0.05**	0.17	dl	**0.01**	47.00	51.39	98.89	#4	
	17	dl	0.08	0.01	**0.10**	0.06	dl	**0.03**	46.97	51.83	99.14	#5	50 irregular
	18	0.01	dl	dl	**0.12**	0.04	dl	**0.02**	46.66	51.54	98.46	#6	100 irregular
	19	dl	dl	dl	**0.08**	0.04	dl	**0.02**	46.78	51.14	99.17	#7	
	20	dl	0.01	0.01	**0.10**	0.04	dl	**0.03**	46.89	52.38	99.52	#8	
	24	dl	dl	dl	**0.10**	0.01	dl	**0.02**	46.41	51.94	98.59	#12	15 framboidal
	46	0.02	0.02	0.03	**dl**	dl	dl	**0.07**	45.85	51.9	97.99	#101	10 framboidal

NORTHERN APPALACHIAN AS-MINED (Continued)													
	Anal #	Se	Cu	Ni	As	Zn	Cd	Co	Fe	S	Total	Grain	Size (microns)/Form
Jul-96	47	dl	0.02	0.11	**0.09**	0.02	dl	**0.02**	46.08	52.67	99.36	#102	10 framboidal
	48	dl	0.08	0.11	**0.09**	dl	dl	**0.02**	44.7	53.24	98.91	#103	10 framboidal
	49	dl	0.08	0.06	**0.09**	0.01	dl	**0.02**	44.21	53.15	98.55	#104	10 framboidal
	50	0.04	0.53	0.28	**0.22**	0.01	dl	**0.06**	45.15	53.64	100.32	#105	10x30 cleat
	53	dl	0.03	0.02	**0.08**	0.01	dl	**0.02**	45.21	52.84	98.27	#109	22 framboidal
	54	dl	dl	dl	**dl**	0.01	dl	**0.01**	46.46	54.09	100.68	#109	20x60 cleat
	55	dl	0.06	0.13	**0.10**	0.02	dl	**0.03**	44.3	52.71	98.26	#110	50x70 framboidal
	56	dl	0.08	0.13	**0.10**	0.01	dl	**0.02**	44.93	52.87	98.98	#111	cluster
	57	dl	0.08	0.09	**0.11**	0.01	dl	**0.03**	44.84	52.85	98.52	#112	
	58	dl	0.02	0.02	**0.07**	0.01	dl	**0.03**	45.49	52.77	98.47	#113	20x40 irregular
	60	dl	dl	0.02	**dl**	0.01	dl	**0.01**	45.96	53.74	99.82	#115	20x30 framb. cluster
	65	dl	0.01	0.01	**0.07**	0.01	dl	**0.03**	46.57	52.85	99.62	#120	20x50 subhedral
	66	0.02	dl	0.01	**0.09**	dl	dl	**0.03**	46.49	52.87	99.6	#121	
	68	dl	0.01	dl	**0.09**	0.02	dl	**0.03**	46.16	53.26	99.63	#123	100x100 framb. cl.
	69	dl	0.01	dl	**0.07**	dl	dl	**0.01**	46.67	53.49	100.36	#124	
	70	0.06	0.13	0.05	**0.16**	0.02	dl	**0.02**	44.77	52.34	97.66	#125	
	71	0.01	0.01	0.02	**0.09**	0.02	dl	**0.04**	45.82	51.69	97.76	#126	
	72	0.08	0.10	0.10	**0.11**	dl	dl	**0.06**	43.78	50.82	95.11	#127	

NORTHERN APPALACHIAN FINES													
Date	Anal#	Se	Cu	Ni	As	Zn	Cd	Co	Fe	S	Total	Grain	Size (microns)/Form
12/26/96	56	dl	dl	dl	0.01	dl	dl	nd	45.87	51.55	97.43	py1.1	20x25 subhedral
	57	0.01	dl	0.00	dl	dl	dl	nd	45.81	51.81	97.63	py1.2	
	59	dl	dl	dl	0.01	dl	dl	nd	46.46	51.61	98.09	py2.1	20 euhedral
	62	dl	0.04	0.19	0.03	dl	dl	nd	45.20	51.78	97.24	py4.2	15x25 subhedral
	63	dl	0.03	0.06	0.03	0.01	dl	nd	44.90	51.22	96.25	py5.1	15 subh./framb.
	64	0.01	dl	0.01	0.08	dl	dl	nd	46.11	52.25	98.47	py6.1	20 subhedral
	67	dl	dl	0.01	0.05	dl	dl	nd	46.73	52.38	99.18	py8.1	30x60 subhedral
	68	0.01	dl	0.01	0.06	dl	dl	nd	46.60	52.56	99.25	py8.2	
	69	0.01	0.01	dl	0.01	dl	dl	nd	45.80	52.37	98.21	py9.1	40x40 subhedral
	70	0.02	dl	dl	dl	dl	dl	nd	45.72	52.05	97.78	py9.2	
	72	dl	dl	dl	dl	dl	dl	nd	45.93	52.00	97.93	py9.3	
	73	0.01	dl	dl	0.08	dl	dl	nd	46.20	52.29	98.57	py10.1	20x60 subhedral
	74	dl	dl	dl	0.06	dl	dl	nd	46.26	52.12	98.45	py10.2	

NORTHERN APPALACHIAN FINES-Continued													
Date	Anal #	Se	Cu	Ni	As	Zn	Cd	Co	Fe	S	Total	Grain	Size (microns)/Form
12/26/96	75	dl	dl	0.01	dl	dl	dl	nd	47.03	52.81	99.86	py11.1	8x65 cleat
	76	dl	dl	dl	0.02	0.00	dl	nd	46.54	51.72	98.29	py11.2	
	77	0.06	0.04	0.09	0.06	dl	dl	nd	46.15	51.48	97.88	py12.1	20x25 subh./euhed.
	78	dl	dl	dl	0.11	dl	dl	nd	45.62	51.86	97.59	py13.1	40x50 subhedral
	79	dl	dl	dl	0.11	dl	dl	nd	46.10	52.36	98.57	13.2	
	80	dl	0.01	0.02	0.04	dl	dl	nd	46.04	51.03	97.14	py14.1	15 framboidal
	81	dl	dl	0.02	0.01	dl	dl	nd	46.13	52.27	98.43	py15.1	10 euhedral
	82	dl	0.02	0.09	0.06	0.01	dl	nd	45.90	51.78	97.85	py16.1	20 framboidal

*dl= below detection limit of 100 ± 100 ppm, except arsenic values listed in boldface (dl=500 ± 500 ppm).
nd=not detected.

Values for Co include a 0.06 wt. % empirical correction factor subtracted from measured values.

						SOUTHERN APPALACHIAN AS MINED							
DATE	ANAL #	Se	Cu	Ni	As	Zn	Cd	Co	Fe	S	Total	Grain	Size (microns)/Form
1/3/97	136	dl	dl	dl	0.14	dl	dl	nd	45.84	50.88	96.86	py1.1	20 framboidal
	137	dl	dl	dl	0.03	dl	dl	nd	46.36	51.41	97.80	Py2.1	25 framboidal
	140	0.02	0.10	0.45	0.30	dl	dl	nd	45.32	50.89	97.08	Py5.1	50x60 framb. cluster
	141	dl	0.11	0.35	0.26	dl	dl	nd	44.93	50.47	96.12	Py5.2	
	142	0.02	0.03	0.03	0.04	dl	dl	nd	46.66	50.76	97.54	Py6.1	40x60 framb. cluster
	143	dl	0.02	0.03	0.03	0.01	dl	nd	46.51	51.15	97.75	Py6.2	
	144	dl	0.03	0.03	0.03	0.01	dl	nd	46.60	51.18	97.88	Py7.1	150x200 framb. cluster
	145	dl	0.01	0.08	0.08	dl	dl	nd	45.60	49.72	95.49	Py7.2	
	146	dl	0.03	0.04	0.04	0.01	dl	nd	45.59	50.42	96.12	Py7.3	
	147	dl	0.02	0.08	0.09	dl	dl	nd	46.42	50.79	97.40	Py7.4	
	148	dl	dl	0.04	3.97	dl	dl	nd	45.59	48.76	98.35	Py8.1	15x150 cleat
	149	dl	dl	0.04	3.22	dl	dl	nd	45.53	47.97	96.76	Py8.2	
12/12/96	16	0.01	0.03	0.07	0.15	0.02	dl	nd	45.70	50.01	96.00	Py1.1	20x40 subhedral
	18	dl	dl	0.01	0.10	dl	dl	nd	46.34	51.12	97.58	Py2.1	40x80 framb. cluster
	19	dl	dl	0.01	0.09	dl	dl	nd	45.61	49.83	95.54	Py2.2	
10/31/96	33		0.05	0.04	**0.13**	dl	dl	nd	44.66	50.17	95.04	SA2.1	15 framboidal
	34		0.02	0.07	**0.16**	0.01	dl	nd	45.13	50.41	95.80	SA3.1	20 framboidal
	36		0.03	0.08	**0.20**	dl	dl	nd	45.38	50.37	96.07	SA5.1	15x20 framb. cluster

SOUTHERN APPALACHIAN AS-MINED-Continued													
		Se	Cu	Ni	As	Zn	Cd	Co	Fe	S	Total	Grain	Size (microns)/Form
10/31/96	37		0.01	0.01	**0.13**	dl	dl	nd	45.99	50.49	96.63	SA5.2	
	42		dl	dl	**4.72**	dl	dl	nd	44.58	49.14	98.43	SA8.1	50x90 subhedral
	43		dl	dl	**2.17**	0.01	dl	nd	45.02	51.71	98.91	SA8.2	
	44		dl	dl	**2.93**	dl	dl	nd	45.80	50.13	98.87	SA9.1	50x70 irregular
	45		dl	dl	**1.15**	0.01	dl	nd	44.92	50.76	96.84	SA10.1	20x100 irregular
	46		dl	0.01	**1.26**	dl	dl	nd	45.30	51.38	97.95	SA10.2	
	72		dl	0.06	**0.27**	0.03	dl	nd	46.02	51.43	97.82	SA12.2	60x160 comp. irreg.
	74		0.03	0.01	**0.20**	dl	dl	nd	46.09	50.95	97.29	SA13.2	70x150 composite
	75		dl	0.02	**0.17**	dl	dl	nd	45.66	50.27	96.12	SA14.1	25 framboidal
	77		dl	0.03	**0.32**	dl	dl	nd	46.13	50.78	97.25	SA15.1	100x250 irregular
	79		dl	0.01	**0.56**	dl	dl	nd	46.34	50.58	97.50	SA15.3	
Jul-96	8	0.01	dl	0.06	**0.14**	0.02	dl	**0.03**	46.28	50.90	97.65	#2	60 irregular
	9	dl	0.01	0.04	**0.14**	0.03	dl	**0.02**	46.61	51.18	98.11	#3	
	10	0.01	0.01	0.03	**0.12**	0.05	dl	**0.02**	46.53	50.97	97.81	#4	
	11	dl	dl	0.08	**0.09**	0.03	dl	**0.04**	45.66	51.07	97.03	#5	9 framboidal
	12	dl	dl	0.08	**0.13**	0.02	dl	**0.03**	46.19	50.44	96.95	#6	9 framboidal
	28	0.01	dl	0.03	**0.12**	0.01	dl	**0.02**	46.32	51.50	98.15	#7	10 framboidal

SOUTHERN APPALACHIAN AS-MINED Continued													
Date	Anal #	Se	Cu	Ni	As	Zn	Cd	Co	Fe	S	Total	Grain	Size (microns)/Form
Jul 96	31	0.01	0.01	dl	**0.15**	0.03	dl	**0.02**	46.94	51.34	98.63	#10	20 irregular
	32	0.03	dl	dl	**0.27**	0.02	dl	**0.03**	46.68	51.59	98.80	#11	
	33	dl	dl	0.02	**0.21**	0.01	dl	**0.04**	45.57	50.01	95.97	#12	10 irregular
	34	0.02	dl	0.01	**0.15**	0.02	dl	**0.03**	46.49	52.07	98.90	#13	20 framboidal
	35	dl	0.03	0.02	**0.13**	0.02	dl	**0.03**	46.31	50.93	97.55	#14	
	36	dl	0.01	0.01	**0.11**	0.04	dl	**0.03**	46.31	51.51	98.13	#15	15 framboidal
	37	dl	0.02	0.14	**0.23**	0.03	dl	**0.29**	44.91	49.37	95.22	#16	10 framboidal
	38	dl	0.05	0.13	**0.19**	0.03	dl	**0.23**	45.74	50.11	96.60	#17	25 irregular
	SOUTHERN APPALACHIAN FINES												
1/3/97	8	0.02	0.04	0.05	0.02	0.01	dl	nd	43.61	51.46	95.20	py1.1	20x40 subhedral
	9	dl	0.02	0.02	dl	0.01	dl	nd	45.23	51.92	97.20	py1.2	
	12	0.00	0.01	0.01	0.01	dl	dl	nd	45.59	51.34	96.97	py3.1	30x40 irregular
	13	dl	0.04	0.02	0.02	dl	dl	nd	45.29	51.20	96.56	py3.2	
	14	dl	dl	0.02	0.03	dl	dl	nd	46.34	51.86	98.25	py4.1	20 framboidal
	15	dl	0.01	0.00	0.20	dl	dl	nd	45.49	51.62	97.33	py5.1	20 framboidal
	16	dl	0.01	0.01	0.87	0.01	dl	nd	45.42	50.33	96.66	py6.1	25x30 subhedral
	21	dl	dl	0.02	0.10	0.01	dl	nd	45.96	51.28	97.36	py10.1	20 double framb.

SOUTHERN APPALACHIAN FINES (Continued)													
Date	Anal #	Se	Cu	Ni	As	Zn	Cd	Co	Fe	S	Total	Grain	Size(microns)/Form
1/3/97	25	0.02	dl	0.01	0.01	dl	dl	nd	45.17	51.36	96.56	py12.1	20x30 irregular
	26	dl	0.01	0.01	dl	0.01	dl	nd	44.69	50.77	95.49	py12.2	
	30	dl	dl	dl	0.64	dl	dl	nd	46.30	52.25	99.19	py14.1	15x20 irregular
	31	dl	dl	0.01	0.93	dl	dl	nd	45.64	51.34	97.93	py15.1	40x50 subh./
	32	dl	dl	0.00	1.18	dl	dl	nd	45.70	51.33	98.22	py15.2	framboid cluster
	33	dl	0.01	0.01	1.18	dl	dl	nd	45.31	51.22	97.73	py15.3	
	34	dl	dl	dl	1.09	dl	dl	nd	45.62	51.81	98.52	py15.4	
	35	dl	0.01	0.03	0.17	0.01	dl	nd	45.53	50.88	96.62	py16.1	25 framboidal
	36	dl	dl	0.04	0.29	dl	dl	nd	45.06	51.18	96.57	py16.2	
	*dl= below detection limit of 100 ± 100 ppm, except arsenic values listed in boldface (dl=500 ± 500 ppm). nd=not determined.												
	Values for Co include a 0.06 wt. % empirical correction factor subtracted from measured values.												

B. Illite and Kaolinite Analysis

Appendix 2b.
Illite and Kaolinite Analyses. As-Mined Coals

Analysis #	K_2O	CaO	Na_2O	Al_2O_3	SiO_2	MgO	Cr_2O_3	MnO	FeO	TiO_2	Total	Comment
						96020101	Eastern Interior					
20	3.31	0.47	0.56	21.45	50.11	3.08	0.03	0.04	6.30	0.30	85.63	illite 5.1
31	3.53	0.22	0.63	21.19	56.02	1.82	0.05	dl	3.04	0.34	86.84	illite 8.3
32	3.34	0.07	1.41	26.53	59.02	1.12	0.03	dl	1.58	0.21	93.32	illite 8.4
19	0.19	0.06	0.04	39.12	46.66	0.09	dl	dl	0.28	0.02	86.46	kaol. 5.1
41	0.32	0.06	0.05	40.30	48.56	0.19	0.03	0.04	0.50	0.02	90.06	kaol. 1.3
						95120401	Southern Appalachian					
50	1.68	0.15	0.44	34.46	50.17	1.22	0.02	0.02	2.93	0.80	91.88	illite 1.3
51	3.36	0.07	0.23	31.04	56.11	1.05	dl	0.04	1.60	0.44	93.96	illite 1.4
52	2.96	0.13	0.37	31.16	53.37	1.24	0.03	dl	2.46	0.80	92.52	illite 1.5
53	2.98	0.14	0.24	32.23	53.30	1.04	0.02	dl	2.25	0.48	92.67	illite 1.6
55	4.23	0.04	0.30	29.92	53.32	1.28	dl	dl	2.25	0.27	91.62	illite 2.2
58	3.76	0.03	0.28	30.54	55.50	1.11	0.02	dl	1.75	0.18	93.18	illite 2.5
61	4.68	0.06	0.20	29.06	56.90	1.12	dl	dl	2.62	0.42	95.07	illite 3.3
63	5.08	0.12	0.23	33.89	51.73	1.25	0.02	dl	1.95	0.86	95.13	illite 3.4
65	6.68	dl	0.22	32.82	51.10	1.51	0.04	dl	1.71	0.50	94.60	illite 4.2
68	2.73	0.18	0.21	32.34	49.08	2.82	0.03	0.02	6.47	0.18	94.06	illite 4.5
69	3.26	0.04	0.19	29.83	58.81	0.84	0.02	dl	1.36	0.13	94.48	illite 5.1
72	3.26	0.07	0.22	27.12	56.91	1.23	0.04	0.02	2.85	3.93	95.64	illite 5.4
77	7.08	dl	0.08	30.98	51.20	1.52	0.05	0.03	2.43	0.39	93.77	illite 6.5
81	4.57	0.10	2.21	35.11	50.22	1.02	dl	0.07	2.41	0.09	95.79	illite 7.4

						95110901	Northern Appalachian					
86	6.41	dl	0.10	33.17	49.87	1.27	dl	dl	2.16	0.26	93.24	illite 1.4
87	3.80	0.15	0.19	30.98	45.87	1.64	0.02	0.04	6.84	0.67	90.21	illite 2.1
88	6.29	0.05	0.22	30.86	46.75	1.76	0.05	dl	6.09	0.38	92.46	illite 2.2
89	7.55	dl	0.03	28.93	48.25	1.83	dl	0.04	6.34	0.21	93.18	illite 2.3
90	5.82	0.04	0.15	31.08	49.08	1.55	0.04	dl	4.88	0.45	93.09	illite 2.4
91	5.56	dl	0.20	35.40	50.60	1.04	0.02	0.03	2.88	0.04	95.78	illite 3.1
92	5.56	0.02	0.16	29.74	51.33	2.50	dl	0.04	3.62	0.31	93.28	illite 3.2
94	5.07	0.05	0.18	25.46	58.99	1.76	dl	dl	2.59	0.33	94.44	illite 3.4
101	1.53	0.29	0.15	34.98	51.83	1.08	dl	dl	2.00	0.02	91.88	illite 4.1
103	1.73	0.24	0.17	34.26	51.81	1.22	dl	0.02	2.03	0.12	91.60	illite 4.3
95	0.05	0.04	0.04	38.93	47.10	0.05	0.02	dl	0.26	0.02	86.52	kaol. 1.1
96	0.09	0.05	0.07	39.21	48.19	0.18	dl	0.02	1.05	dl	88.86	kaol. 1.2
97	0.11	0.06	0.06	39.72	48.61	0.15	0.02	dl	0.92	0.08	89.73	kaol. 1.3