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Contract No. 210-76-0119

# FINAL REPORT

# Chemical and Physical Characterization

# of

# Smelter Dusts

Submitted by

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16. Abstracts

In support of the National Institute for Occupational Safety and Health Contract No. 210-76-0119, Walter C. McCrone Associates, Inc. acted as a contractor to perform a physical and chemical characterization of smelter dusts. Specifically the objective of this work was to obtain for each of five smelter samples as complete a physical and chemical characterization as possible for at least 1000 individual particles from each dust. In performing this study an automated electron microprobe was used to analyze the representative particles from the five samples. The analytical techniques used in performing the statement of work along with the data generated from each of the particles analyzed were detailed in bi-monthly progress reports throughout the period of performance of the contract. Due to the nature of the statement of work and due to the detail of information provided in the bi-monthly status reports, details of our analytical progress during the performance of the contract has been kept to a minimum and in this report we have detailed the techniques used for sample preparation, for sample analyses, instrument conditions and an evaluation of the data itself.

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smelters, dusts, analytical-methods, sampling

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## I. INTRODUCTION

In support of the National Institute for Occupational Safety and Health Contract No. 210-76-0119, Walter C. McCrone Associates, Inc. acted as a contractor to perform a physical and chemical characterization of smelter dusts. Specifically the objective of this work was to obtain for each of five smelter dust samples as complete a physical and chemical characterization as possible for at least 1000 individual particles from each dust. In performing this study an automated electron microprobe was used to analyze the representative particles from the five samples. In total, 6027 particles were analyzed using the fully automated electron microprobe. That is, 1657 from Sample 1, 1005 from Sample 2, 1155 from Sample 3, 1029 from Sample 4 and 1081 from Sample 5. The analytical techniques used in performing the statement of work along with the data generated from each of the particles analyzed were detailed in bi-monthly progress reports throughout the period of performance of the contract. Forwarded to the Project Officer under separate cover are all of the raw data developed in the performance of this contract. In addition to this raw data,appended to this report are the computerized tables summarizing the analytical results for each of the five dust samples analyzed. That is,for each of the five samples there are five different tables. Table A-1 details the particle size distribution for each sample, Table A-2 summarizes the normalized major elements for each sample, Table A-3 gives a further breakdown of the normalized major elements, Table A-4 lists the total major elements from the particles analyzed in the sample and Table A-5 summarizes the average particle size of each of the major elements within the sample. Due to the nature of the statement of work and due to the detail of information provided in the bi-monthly status reports, details of our analytical progress during the performance of the contract has been kept to a minimum; whereas in this report we have taken the opportunity to detail the techniques used for sample preparation, for sample analyses, instrument conditions and an evaluation of the data itself. For example, how good is the data generated using the automated electron microprobe.

Introduction, cont'd.

What are the short comings in the data, in the instrument itself and in the methods used? And, finally, a comparison of the various bulk analyses performed and the individual particle analyses performed using the automated electron microprobe.

## II. SAMPLE PREPARATION

The five samples for electron microprobe analysis were received in glass vials with each vial containing the amount of sample shown in Table 1. The samples were composed of small particles ranging in size from sub-micrometer to approximately 10 $\mu$m. Most of the particles in each vial were heavily agglomerated therefore for microscopical examination the particles had to be dispersed on a glass slide using immersion oil and xylene. Our normal procedure for electron microprobe analyses involves either individually mounting particles on a polished beryllium plate or dispersing a group of many particles for automated analyses. Aside from the ease in visual observation a smooth highly reflective beryllium plate is used, as beryllium yields an extremely low x-ray background and therefore being indetectable in the electron microprobe. Per the suggestion of the project officer, we used water as the dispersion medium and after prolonged ultrasonic agitation, small droplets of the dispersed particles were evaporated onto a beryllium plate and examined in the light microscope. Since the majority of the particles were still agglomerated after evaporation, several organic solvents were tried as a dispersant to achieve a better separation. Experimentally we found that the best particle separation was obtained using xylene at densities below 0.5% by weight (0.5 g dust in 100 g of xylene). After prolonged ultrasonic agitation a few droplets of the suspension were again placed on a beryllium plate and allowed to evaporate. Visual examination verified that at least 95% of the particles had been separated and that the degree of agglomeration was negligible. Two sets of such samples were then prepared from each vial. Each preparation covered an area of approximately 9 $mm^2$ ($9 \times 10^6$ $\mu m^2$) and with a density of one particle per 1000 square micrometers, a total of approximately 9000 small particles were mounted for analysis. The inherent electrostatic forces between the particles and the beryllium substrate and the presence of traces of soluble organics within each sample were sufficient to keep the particles attached to the mounting plate during our electron microprobe analysis.

In addition to the two sets of samples for discrete (individual) particle analysis, an additional set of samples with high particle density for bulk analysis was prepared. These samples were easily prepared by suspending a large number of particles in a small quantity of xylene and then evaporating relatively large droplets onto a beryllium plate. Each of these bulk samples contained well over a million small particles. As we have determined from our previous experience small particles (below 10 $\mu$m) do not require an electrically conductive coating, thus none was applied to these samples.

## III. INSTRUMENTATION AND OPERATING CONDITIONS

Chemical analysis of the small particles from each of the samples was performed using a completely automated Applied Research Laboratory EMX type electron microprobe. During the analyses for these studies the electron microprobe was operated with an accelerating voltage of 20 kV, a specimen current on beryllium of 25 nA and a 0.5 $\mu$m diameter electrom beam. Each of the three crystal spectrometers mounted within the EMX were used for the detection of 25 predetermined x-ray lines and the measurement of their respective intensities. For example, spectrometer number one is fitted with a lithium fluoride crystal which was used for the detection of mostly heavy and intermediate atomic weight elements. Spectrometer number two has an ADP crystal and was used for the detection of both light and heavy elements. Spectrometer number three has a KAP crystal and was used to detect most of the light elements such as sodium, fluorine, oxygen, etc. Table 2 lists the elements detectable for each of the three spectrometers. Each x-ray channel has a pulse height analyzer which was permanently set on a threshold of 0.5 volts with an electronic window of 10 v. The beam current was maintained constant by the digital computer.

## IV. SOFTWARE

The completely automated electron microprobe was entirely under the control of our PDP-11/15 computer system which had been interfaced to the EMX through an especially designed McCrone interface system. Although several specific computer programs have been developed, the software system used for this study was designed to detect small particles by changes in the specimen current. The small particle analysis program was designed to locate a small particle mounted on a polished substrate, measure its size and calculate its chemical composition from the measured x-ray intensities. Using the corresponding input parameters the teletype prints out the total rectangular area searched, the stage stepping increments, the beam scan area and the number of raster lines within the beam scan area.

The detection of a particle is perceived by the computer as a decrease in the absorbed specimen current signal as the beam rasters through a search area of approximately 24x30 $\mu$m. Specifically, a decrease in the specimen current signifies the presence of material with an average atomic number greater than that of the beryllium substrate (Z=4). As beryllium is a low atomic number element, over 99% of the particles have a higher backscattering factor, thus giving a lower specimen current. For example, light minerals such as $Al_2O_3$ cause an electron backscatter about 10% greater than that of beryllium. However, materials which cause less than 10% backscatter are usually organic in nature and cannot be differentiated by automated microprobe analyses. The input parameter for the decrease in specimen current can be chosen so that only material of general interest would be selected for analysis. For small particles the electron backscatter decreases rapidly due to the particle size effect; therefore during the study the increment for the change in specimen current relative to the substrate ranged from 2-5%.

When a decrease in specimen current is detected within the programmed value, the beam transverses the particle in X and Y directions. The computer then calculates the center of the particle and positions the beam on the particle. If the average dimension is smaller than the input requirement

(>0.5 $\mu$m particle size), the particle is rejected and the beam continues searching for another particle. If the particle physically overlaps the perimeter of the raster area the beam is centered only on that portion of the particle which is within the raster area. For practical reasons minimum particle size of 0.5 to 1 $\mu$m was accepted for the routine mass scan work. A minimum particle size can be established at any greater value depending on the requirements (degree of dispersion and maximum particle size) for the sample. Once the beam is positioned on the particle the spectrometers then cycle through the normal operating range to provide a complete semiquantitative analysis. The chemical composition, raster area and particle size are determined and recorded within the computer system and thereafter printed out by the teletype. The new data are stored on magnetic tape and computed values may be output onto various periphials. After completing an analysis, or if a particle is not detected within the raster area, the stage moves to the next raster position. During this process the beam current is monitored for drifts and adjusted if necessary. This sequence is repeated until the dimensional limits of the mass scan are reached or an operational failure occurs.

The actual analysis of a particle is performed as the spectrometers are reset to the initial position and the three scanners start to operate in parallel to measure the intensity of the 25 elements and 12 background positions. The counting or accumulation period used throughout this study was 10 seconds as manually preset on the timers. Each measured value is read and stored by the computer after the scanner cycle has been completed.

The percentages of the elements as detected are calculated and corrected for background. These values are then stored within the computer and printed out on the teletype in decreasing order of the composition within the percentage categories. The categories are as follows: major for elemental percentage >5%; minor for 1-5%; and trace for <1%. Elements present in trace quantities are not printed out.

The elemental percentages are calculated using a standard intensity file and a general background correction file. The standard intensities are compiled from primary x-ray line intensities measured on the pure elemental standards.

In turn, the general background file is computed from the interelement interference file and the measured background intensities. The interelement interference file is generated by analyzing each standard and extracting all the background intensities other than primary lines. This generated information therefore provides the intensity ratios that any element contributes to any other element's primary intensity measurement. As these files are sensitive to individual spectrometer misalignment or drifts they are monitored on a regular basis. The continuum for background values are determined by fitting an empirical curve to the values measured at selected spectral background positions. The interference and background calculations are then summed and subtracted from the total observed counts for each element. The elemental composition is calculated to an unnormalized first approximation by ratioing the net counts to the expected values for pure elements. The concentrations are then normalized to 100%.

An example of the actual data generated and printed out during an analysis is shown in Table 3. The first six columns of the analysis printout list the chemical symbol of the detected element and the respective raw count data. Column 7 lists the unnormalized-background-corrected first approximations for the major and minor concentrations. These values vary with the established specimen current and accumulation intervals as well as sample variety. Column 8 represents the background-corrected intensities normalized to 100% and Column 9 gives the standard deviation based upon the counting statistics from the normalized column.

The analytical data determined from our electron microprobe analyses are semiquantitative in nature since no atomic number, absorption, or fluorescence corrections were performed on any of the sample data. The absence of a ZAF correction generally introduces a relative error of 2-5%. In extreme cases such as very light elements in a heavy matrix this relative error could be as large as 20-30%. However, on very small particles (approximately 2 $\mu$m in size) the ZAF correction is not applicable since the absorption effect (the most important of all the corrections) is reduced significantly due to the physical size of the particle. That is, the shorter the absorption path the less the x-rays are absorbed by the particle itself.

## V. STANDARDIZATION, INTENSITIES, PEAKS, BACKGROUND FILES

Before performing the electron microprobe analyses both the microprobe and the computer were standardized following routine procedure. After adjusting the sample current to a predetermined value (25 nA) at a 20 kV accelerating potential, a pure element standard or a pure compound was analyzed and evaluated. This standardization and/or calibration procedure consists of locating the precise position of the major x-ray line for the standard, measuring the number of x-ray counts for 10 seconds and then storing both of these values in the computer memory. At various settings near the major peak a number of positions are selected for measuring background intensities and in turn these values are also stored in the computer for later use in calculating the background file.

As in most highly sophisticated analytical instruments calibration is an essential task. As the electron microprobe has been operating in an automated mode for nearly ten years, the x-ray spectrometers were continuously checked to verify their positionings for both the major lines and the background. Using a series of standards the intensities of these lines were routinely measured after the analysis of approximately 500 small particles.

The necessity for such frequent measurements is the frequent changes in these values. As the x-ray diffracting crystals normally deteriorate with use and lose their efficiency, alignment of the x-ray spectrometers is extremely critical. Any slight misalignment could cause up to a 20% change in the x-ray intensity of a particular element within a week's time. Another source of fluctuation in the x-ray intensities is the proportional counter itself; the counters are affected by temperature, pressure (flow type) and changes in voltage and thus can also cause significant changes in the respective intensities. The factors mentioned will affect the count rates of both the x-ray peaks and the background positions. Since the count rates determined for backgrounds are commonly one to two orders of magnitude lower than the peaks of interest,

background fluctuations can easily affect the analytical results more severely. Therefore, in our analyses we had to use more than one background file for each sample in order to maintain consistency with the analytical results.

Most of the elements searched for within our analyses were easily analyzed but some presented special problems. For example, the most serious problem was encountered in analysis of oxygen due to a low crystal efficiency in detecting this element. With the background values for oxygen at 2-5 cts/sec and the peak values at 20-50 cts/sec the slightest drift or change in the spectrometer alignment produced erroneous results ranging from a total absence of oxygen to an abundance of various oxides. Therefore, the measurement of oxygen was specifically watched.

Similar difficulties were experienced with some of the other light elements such as fluorine or sodium. In order to detect these elements when the count rates were at a low level, the sensitivity had to be increased; in some instances the sensitivity increased so much that the computer program occasionally calculated and produced particles with unusual compositions. For example, the detection of iodine as shown in Table A5-2 is artificially detected due to the low background. Such results were sometimes further caused by spurious electrical pulses during elemental peak measurements.

## VI. SAMPLE ANALYSES

For each of the five samples, approximately 1000 particles from the finely dispersed sample preparation were analyzed for 25 elements, using the automated mass scan technique. The data generated during these analyses are summarized within the data tables of the Appendix. Although we were very pleased with the overall performance of the instrument during our analyses, we experienced numerous short comings as the analytical work progressed. Thus, the subsequent analytical procedure was rectified and readjusted with more than 1000 particles being analyzed from each sample. The main reason for analyzing particles in excess of 1000 was to compensate for the earlier data that was unfortunately poor data. In particular, during the analysis of Sample 1 the operating conditions deteriorated and had to be readjusted thus causing poor

and erroneous data on the first few hundred particles. The analytical data obtained during our automated electron microprobe analysis of the small particles from each of the samples are presented in the various individual computer printouts. For each particle analyzed the computer printout contains the sample and particle number, the size and the elemental components detected in major and minor quantities. A summary of the results obtained on all the particles are presented in six different tables prepared for each of the samples (see Appendix A). The tables are as follows:

Table A-1 summarizes for each sample the particle size distribution of the particles analyzed. The particles are characterized into nine size ranges varying from <1 $\mu$m to >8 $\mu$m. The table contains the number and percentage of particles in each size range.

Table A-2 summarizes the chemical composition of all the particles into two groups: elements with concentrations of 5-10% and those with >10% concentration by weight.

Table A-3 is similar to Table A-2 except that it contains a more detailed division of the particles according to chemical composition in each percentage range, i.e., 5-10%, 10-30%, 30-50%, 50-75% and over 75%.

Table A-4 lists the total number of particles for each major element identified in a particular sample.

Table A-5 summarizes the relationship between the size, the chemical composition and frequency of the occurrence of the particles in each sample.

A. Bulk Analysis Data

As mentioned in the sample preparation section, five bulk samples, one from each sample, were prepared for electron microprobe analyses. Following our analyses of these bulk samples a comparison of the resultant data was made with these data supplied by NIOSH from their bulk analyses of similar samples. The results of both bulk analysis are given in the attached block diagrams in Figures 1 and 2. Despite the general apparent agreement of these

results, there are considerable differences in the relative proportions of certain elements. In our opinion the major reason for the discrepancy appears to be the sampling technique which in spite of the small particle size is extremely difficult to use and thus cannot ensure a homogeneous sample mounting. Also due to the presence of a small quantity of artifacts such as fibers, organic material and other trace materials, and due to the presence of inherent electrostatic forces on the particles, there is a significant degree of segregation present within the vials. This resultant segregation is a major cause of the heterogeneous samplings from the glass vials. We believe that the initial bulk analyses which we performed prior to our single particle analyses are more representative of the samples than those data supplied to us.

Another possible source of disparity in the comparison of the two data sets is the solubility of some of the dust components in the dispersant solvent, xylene. An additional source of discrepancy was the segregation of the particles after they had been dispersed and dried within the droplets on the beryllium plate. During our microscopical examination, it was observed that a distinct segregation of smaller particles had occurred towards the periphery of the droplet while the center portion contained relatively coarse particles. This segregation was partially compensated for by the random sampling of the areas for electron microprobe analysis within the droplets.

B. Data Fluctuations and Limitations

One of the first problems encountered was the steady drift of the sample current due to rapid aging of the instrument filament. Although the computer is programmed to compensate for such drifts with a relatively short response time, the drifts had caused a number of erroneous readings before the computer could react. These therefore caused a number of non-existent particles to be analyzed revealing results which were more or less random.

We also realized that the stability of the spectrometers varied from spectrometer to spectrometer resulting in non-detectable elemental peaks. This error was rectified by more frequently aligning the spectrometers and by continuously checking the intensities for most of the elements.

The inherent problems caused during the analysis of agglomerated particles was avoided only by extreme sample dispersion. Such sparse dispersions, however, increased the time for analysis as the computer required considerably more time to search for the particles than in the early work where the dispersion of the particles was significantly less. Therefore, in reality, our average speed of analysis ranged from 5 to 8 particles per hour.

Another factor which affected our average analysis time was the electron microprobe filament life. The filament life is definitely too short for a fully automated analysis since without manual readjustment and reduction of the filament current, the filament would last only a few hours. A considerably longer filament life time was obtained by manually reducing the filament current every few hours. It is possible to computerize this part of the procedure and it will probably be incorporated into the program in the future.

The analytical data generated during our analyses show a general correlation between the hardness of the dust components and particle size. For example, in general relatively hard compounds such as silicates were found predominantly in the larger particle sizes while the softer compounds were found more within the smaller size ranges. Also the bulk analyses data shown in Figure 1 reveal good correlation with the individual analyses summarized in Figures 3-8. It should be noted, however, that the lower limit of particle size for analysis was dictated by the electron beam size at 25 nA sample current. This limitation was 0.5 $\mu$m and it restricted our complete evaluation of the particle size versus sample composition study. It is apparent from our examination of the samples that a large percentage of the small particles are below 0.4 $\mu$m and thus they could not be analyzed within the present instrumentation while operating in an automatic mode.

Although not a major problem, an occasional electrical pulse within the main power supply and within other electronic components about our building may have caused erratic reading on the x-ray counters thus creating extremely high intensities on the spectrometers. This electronic noise is very difficult to eliminate and was corrected or compensated for within this work by a purely

statistical analysis of the results obtained from analyzing a large number of particles from each sample. Knowing the average background and the collected intensities of pure elements, the percentages of each element in a small particle was calculated. Since the total x-ray intensity from a small particle decreases as a function of particle size, the percentage of all detectable elements were then normalized and the elements above 5% were categorized as major and minor elements.

Table 1. Net Weight of Each Dust Sample as Received

| Sample no. | Weight (g) |
|---|---|
| 1 | 11.0 |
| 2 | 8.8 |
| 3 | 4.8 |
| 4 | 4.8 |
| 5 | 5.8 |

| SPECTROMETER #1 | | | | | SPECTROMETER #2 | | | | | SPECTROMETER #3 | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Atomic number | Chemical symbol & BKG stop | Spectrometer positions Theor. | Act | Intensity Cts/10 sec. | Atomic number | Chemical symbol & BKG stop | Spectrometer positions Theor. | Act | Intensity Cts/10 sec. | Atomic Number | Chemical symbol & BKG stop | Spectrometer positions Theor. | Act | Intensity Cts/10 sec. |
| 19 | K | 3454 | 3454 | 4600 | | B7 | | 3760 | 15 | 8 | O | 3655 | 3640 | 135 |
| | B1* | | 3470 | 70 | 12 | Mg | 3743 | 3754 | 3300 | | B10 | | 3500 | 12 |
| 51 | Sb | 3439 | 3436 | 64000 | 33 | As | 3661 | 3670 | 2300 | 9 | F | 2822 | 2842 | 1400 |
| 20 | Ca | 3361 | 3356 | 120000 | 34 | Se | 3403 | 3408 | 10000 | | B11 | | 2500 | 40 |
| 52 | Te | 3289 | 3287 | 47000 | 35 | Br | 3170 | 3175 | 6000 | 11 | Na | 1836 | 1850 | 100000 |
| | B2 | | 3165 | 60 | 13 | Al | 3156 | 3160 | 55000 | | B12 | | 1700 | 170 |
| 55 | I | 3148 | 3146 | 30000 | 14 | Si | 2697 | 2700 | 72000 | | | | | |
| 22 | Ti | 2750 | 2750 | .74000 | | B8 | | 2600 | 45 | | | | | |
| 23 | V | 2505 | 2506 | 54000 | 80 | Hg | 2145 | 2136 | 8000 | | | | | |
| | B3 | | 2325 | 90 | 81 | Ti | 2067 | 2065 | 12000 | | | | | |
| | B4 | | 2305 | 95 | 16 | S | 2033 | 2031 | 21000 | | | | | |
| 24 | Cr | 2291 | 2294 | 63000 | 82 | Pb | 2000 | 1998 | 14000 | | | | | |
| | B5 | | 2050 | 114 | | B9 | | 1950 | 60 | | | | | |
| 26 | Fe | 1937 | 1944 | 5300 | 48 | Cd | 1415 | 1486 | 28300 | | | | | |
| 29 | Cu | 1542 | 1547 | 3400 | | | | | | | | | | |
| | B6 | | 1500 | 300 | | | | | | | | | | |
| 30 | Zn | 1436 | 1442 | 2700 | | | | | | | | | | |

* The B-numbers refer to the various background stops

Table 2. List of elements analyzed in the electron microprobe with the spectrometer stopping points for peaks and backgrounds and the x-ray count rates.

Table 3. An example of the data output during an analysis

REPORT OUTPUT ON 2- 1-78

****** DATA ACQUIRED ON ******
1-13-78

SAMPLE NR - N-3-1 NITNR - 1
NIT SIZE = 3.95

TOTAL COUNTS FOR ALL ELEMENTS REQD.

| Column 1 | | Column 2 | | Column 3 | | Column 4 | | Column 5 | | Column 6 | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | 37. | K | 32. | SB | 19. | CA | 40. | TE | 36. | | 25. |
| I | 29. | TI | 53. | V | 50. | | 41. | | 54. | CR | 60. |
| | 38. | FE | 61. | CU | 251. | | 296. | ZN | 468. | | 41. |
| AS | 50. | SE | 42. | BR | 24. | AL | 29. | SI | 63. | | 56. |
| HG | 69. | TL | 67. | S | 75. | PB | 76. | | 73. | CD | 320. |
| O | 76. | | 53. | F | 47. | | 57. | NA | 56. | | 69. |
| MG | 70. | XX | 0. | | 0. | | 0. | | 0. | 0. | 0. |

| EL | Column 7 MEASURED | Column 8 MEAS. % | Column 9 STD. DEV. |
|---|---|---|---|
| MAJOR ** | | | |
| ZN | 0.90 | 43.70 | 15.00 |
| PB | 0.70 | 34.60 | 6.90 |
| CD | 0.20 | 11.70 | 1.50 |
| S | 0.10 | 7.40 | 1.20 |
| MINOR * | | | |
| SI | 0.10 | 2.60 | 1.30 |

** MAJOR ELEMENTS ARE ABOVE 5% NORMALIZED
* MINOR ELEMENTS ARE BETWEEN 1% AND 5% NORMALIZED

***** ALL ELEMENTS (NORMALIZED) BELOW 1% ARE NOT PRINTED *****

Columns 1-6: Lists the raw count data for the chemical symbols of elements detected and the respective background data

Column 7: Unnormalized-background-corrected first approximations

Column 8: Background corrected intensities normalized to 100%

Column 9: Standard deviation associated with Column 8

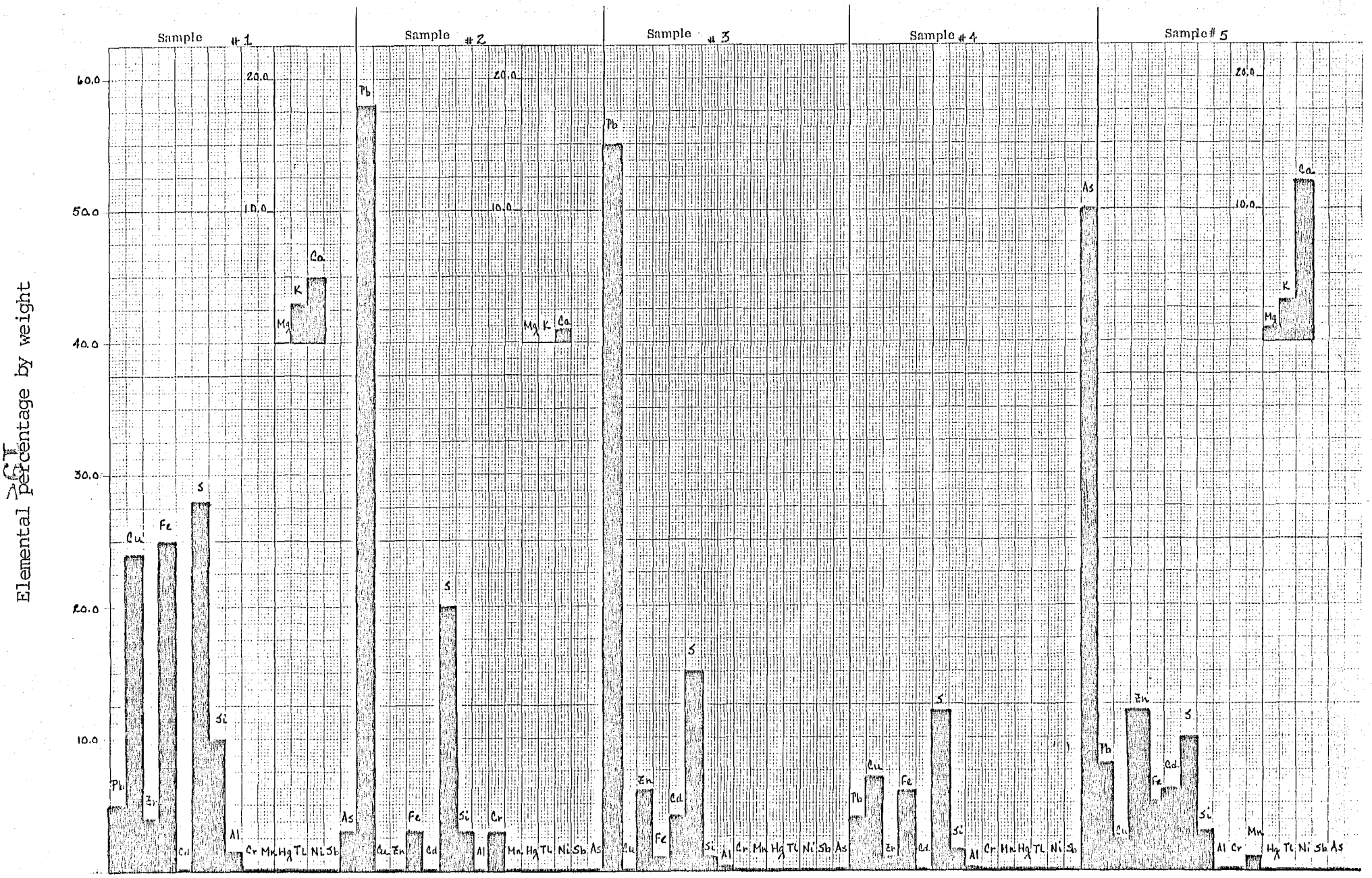


Figure 1. Bar graph showing the elemental composition of the five dust samples as determined from McCrone Associates' analyses

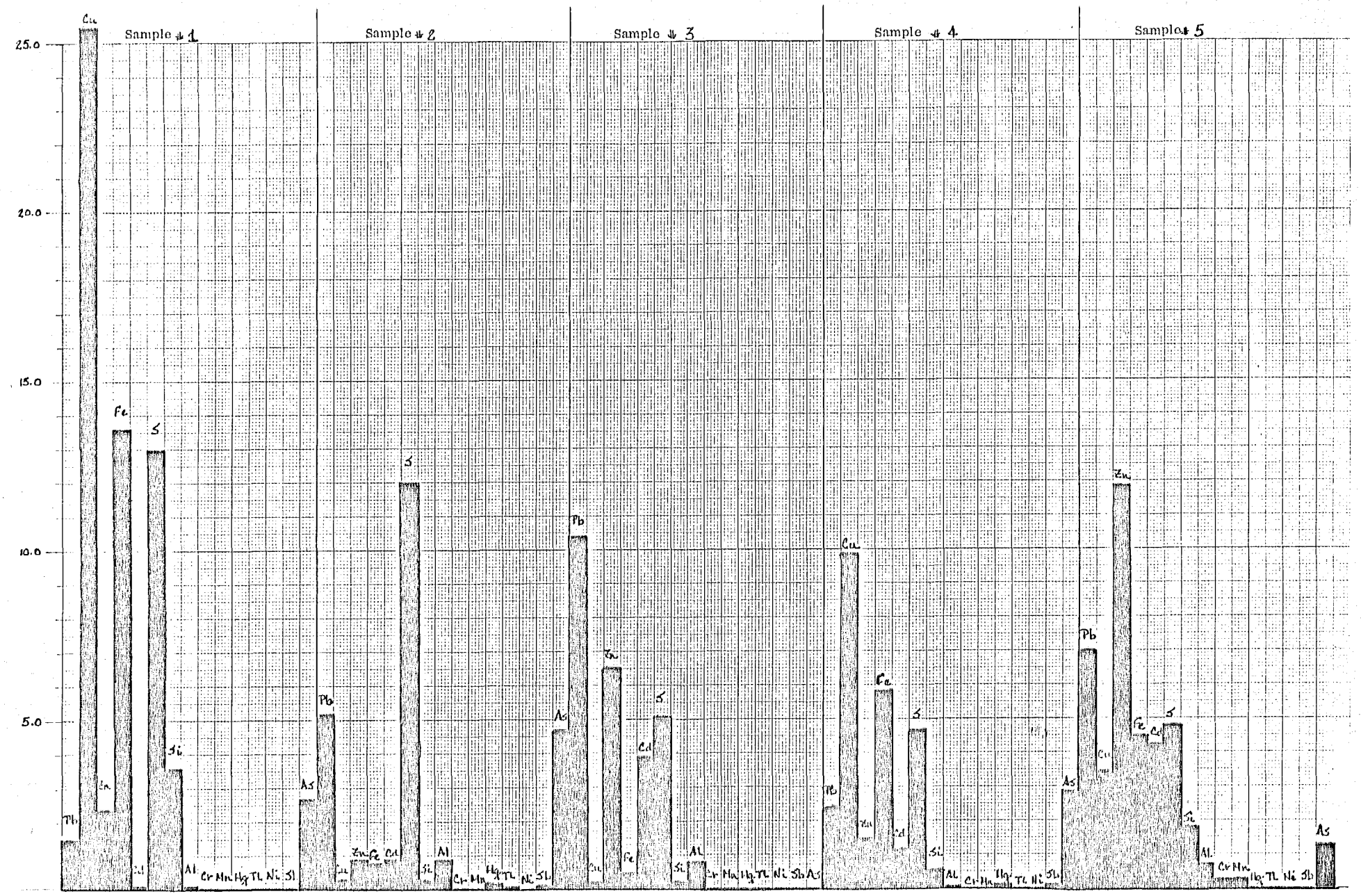


Figure 2. Bar graph showing the chemical composition of the five dust samples as determined by NIOSH

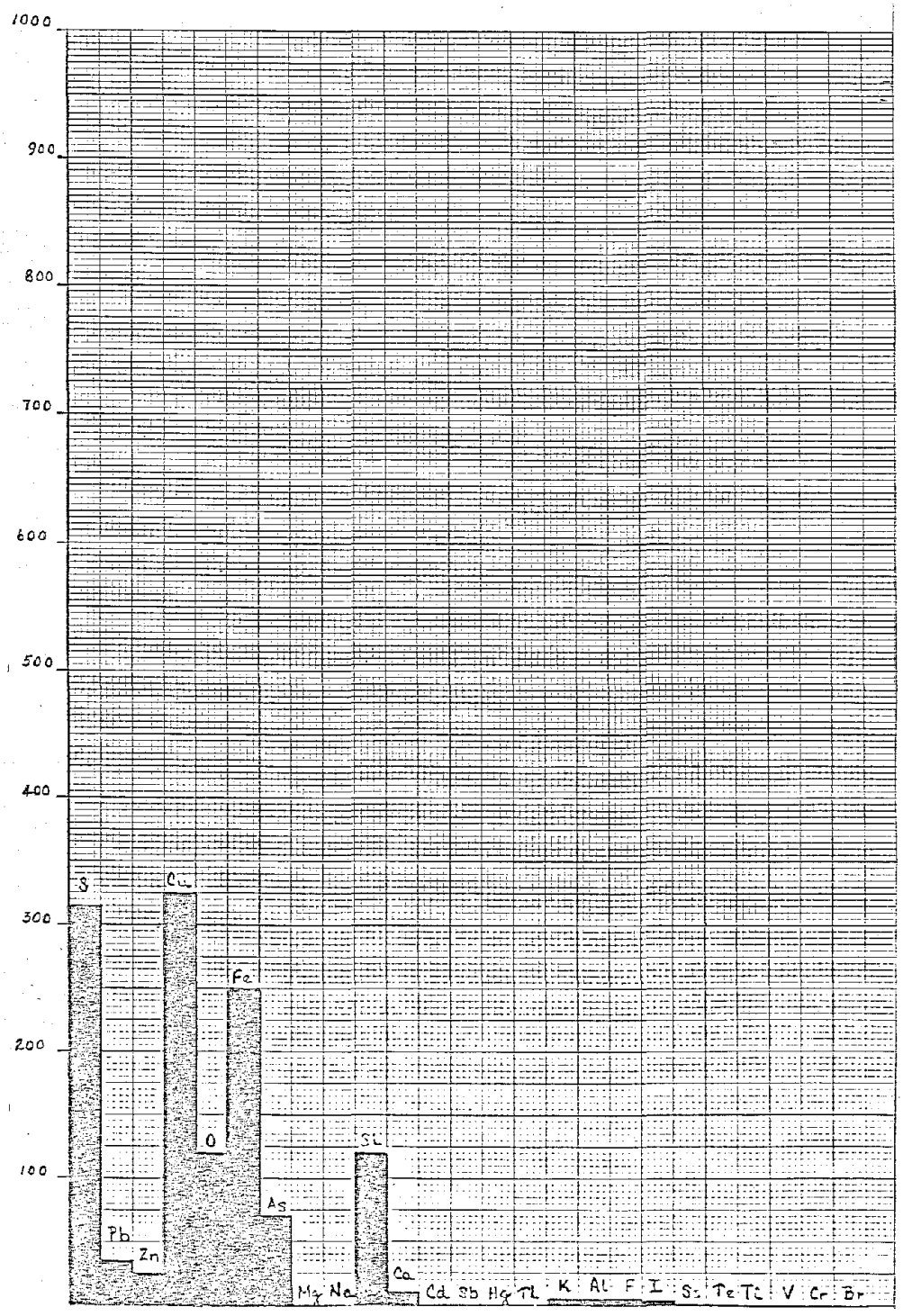


Figure 3. Bar graph showing elemental composition of sample 1-1 as determined by McCrone Associates by number of particles for major elements.

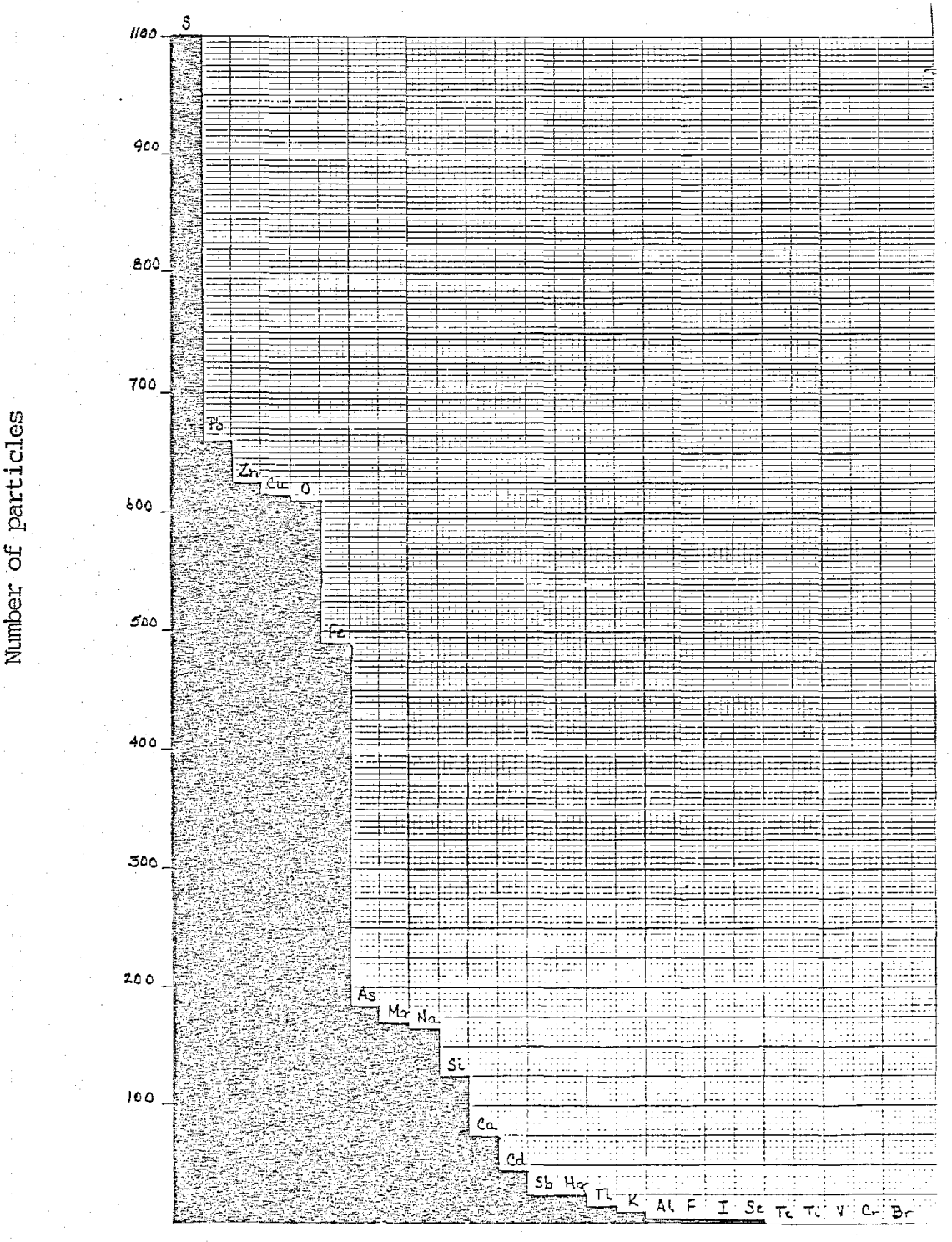


Figure 4. Bar graph showing elemental composition of sample 1-2 as determined by McCrone Associates by number of particles for major elements.

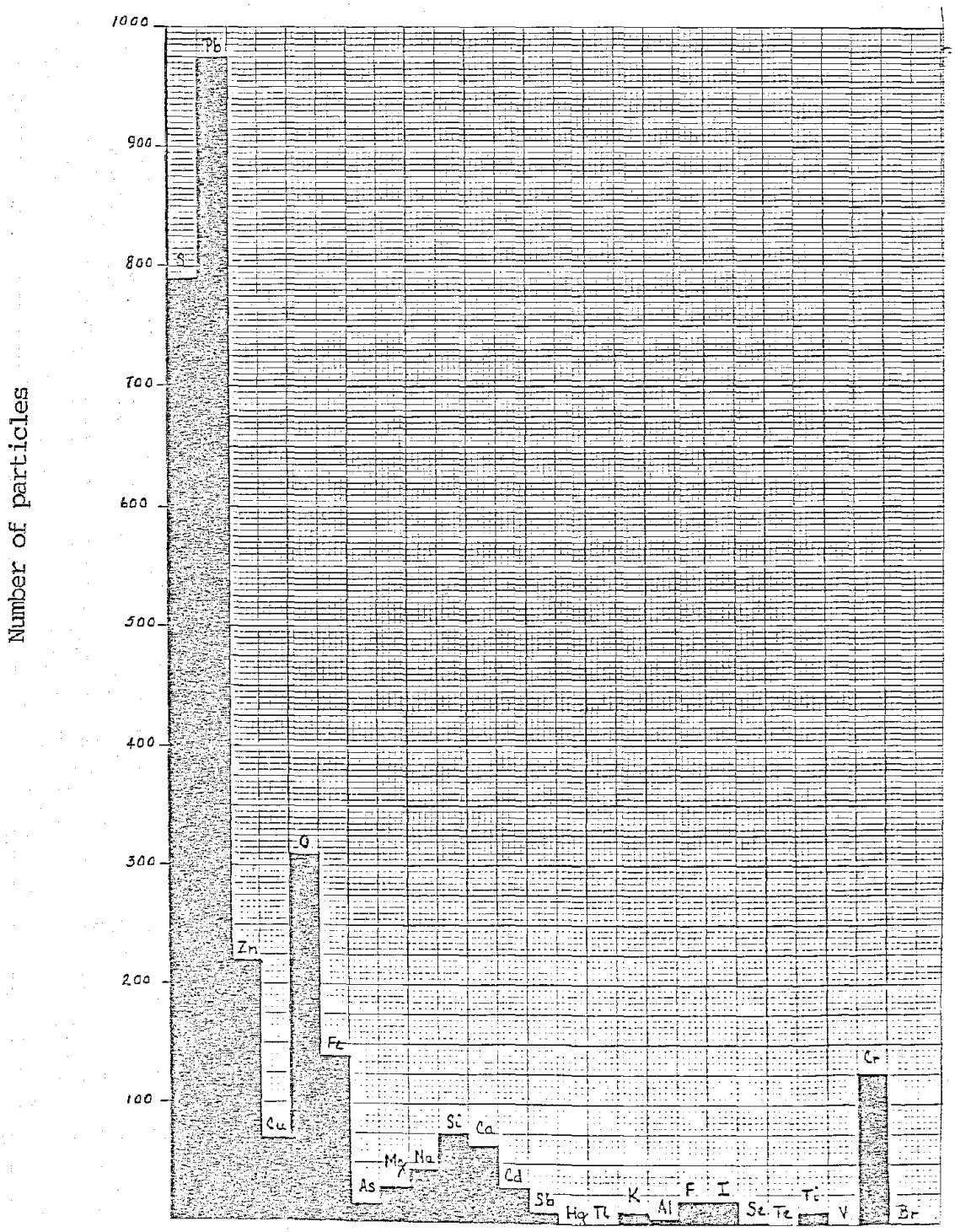


Figure 5. Bar graph showing elemental composition of sample 2 as determined by McCrone Associates by number of particles for major elements.

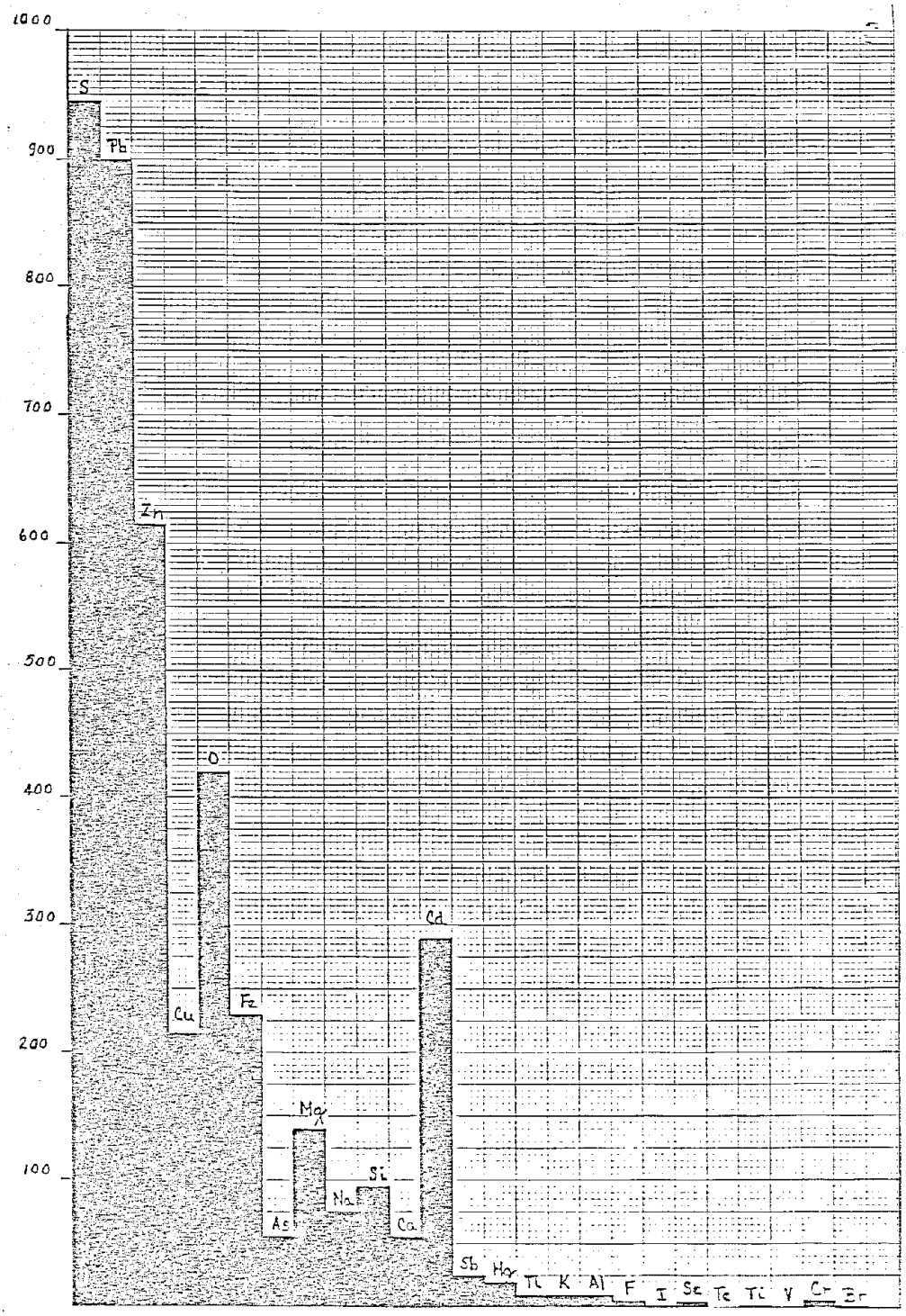


Figure 6. Bar graph showing elemental composition of sample 3 as determined by McCrone Associates by number of particles for major elements.

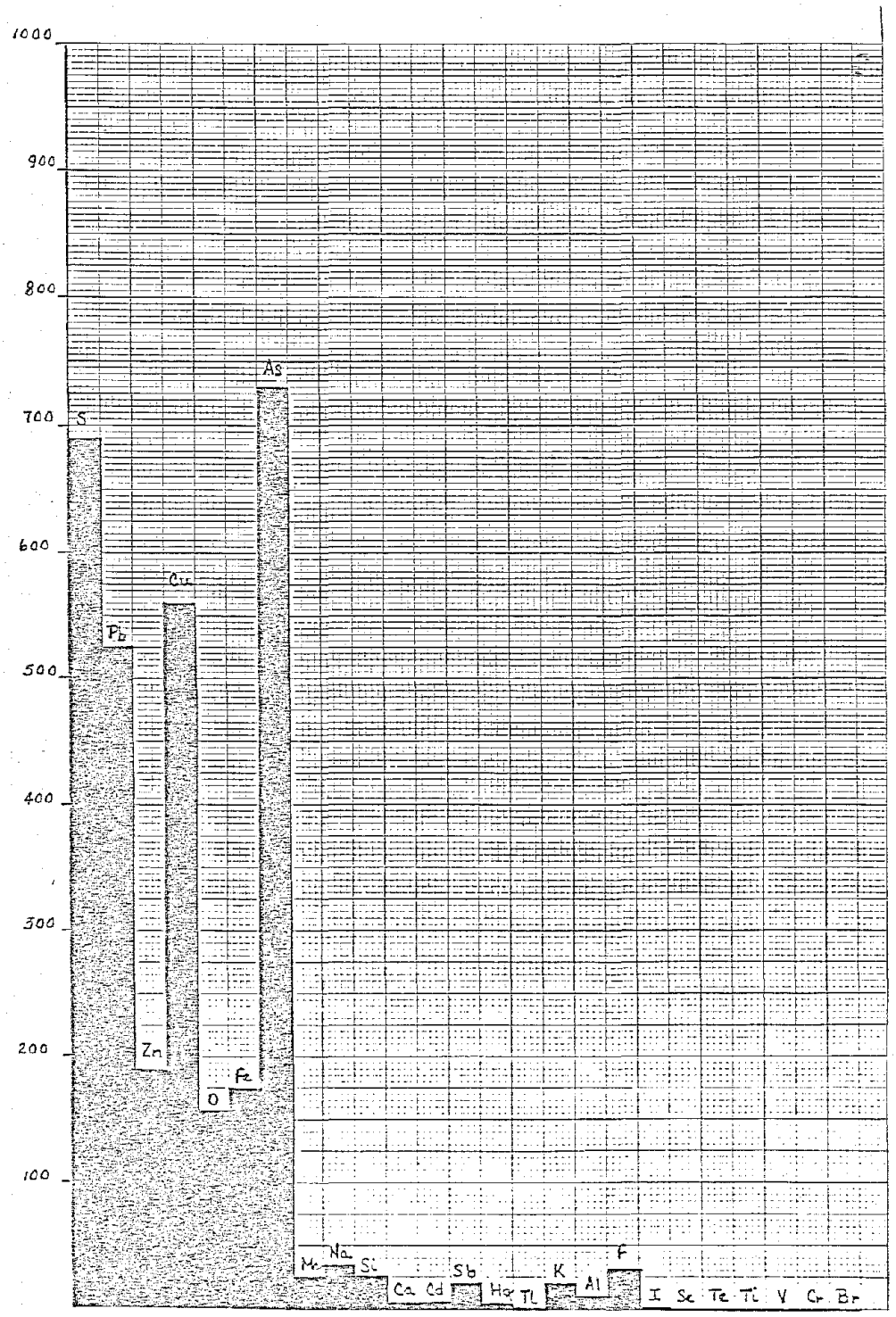


Figure 7. Bar graph showing elemental composition of sample 4 as determined by McCrone Associates by number of particles for major elements.

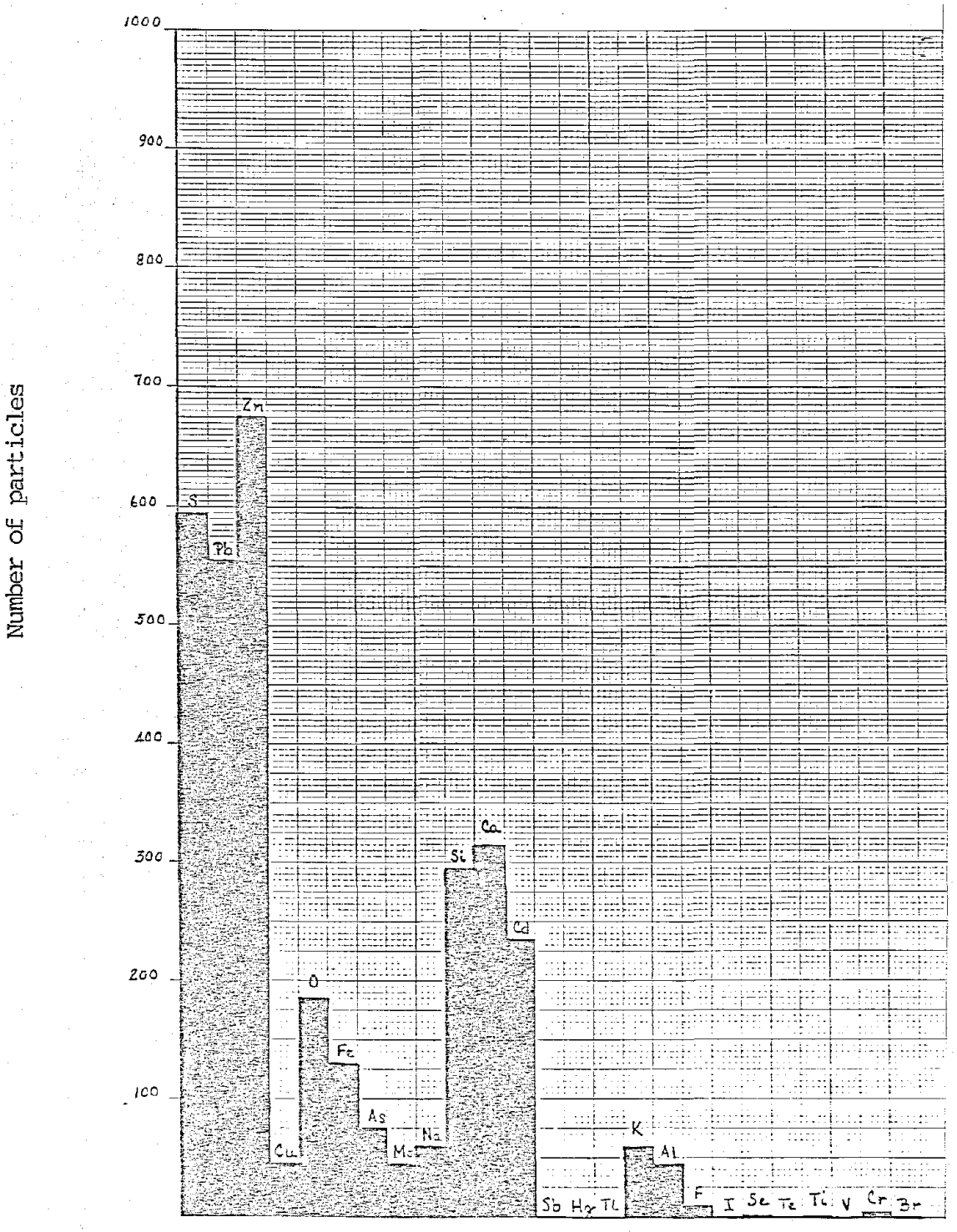


Figure 8. Bar graph showing elemental composition of sample 5 as determined by McCrone Associates by number of particles for major elements.

Appendix A

Computerized Tables Summarizing
the Analytical Results for the Five
Smelter Dust Samples

Note: Table numbers are detailed as follows:

Table A-1-1

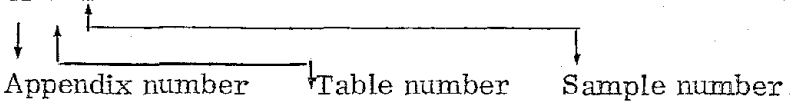

Table A-1-1, Sample 1

REPORT OUTPUT ON 2-14-78

DISTRIBUTION OF NIT SIZES FOR SAMPLE 1

| SIZE RANGE | NUMBER | PERCENT |
|---|---|---|
| 0 - 1 | 195. | 16.10 |
| 1 - 2 | 528. | 43.60 |
| 2 - 3 | 293. | 24.19 |
| 3 - 4 | 99. | 8.18 |
| 4 - 5 | 36. | 2.97 |
| 5 - 6 | 21. | 1.73 |
| 6 - 7 | 20. | 1.65 |
| 7 - 8 | 10. | 0.83 |
| OVER 8 | 9. | 0.74 |

NUMBER OF NITS FOUND = 1211

Table A-2-1, Sample 1

SUMMARY OF NORMALIZED MAJOR ELEMENTS
IN SAMPLE NUMBER 1

PARTICLE NUMBERS 1 THROUGH 1211

TOTAL OF ELEMENTS GREATER THAN 10%

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| K | 3 | SB | 0 | CA | 70 | TE | 0 | I | 2 |
| TI | 0 | V | 0 | CR | 0 | FE | 331 | CU | 325 |
| ZN | 442 | AS | 105 | SE | 0 | BR | 0 | AL | 6 |
| SI | 47 | HG | 1 | TL | 0 | S | 966 | PB | 429 |
| CD | 1 | O | 582 | F | 3 | NA | 48 | MG | 109 |

ELEMENTS BETWEEN 5% AND LESS THAN 10%

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| K | 9 | SB | 23 | CA | 6 | TE | 0 | I | 1 |
| TI | 0 | V | 0 | CR | 0 | FE | 161 | CU | 290 |
| ZN | 184 | AS | 82 | SE | 3 | BR | 0 | AL | 1 |
| SI | 76 | HG | 22 | TL | 13 | S | 100 | PB | 230 |
| CD | 47 | O | 26 | F | 1 | NA | 111 | MG | 59 |

Table A-2-1, cont'd, Sample 1

SUMMARY OF NORMALIZED MAJOR ELEMENTS
IN SAMPLE NUMBER N-1-1

PARTICLE NUMBERS 1 THROUGH 446

TOTAL OF ELEMENTS GREATER THAN 10%

| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| K | 3 | SB | 0 | CA | 4 | TE | 0 | I | 2 |
| TI | 0 | V | 0 | CR | 0 | FE | 221 | CU | 255 |
| ZN | 17 | AS | 63 | SE | 0 | BR | 0 | AL | 6 |
| SI | 47 | HG | 0 | TL | 0 | S | 246 | PB | 30 |
| CD | 0 | O | 119 | F | 3 | NA | 0 | MG | 0 |

ELEMENTS BETWEEN 5% AND LESS THAN 10%

| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| K | 2 | SB | 0 | CA | 6 | TE | 0 | I | 1 |
| TI | 0 | V | 0 | CR | 0 | FE | 27 | CU | 68 |
| ZN | 7 | AS | 9 | SE | 0 | BR | 0 | AL | 1 |
| SI | 74 | HG | 0 | TL | 0 | S | 70 | PB | 7 |
| CD | 0 | O | 0 | F | 1 | NA | 0 | MG | 0 |

Table A-3-1, Sample 1

SUMMARY OF NORMALIZED MAJOR ELEMENTS
IN SAMPLE NUMBER 1

PARTICLE NUMBERS 1 THROUGH 1211

ELEMENTS BETWEEN 75% AND 100%

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| K | 0 | SB | 0 | CA | 0 | TE | 0 | I | 0 |
| TI | 0 | V | 0 | CR | 0 | FE | 19 | CU | 14 |
| ZN | 0 | AS | 14 | SE | 0 | BR | 0 | AL | 0 |
| SI | 0 | HG | 0 | TL | 0 | S | 8 | PB | 0 |
| CD | 0 | O | 80 | F | 0 | NA | 0 | MG | 0 |

ELEMENTS BETWEEN 50% AND 75%

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| K | 0 | SB | 0 | CA | 5 | TE | 0 | I | 0 |
| TI | 0 | V | 0 | CR | 0 | FE | 76 | CU | 75 |
| ZN | 4 | AS | 29 | SE | 0 | BR | 0 | AL | 2 |
| SI | 0 | HG | 0 | TL | 0 | S | 39 | PB | 1 |
| CD | 0 | O | 27 | F | 0 | NA | 6 | MG | 0 |

ELEMENTS BETWEEN 30% AND 50%

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| K | 2 | SB | 0 | CA | 14 | TE | 0 | I | 1 |
| TI | 0 | V | 0 | CR | 0 | FE | 88 | CU | 84 |
| ZN | 10 | AS | 14 | SE | 0 | BR | 0 | AL | 1 |
| SI | 3 | HG | 0 | TL | 0 | S | 236 | PB | 24 |
| CD | 0 | O | 259 | F | 0 | NA | 19 | MG | 1 |

ELEMENTS BETWEEN 10% AND 30%

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| K | 1 | SB | 0 | CA | 51 | TE | 0 | I | 1 |
| TI | 0 | V | 0 | CR | 0 | FE | 148 | CU | 152 |
| ZN | 428 | AS | 48 | SE | 0 | BR | 0 | AL | 3 |
| SI | 44 | HG | 1 | TL | 0 | S | 683 | PB | 404 |
| CD | 1 | O | 216 | F | 3 | NA | 23 | MG | 108 |

ELEMENTS BETWEEN 5% AND 10%

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| K | 9 | SB | 23 | CA | 6 | TE | 0 | I | 1 |
| TI | 0 | V | 0 | CR | 0 | FE | 161 | CU | 290 |
| ZN | 184 | AS | 82 | SE | 3 | BR | 0 | AL | 1 |
| SI | 76 | HG | 22 | TL | 13 | S | 100 | PB | 230 |
| CD | 47 | O | 26 | F | 1 | NA | 111 | MG | 59 |

Table A-3-1 cont'd, Sample 1

SUMMARY OF NORMALIZED MAJOR ELEMENTS
IN SAMPLE NUMBER N-1-1

PARTICLE NUMBERS 1 THROUGH 446

ELEMENTS BETWEEN 75% AND 100%

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| K | 0 | SB | 0 | CA | 0 | TE | 0 | I | 0 |
| TI | 0 | V | 0 | CR | 0 | FE | 19 | CU | 14 |
| ZN | 0 | AS | 12 | SE | 0 | BR | 0 | AL | 0 |
| SI | 0 | HG | 0 | TL | 0 | S | 8 | PB | 0 |
| CD | 0 | O | 80 | F | 0 | NA | 0 | MG | 0 |

ELEMENTS BETWEEN 50% AND 75%

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| K | 0 | SB | 0 | CA | 1 | TE | 0 | I | 0 |
| TI | 0 | V | 0 | CR | 0 | FE | 76 | CU | 75 |
| ZN | 4 | AS | 14 | SE | 0 | BR | 0 | AL | 2 |
| SI | 0 | HG | 0 | TL | 0 | S | 8 | PB | 1 |
| CD | 0 | O | 23 | F | 0 | NA | 0 | MG | 0 |

ELEMENTS BETWEEN 30% AND 50%

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| K | 2 | SB | 0 | CA | 0 | TE | 0 | I | 1 |
| TI | 0 | V | 0 | CR | 0 | FE | 78 | CU | 70 |
| ZN | 4 | AS | 8 | SE | 0 | BR | 0 | AL | 1 |
| SI | 3 | HG | 0 | TL | 0 | S | 58 | PB | 2 |
| CD | 0 | O | 7 | F | 0 | NA | 0 | MG | 0 |

ELEMENTS BETWEEN 10% AND 30%

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| K | 1 | SB | 0 | CA | 3 | TE | 0 | I | 1 |
| TI | 0 | V | 0 | CR | 0 | FE | 48 | CU | 96 |
| ZN | 9 | AS | 29 | SE | 0 | BR | 0 | AL | 3 |
| SI | 44 | HG | 0 | TL | 0 | S | 172 | PB | 27 |
| CD | 0 | O | 9 | F | 3 | NA | 0 | MG | 0 |

ELEMENTS BETWEEN 5% AND 10%

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| K | 2 | SB | 0 | CA | 6 | TE | 0 | I | 1 |
| TI | 0 | V | 0 | CR | 0 | FE | 27 | CU | 68 |
| ZN | 7 | AS | 9 | SE | 0 | BR | 0 | AL | 1 |
| SI | 74 | HG | 0 | TL | 0 | S | 70 | PB | 7 |
| CD | 0 | O | 0 | F | 1 | NA | 0 | MG | 0 |

Table A-4-1, Sample 1

REPORT OUTPUT ON 2-14-78

TOTAL NUMBER OF MAJOR ELEMENTS FOR SAMPLE 1

| | | |
|---|---|---|
| S | = | 1066 |
| PB | = | 659 |
| ZN | = | 626 |
| CU | = | 615 |
| O | = | 608 |
| FE | = | 492 |
| AS | = | 187 |
| MG | = | 168 |
| NA | = | 159 |
| SI | = | 123 |
| CA | = | 76 |
| CD | = | 48 |
| SB | = | 23 |
| HG | = | 23 |
| TL | = | 13 |
| K | = | 12 |
| AL | = | 7 |
| F | = | 4 |
| I | = | 3 |
| SE | = | 3 |
| TE | = | 0 |
| TI | = | 0 |
| V | = | 0 |
| CR | = | 0 |
| BR | = | 0 |

NUMBER OF NITS FOUND = 1211

Table A-4-1, cont'd, Sample 1

REPORT OUTPUT ON 2-14-78

TOTAL NUMBER OF MAJOR ELEMENTS FOR SAMPLE N-1-1

| | | |
|---|---|---|
| CU | = | 323 |
| S | = | 316 |
| FE | = | 248 |
| SI | = | 121 |
| O | = | 119 |
| AS | = | 72 |
| PB | = | 37 |
| ZN | = | 24 |
| CA | = | 10 |
| AL | = | 7 |
| K | = | 5 |
| F | = | 4 |
| I | = | 3 |
| SB | = | 0 |
| TE | = | 0 |
| TI | = | 0 |
| V | = | 0 |
| CR | = | 0 |
| SE | = | 0 |
| BR | = | 0 |
| HG | = | 0 |
| TL | = | 0 |
| CD | = | 0 |
| NA | = | 0 |
| MG | = | 0 |

NUMBER OF NITS FOUND = 446

Table A-5-1, Sample 1

REPORT OUTPUT ON 2-14-78

AVERAGE PARTICLE SIZE OF MAJOR ELEMENTS FOR SAMPLE 1

| ELEMENT | AVERAGE SIZE | FREQUENCY OF OCCURENCE |
|---|---|---|
| AL | 3.09 | 7 |
| FE | 2.32 | 492 |
| AS | 2.22 | 187 |
| CU | 2.20 | 615 |
| PB | 2.14 | 659 |
| O | 2.12 | 608 |
| ZN | 2.10 | 626 |
| S | 2.09 | 1066 |
| MG | 2.09 | 168 |
| CD | 2.07 | 48 |
| CA | 2.03 | 76 |
| NA | 1.96 | 159 |
| SE | 1.90 | 3 |
| SB | 1.88 | 23 |
| SI | 1.84 | 123 |
| K | 1.69 | 12 |
| HG | 1.61 | 23 |
| F | 1.59 | 4 |
| TL | 1.36 | 13 |
| I | 1.29 | 3 |
| TE | 0.00 | 0 |
| TI | 0.00 | 0 |
| V | 0.00 | 0 |
| CR | 0.00 | 0 |
| BR | 0.00 | 0 |

NUMBER OF NITS FOUND = 1211

Table A-5-1, cont'd, Sample 1

REPORT OUTPUT ON 2-14-78

AVERAGE PARTICLE SIZE OF MAJOR ELEMENTS FOR SAMPLE N-1-1

| ELEMENT | AVERAGE SIZE | FREQUENCY OF OCCURENCE |
|---|---|---|
| AL | 3.09 | 7 |
| CA | 2.90 | 10 |
| AS | 2.74 | 72 |
| FE | 2.58 | 248 |
| PB | 2.58 | 37 |
| ZN | 2.50 | 24 |
| O | 2.32 | 119 |
| CU | 2.21 | 323 |
| S | 2.18 | 316 |
| SI | 1.83 | 121 |
| K | 1.62 | 5 |
| F | 1.59 | 4 |
| I | 1.29 | 3 |
| SB | 0.00 | 0 |
| TE | 0.00 | 0 |
| TI | 0.00 | 0 |
| V | 0.00 | 0 |
| CR | 0.00 | 0 |
| SE | 0.00 | 0 |
| BR | 0.00 | 0 |
| HG | 0.00 | 0 |
| TL | 0.00 | 0 |
| CD | 0.00 | 0 |
| NA | 0.00 | 0 |
| MG | 0.00 | 0 |

NUMBER OF HITS FOUND = 446

Table A-1-2, Sample 2

REPORT OUTPUT ON 1-25-78

DISTRIBUTION OF NIT SIZES FOR SAMPLE 2

| SIZE RANGE | NUMBER | PERCENT |
|---|---|---|
| 0 - 1 | 101. | 10.05 |
| 1 - 2 | 419. | 41.69 |
| 2 - 3 | 224. | 22.29 |
| 3 - 4 | 99. | 9.85 |
| 4 - 5 | 58. | 5.77 |
| 5 - 6 | 31. | 3.08 |
| 6 - 7 | 36. | 3.58 |
| 7 - 8 | 16. | 1.59 |
| OVER 8 | 21. | 2.09 |

NUMBER OF NITS FOUND = 1005

Table A-2-2, Sample 2

SUMMARY OF NORMALIZED MAJOR ELEMENTS
IN SAMPLE NUMBER 2

PARTICLE NUMBERS 1 THROUGH 1005

TOTAL OF ELEMENTS GREATER THAN 10%

| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| K | 8 | SB | 1 | CA | 30 | TE | 0 | I | 21 |
| TI | 2 | V | 0 | CR | 62 | FE | 94 | CU | 23 |
| ZN | 175 | AS | 10 | SE | 0 | BR | 0 | AL | 3 |
| SI | 32 | HG | 0 | TL | 0 | S | 409 | PB | 908 |
| CD | 5 | O | 301 | F | 17 | NA | 2 | MG | 7 |

ELEMENTS BETWEEN 5% AND LESS THAN 10%

| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| K | 1 | SB | 11 | CA | 36 | TE | 0 | I | 1 |
| TI | 9 | V | 0 | CR | 63 | FE | 45 | CU | 48 |
| ZN | 45 | AS | 6 | SE | 1 | BR | 0 | AL | 0 |
| SI | 41 | HG | 0 | TL | 0 | S | 381 | PB | 68 |
| CD | 25 | O | 7 | F | 5 | NA | 41 | MG | 22 |

Table A-3-2, Sample 2

SUMMARY OF NORMALIZED MAJOR ELEMENTS
IN SAMPLE NUMBER 2

PARTICLE NUMBERS 1 THROUGH 1005

ELEMENTS BETWEEN 75% AND 100%

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| K | 1 | SB | 0 | CA | 0 | TE | 0 | I | 0 |
| TI | 0 | V | 0 | CR | 3 | FE | 1 | CU | 0 |
| ZN | 0 | AS | 0 | SE | 0 | BR | 0 | AL | 0 |
| SI | 1 | HG | 0 | TL | 0 | S | 7 | PB | 451 |
| CD | 0 | O | 3 | F | 2 | NA | 0 | MG | 0 |

ELEMENTS BETWEEN 50% AND 75%

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| K | 2 | SB | 0 | CA | 0 | TE | 0 | I | 0 |
| TI | 0 | V | 0 | CR | 1 | FE | 5 | CU | 0 |
| ZN | 0 | AS | 1 | SE | 0 | BR | 0 | AL | 1 |
| SI | 1 | HG | 0 | TL | 0 | S | 8 | PB | 187 |
| CD | 0 | O | 23 | F | 2 | NA | 0 | MG | 0 |

ELEMENTS BETWEEN 30% AND 50%

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| K | 2 | SB | 0 | CA | 0 | TE | 0 | I | 4 |
| TI | 0 | V | 0 | CR | 4 | FE | 24 | CU | 7 |
| ZN | 15 | AS | 4 | SE | 0 | BR | 0 | AL | 0 |
| SI | 0 | HG | 0 | TL | 0 | S | 18 | PB | 104 |
| CD | 0 | O | 126 | F | 3 | NA | 0 | MG | 0 |

ELEMENTS BETWEEN 10% AND 30%

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| K | 3 | SB | 1 | CA | 30 | TE | 0 | I | 17 |
| TI | 2 | V | 0 | CR | 54 | FE | 64 | CU | 16 |
| ZN | 160 | AS | 5 | SE | 0 | BR | 0 | AL | 2 |
| SI | 30 | HG | 0 | TL | 0 | S | 376 | PB | 166 |
| CD | 5 | O | 149 | F | 10 | NA | 2 | MG | 7 |

ELEMENTS BETWEEN 5% AND 10%

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| K | 1 | SB | 11 | CA | 36 | TE | 0 | I | 1 |
| TI | 9 | V | 0 | CR | 63 | FE | 45 | CU | 48 |
| ZN | 45 | AS | 6 | SE | 1 | BR | 0 | AL | 0 |
| SI | 41 | HG | 0 | TL | 0 | S | 381 | PB | 68 |
| CD | 25 | O | 7 | F | 5 | NA | 41 | MG | 22 |

Table A-4-2, Sample 2

REPORT OUTPUT ON 1-25-78

TOTAL NUMBER OF MAJOR ELEMENTS FOR SAMPLE 2

| | | |
|---|---|---|
| PB | = | 976 |
| S | = | 790 |
| O | = | 308 |
| ZN | = | 220 |
| FE | = | 139 |
| CR | = | 125 |
| SI | = | 73 |
| CU | = | 71 |
| CA | = | 66 |
| NA | = | 43 |
| CD | = | 30 |
| MG | = | 29 |
| I | = | 22 |
| F | = | 22 |
| AS | = | 16 |
| SB | = | 12 |
| TI | = | 11 |
| K | = | 9 |
| AL | = | 3 |
| SE | = | 1 |
| TE | = | 0 |
| V | = | 0 |
| BR | = | 0 |
| HG | = | 0 |
| TL | = | 0 |

NUMBER OF NITS FOUND = 1005

Table A-5-2, Sample 2

REPORT OUTPUT ON 1-24-78

AVERAGE PARTICLE SIZE OF MAJOR ELEMENTS FOR SAMPLE 2

| ELEMENT | AVERAGE SIZE | FREQUENCY OF OCCURENCE |
|---|---|---|
| I | 3.62 | 22 |
| K | 3.34 | 9 |
| F | 3.06 | 22 |
| AL | 2.99 | 3 |
| AS | 2.79 | 16 |
| FE | 2.60 | 139 |
| PB | 2.54 | 976 |
| TI | 2.53 | 11 |
| S | 2.41 | 790 |
| CR | 2.41 | 125 |
| SI | 2.36 | 73 |
| O | 2.25 | 308 |
| NA | 2.14 | 43 |
| ZN | 2.11 | 220 |
| CA | 2.09 | 66 |
| SB | 2.08 | 12 |
| CU | 2.04 | 71 |
| MG | 2.02 | 29 |
| CD | 1.97 | 30 |
| SE | 1.55 | 1 |
| TE | 0.00 | 0 |
| V | 0.00 | 0 |
| BR | 0.00 | 0 |
| HG | 0.00 | 0 |
| TL | 0.00 | 0 |

NUMBER OF HITS FOUND = 1005

Table A-1-3, Sample 3

REPORT OUTPUT ON 2- 7-78

DISTRIBUTION OF NIT SIZES FOR SAMPLE 3

| SIZE RANGE | NUMBER | PERCENT |
|---|---|---|
| 0 - 1 | 174. | 15.06 |
| 1 - 2 | 484. | 41.90 |
| 2 - 3 | 237. | 20.52 |
| 3 - 4 | 112. | 9.70 |
| 4 - 5 | 50. | 4.33 |
| 5 - 6 | 39. | 3.38 |
| 6 - 7 | 28. | 2.42 |
| 7 - 8 | 23. | 1.99 |
| OVER 8 | 8. | 0.69 |

NUMBER OF NITS FOUND = 1155

Table A-2-3, Sample 3

SUMMARY OF NORMALIZED MAJOR ELEMENTS
IN SAMPLE NUMBER 3

PARTICLE NUMBERS 1 THROUGH 1155

TOTAL OF ELEMENTS GREATER THAN 10%

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| K | 7 | SB | 11 | CA | 53 | TE | 0 | I | 1 |
| TI | 1 | V | 0 | CR | 3 | FE | 112 | CU | 82 |
| ZN | 477 | AS | 17 | SE | 0 | BR | 0 | AL | 4 |
| SI | 35 | HG | 1 | TL | 0 | S | 784 | PB | 726 |
| CD | 174 | O | 397 | F | 6 | NA | 1 | MG | 95 |

ELEMENTS BETWEEN 5% AND LESS THAN 10%

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| K | 4 | SB | 14 | CA | 2 | TE | 0 | I | 0 |
| TI | 0 | V | 0 | CR | 2 | FE | 116 | CU | 132 |
| ZN | 136 | AS | 40 | SE | 4 | BR | 0 | AL | 8 |
| SI | 62 | HG | 19 | TL | 8 | S | 162 | PB | 176 |
| CD | 115 | O | 24 | F | 0 | NA | 75 | MG | 45 |

Table A-3-3, Sample 3

SUMMARY OF NORMALIZED MAJOR ELEMENTS
IN SAMPLE NUMBER 3

PARTICLE NUMBERS 1 THROUGH 1155

ELEMENTS BETWEEN 75% AND 100%

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| K | 1 | SB | 4 | CA | 1 | TE | 0 | I | 0 |
| TI | 0 | V | 0 | CR | 0 | FE | 13 | CU | 1 |
| ZN | 30 | AS | 7 | SE | 0 | BR | 0 | AL | 0 |
| SI | 1 | HG | 0 | TL | 0 | S | 13 | PB | 152 |
| CD | 12 | O | 47 | F | 4 | NA | 0 | MG | 3 |

ELEMENTS BETWEEN 50% AND 75%

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| K | 1 | SB | 2 | CA | 8 | TE | 0 | I | 0 |
| TI | 0 | V | 0 | CR | 1 | FE | 6 | CU | 2 |
| ZN | 41 | AS | 3 | SE | 0 | BR | 0 | AL | 2 |
| SI | 3 | HG | 0 | TL | 0 | S | 13 | PB | 183 |
| CD | 4 | O | 22 | F | 1 | NA | 0 | MG | 0 |

ELEMENTS BETWEEN 30% AND 50%

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| K | 1 | SB | 1 | CA | 36 | TE | 0 | I | 1 |
| TI | 0 | V | 0 | CR | 1 | FE | 9 | CU | 1 |
| ZN | 80 | AS | 6 | SE | 0 | BR | 0 | AL | 0 |
| SI | 2 | HG | 0 | TL | 0 | S | 145 | PB | 109 |
| CD | 6 | O | 190 | F | 1 | NA | 0 | MG | 3 |

ELEMENTS BETWEEN 10% AND 30%

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| K | 4 | SB | 4 | CA | 8 | TE | 0 | I | 0 |
| TI | 1 | V | 0 | CR | 1 | FE | 84 | CU | 78 |
| ZN | 326 | AS | 1 | SE | 0 | BR | 0 | AL | 2 |
| SI | 29 | HG | 1 | TL | 0 | S | 613 | PB | 282 |
| CD | 152 | O | 138 | F | 0 | NA | 1 | MG | 89 |

ELEMENTS BETWEEN 5% AND 10%

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| K | 4 | SB | 14 | CA | 2 | TE | 0 | I | 0 |
| TI | 0 | V | 0 | CR | 2 | FE | 116 | CU | 132 |
| ZN | 136 | AS | 40 | SE | 4 | BR | 0 | AL | 8 |
| SI | 62 | HG | 19 | TL | 8 | S | 162 | PB | 176 |
| CD | 115 | O | 24 | F | 0 | NA | 75 | MG | 45 |

Table A-4-3, Sample 3

REPORT OUTPUT ON 2- 7-78

TOTAL NUMBER OF MAJOR ELEMENTS FOR SAMPLE 3

| | | |
|---|---|---|
| S | = | 946 |
| PB | = | 902 |
| ZN | = | 613 |
| O | = | 421 |
| CD | = | 289 |
| FE | = | 228 |
| CU | = | 214 |
| MG | = | 140 |
| SI | = | 97 |
| NA | = | 76 |
| AS | = | 57 |
| CA | = | 55 |
| SB | = | 25 |
| HG | = | 20 |
| AL | = | 12 |
| K | = | 11 |
| TL | = | 8 |
| F | = | 6 |
| CR | = | 5 |
| SE | = | 4 |
| I | = | 1 |
| TI | = | 1 |
| TE | = | 0 |
| V | = | 0 |
| BR | = | 0 |

NUMBER OF NITS FOUND = 1155

Table A-5-3, Sample 3

REPORT OUTPUT ON 2- 7-78

AVERAGE PARTICLE SIZE OF MAJOR ELEMENTS FOR SAMPLE 3

| ELEMENT | AVERAGE SIZE | FREQUENCY OF OCCURENCE |
|---|---|---|
| CR | 6.26 | 5 |
| F | 4.97 | 6 |
| AL | 2.78 | 12 |
| AS | 2.45 | 57 |
| SI | 2.38 | 97 |
| SB | 2.34 | 25 |
| K | 2.32 | 11 |
| PB | 2.30 | 902 |
| S | 2.22 | 946 |
| CD | 2.19 | 289 |
| MG | 2.18 | 140 |
| O | 2.17 | 421 |
| FE | 2.16 | 228 |
| ZN | 2.16 | 613 |
| CU | 2.12 | 214 |
| NA | 2.02 | 76 |
| CA | 1.91 | 55 |
| SE | 1.74 | 4 |
| HG | 1.65 | 20 |
| TL | 1.48 | 8 |
| TI | 1.26 | 1 |
| I | 1.03 | 1 |
| TE | 0.00 | 0 |
| V | 0.00 | 0 |
| BR | 0.00 | 0 |

NUMBER OF HITS FOUND = 1155

Table A-1-4, Sample 4

REPORT OUTPUT ON 2-15-78

DISTRIBUTION OF NIT SIZES FOR SAMPLE 4

| SIZE RANGE | NUMBER | PERCENT |
|---|---|---|
| 0 - 1 | 120. | 10.63 |
| 1 - 2 | 592. | 52.44 |
| 2 - 3 | 181. | 16.03 |
| 3 - 4 | 99. | 8.77 |
| 4 - 5 | 48. | 4.25 |
| 5 - 6 | 27. | 2.39 |
| 6 - 7 | 24. | 2.13 |
| 7 - 8 | 20. | 1.77 |
| OVER 8 | 18. | 1.59 |

NUMBER OF NITS FOUND = 1129

Table A-2-4, Sample 4

SUMMARY OF NORMALIZED MAJOR ELEMENTS
IN SAMPLE NUMBER 4

PARTICLE NUMBERS 1 THROUGH 1129

TOTAL OF ELEMENTS GREATER THAN 10%

| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| K | 2 | SB | 15 | CA | 1 | TE | 0 | I | 0 |
| TI | 0 | V | 0 | CR | 0 | FE | 131 | CU | 345 |
| ZN | 132 | AS | 718 | SE | 0 | BR | 0 | AL | 8 |
| SI | 19 | HG | 0 | TL | 0 | S | 469 | PB | 312 |
| CD | 0 | O | 143 | F | 29 | NA | 4 | MG | 11 |

ELEMENTS BETWEEN 5% AND LESS THAN 10%

| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| K | 18 | SB | 5 | CA | 3 | TE | 0 | I | 1 |
| TI | 0 | V | 0 | CR | 0 | FE | 44 | CU | 214 |
| ZN | 58 | AS | 10 | SE | 0 | BR | 0 | AL | 1 |
| SI | 7 | HG | 3 | TL | 1 | S | 199 | PB | 211 |
| CD | 5 | O | 10 | F | 2 | NA | 33 | MG | 15 |

Table A-3-4, Sample 4

SUMMARY OF NORMALIZED MAJOR ELEMENTS
IN SAMPLE NUMBER 4

PARTICLE NUMBERS 1 THROUGH 1129

ELEMENTS BETWEEN 75% AND 100%

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| K | 0 | SB | 0 | CA | 0 | TE | 0 | I | 0 |
| TI | 0 | V | 0 | CR | 0 | FE | 6 | CU | 10 |
| ZN | 15 | AS | 325 | SE | 0 | BR | 0 | AL | 0 |
| SI | 3 | HG | 0 | TL | 0 | S | 58 | PB | 5 |
| CD | 0 | O | 8 | F | 1 | NA | 0 | MG | 0 |

ELEMENTS BETWEEN 50% AND 75%

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| K | 0 | SB | 0 | CA | 0 | TE | 0 | I | 0 |
| TI | 0 | V | 0 | CR | 0 | FE | 29 | CU | 40 |
| ZN | 27 | AS | 222 | SE | 0 | BR | 0 | AL | 1 |
| SI | 0 | HG | 0 | TL | 0 | S | 35 | PB | 64 |
| CD | 0 | O | 14 | F | 3 | NA | 0 | MG | 0 |

ELEMENTS BETWEEN 30% AND 50%

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| K | 0 | SB | 9 | CA | 1 | TE | 0 | I | 0 |
| TI | 0 | V | 0 | CR | 0 | FE | 22 | CU | 65 |
| ZN | 25 | AS | 110 | SE | 0 | BR | 0 | AL | 1 |
| SI | 7 | HG | 0 | TL | 0 | S | 109 | PB | 76 |
| CD | 0 | O | 38 | F | 3 | NA | 0 | MG | 0 |

ELEMENTS BETWEEN 10% AND 30%

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| K | 2 | SB | 6 | CA | 0 | TE | 0 | I | 0 |
| TI | 0 | V | 0 | CR | 0 | FE | 74 | CU | 230 |
| ZN | 65 | AS | 61 | SE | 0 | BR | 0 | AL | 6 |
| SI | 9 | HG | 0 | TL | 0 | S | 267 | PB | 167 |
| CD | 0 | O | 83 | F | 22 | NA | 4 | MG | 11 |

ELEMENTS BETWEEN 5% AND 10%

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| K | 18 | SB | 5 | CA | 3 | TE | 0 | I | 1 |
| TI | 0 | V | 0 | CR | 0 | FE | 44 | CU | 214 |
| ZN | 58 | AS | 10 | SE | 0 | BR | 0 | AL | 1 |
| SI | 7 | HG | 3 | TL | 1 | S | 199 | PB | 211 |
| CD | 5 | O | 10 | F | 2 | NA | 33 | MG | 15 |

Table A-4-4, Sample 4

REPORT OUTPUT ON 2-15-78

TOTAL NUMBER OF MAJOR ELEMENTS FOR SAMPLE 4

| | | |
|---|---|---|
| AS | = | 728 |
| S | = | 668 |
| CU | = | 559 |
| PB | = | 523 |
| ZN | = | 190 |
| FE | = | 175 |
| O | = | 153 |
| NA | = | 37 |
| F | = | 31 |
| SI | = | 26 |
| MG | = | 26 |
| K | = | 20 |
| SB | = | 20 |
| AL | = | 9 |
| CD | = | 5 |
| CA | = | 4 |
| HG | = | 3 |
| I | = | 1 |
| TL | = | 1 |
| TE | = | 0 |
| TI | = | 0 |
| V | = | 0 |
| CR | = | 0 |
| SE | = | 0 |
| BR | = | 0 |

NUMBER OF NITS FOUND = 1129

Table A-5-4, Sample 4

REPORT OUTPUT ON 2-15-78

AVERAGE PARTICLE SIZE OF MAJOR ELEMENTS FOR SAMPLE 4

| ELEMENT | AVERAGE SIZE | FREQUENCY OF OCCURENCE |
|---|---|---|
| K | 3.09 | 20 |
| FE | 2.88 | 175 |
| SI | 2.69 | 26 |
| AS | 2.39 | 728 |
| CA | 2.25 | 4 |
| O | 2.23 | 153 |
| S | 2.22 | 668 |
| CU | 2.15 | 559 |
| PB | 2.14 | 523 |
| F | 2.09 | 31 |
| AL | 2.09 | 9 |
| ZN | 2.06 | 190 |
| NA | 2.01 | 37 |
| MG | 1.94 | 26 |
| SB | 1.90 | 20 |
| TL | 1.86 | 1 |
| HG | 1.83 | 3 |
| CD | 1.48 | 5 |
| I | 1.35 | 1 |
| TE | 0.00 | 0 |
| TI | 0.00 | 0 |
| V | 0.00 | 0 |
| CR | 0.00 | 0 |
| SE | 0.00 | 0 |
| BR | 0.00 | 0 |

NUMBER OF HITS FOUND = 1129

Table A-1-5, Sample 5

REPORT OUTPUT ON 1-30-78

TOTAL NUMBER OF MAJOR ELEMENTS FOR SAMPLE 5

| | | |
|---|---|---|
| ZN | = | 674 |
| S | = | 593 |
| PB | = | 554 |
| CA | = | 314 |
| SI | = | 295 |
| CD | = | 233 |
| O | = | 183 |
| FE | = | 129 |
| AS | = | 76 |
| NA | = | 60 |
| K | = | 58 |
| AL | = | 47 |
| CU | = | 46 |
| MG | = | 44 |
| F | = | 9 |
| CR | = | 4 |
| SB | = | 1 |
| TI | = | 1 |
| SE | = | 1 |
| BR | = | 1 |
| TE | = | 0 |
| I | = | 0 |
| V | = | 0 |
| HG | = | 0 |
| TL | = | 0 |

NUMBER OF NITS FOUND = 1081

Table A-2-5, Sample 5

SUMMARY OF NORMALIZED MAJOR ELEMENTS
IN SAMPLE NUMBER 5

PARTICLE NUMBERS 1 THROUGH 1081

TOTAL OF ELEMENTS GREATER THAN 10%

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| K | 54 | SB | 1 | CA | 211 | TE | 0 | I | 0 |
| TI | 1 | V | 0 | CR | 2 | FE | 96 | CU | 28 |
| ZN | 601 | AS | 61 | SE | 1 | BR | 1 | AL | 37 |
| SI | 140 | HG | 0 | TL | 0 | S | 330 | PB | 471 |
| CD | 92 | O | 181 | F | 8 | NA | 18 | MG | 12 |

ELEMENTS BETWEEN 5% AND LESS THAN 10%

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| K | 4 | SB | 0 | CA | 103 | TE | 0 | I | 0 |
| TI | 0 | V | 0 | CR | 2 | FE | 33 | CU | 18 |
| ZN | 73 | AS | 15 | SE | 0 | BR | 0 | AL | 10 |
| SI | 155 | HG | 0 | TL | 0 | S | 263 | PB | 83 |
| CD | 141 | O | 2 | F | 1 | NA | 42 | MG | 32 |

Table A-3-5, Sample 5

SUMMARY OF NORMALIZED MAJOR ELEMENTS
IN SAMPLE NUMBER 5

PARTICLE NUMBERS 1 THROUGH 1081

ELEMENTS BETWEEN 75% AND 100%

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| K | 0 | SB | 0 | CA | 19 | TE | 0 | I | 0 |
| TI | 0 | V | 0 | CR | 0 | FE | 10 | CU | 0 |
| ZN | 129 | AS | 5 | SE | 0 | BR | 0 | AL | 1 |
| SI | 8 | HG | 0 | TL | 0 | S | 91 | PB | 58 |
| CD | 1 | O | 38 | F | 1 | NA | 0 | MG | 3 |

ELEMENTS BETWEEN 50% AND 75%

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| K | 24 | SB | 0 | CA | 58 | TE | 0 | I | 0 |
| TI | 0 | V | 0 | CR | 0 | FE | 10 | CU | 1 |
| ZN | 196 | AS | 6 | SE | 0 | BR | 0 | AL | 3 |
| SI | 4 | HG | 0 | TL | 0 | S | 16 | PB | 91 |
| CD | 2 | O | 32 | F | 2 | NA | 1 | MG | 2 |

ELEMENTS BETWEEN 30% AND 50%

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| K | 15 | SB | 0 | CA | 44 | TE | 0 | I | 0 |
| TI | 0 | V | 0 | CR | 0 | FE | 24 | CU | 5 |
| ZN | 131 | AS | 11 | SE | 0 | BR | 0 | AL | 13 |
| SI | 15 | HG | 0 | TL | 0 | S | 13 | PB | 89 |
| CD | 8 | O | 67 | F | 0 | NA | 1 | MG | 1 |

ELEMENTS BETWEEN 10% AND 30%

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| K | 15 | SB | 1 | CA | 90 | TE | 0 | I | 0 |
| TI | 1 | V | 0 | CR | 2 | FE | 52 | CU | 22 |
| ZN | 145 | AS | 39 | SE | 1 | BR | 1 | AL | 20 |
| SI | 113 | HG | 0 | TL | 0 | S | 210 | PB | 233 |
| CD | 81 | O | 44 | F | 5 | NA | 16 | MG | 6 |

ELEMENTS BETWEEN 5% AND 10%

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| K | 4 | SB | 0 | CA | 103 | TE | 0 | I | 0 |
| TI | 0 | V | 0 | CR | 2 | FE | 33 | CU | 18 |
| ZN | 73 | AS | 15 | SE | 0 | BR | 0 | AL | 10 |
| SI | 155 | HG | 0 | TL | 0 | S | 263 | PB | 83 |
| CD | 141 | O | 2 | F | 1 | NA | 42 | MG | 32 |

Table A-4-5, Sample 5

REPORT OUTPUT ON 1-30-78

TOTAL NUMBER OF MAJOR ELEMENTS FOR SAMPLE 5

| | | |
|---|---|---|
| ZN | = | 674 |
| S | = | 593 |
| PB | = | 554 |
| CA | = | 314 |
| SI | = | 295 |
| CD | = | 233 |
| O | = | 183 |
| FE | = | 129 |
| AS | = | 76 |
| NA | = | 60 |
| K | = | 58 |
| AL | = | 47 |
| CU | = | 46 |
| MG | = | 44 |
| F | = | 9 |
| CR | = | 4 |
| SB | = | 1 |
| TI | = | 1 |
| SE | = | 1 |
| BR | = | 1 |
| TE | = | 0 |
| I | = | 0 |
| V | = | 0 |
| HG | = | 0 |
| TL | = | 0 |

NUMBER OF NITS FOUND = 1081

Table A-5-5, Sample 5

REPORT OUTPUT ON 1-30-78

AVERAGE PARTICLE SIZE OF MAJOR ELEMENTS FOR SAMPLE 5

| ELEMENT | AVERAGE SIZE | FREQUENCY OF OCCURENCE |
|---|---|---|
| SE | 6.23 | 1 |
| SB | 5.83 | 1 |
| BR | 4.21 | 1 |
| PB | 2.97 | 554 |
| AS | 2.90 | 76 |
| FE | 2.84 | 129 |
| SI | 2.80 | 295 |
| S | 2.80 | 593 |
| ZN | 2.77 | 674 |
| CA | 2.60 | 314 |
| CU | 2.58 | 46 |
| AL | 2.53 | 47 |
| CR | 2.43 | 4 |
| K | 2.41 | 58 |
| MG | 2.36 | 44 |
| F | 2.36 | 9 |
| CD | 2.29 | 233 |
| O | 2.29 | 183 |
| NA | 2.26 | 60 |
| TI | 1.27 | 1 |
| TE | 0.00 | 0 |
| I | 0.00 | 0 |
| V | 0.00 | 0 |
| HG | 0.00 | 0 |
| TL | 0.00 | 0 |

NUMBER OF HITS FOUND = 1081