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Report Title: Characterization of High Density Sludge from Britannia Beach,
British Columbia

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EXECUTIVE SUMMARY

The Britannia Mine in British Columbia was in operation from 1902 to 1974. Acid Rock Drainage (ARD) from the orphaned mine has been flowing into the receiving environment since long before closure in 1974. Pilot scale tests of high density sludge (HDS) water treatment were carried out in February – March 2002 on site. Canadian Environmental and Metallurgical Inc. (CEMI) conducted the testing on site and produced high density metal-hydroxide sludge material. CANMET Mining and Mineral Sciences Laboratories (MMSL) in Ottawa, obtained samples of the sludge from these pilot tests to characterize the material chemically, physically and mineralogically.

The sludge was made up of both amorphous and crystalline material. Minerals identified in the sludge were crystalline ettringite, calcite, gypsum and quartz.

This report outlines the nature of the sludge samples, as received from the pilot tests, however tests on fresh sludge may display different characteristics than observed. Age, preservation and/or storage of the samples are expected to impact sludge characteristics. To ascertain the degree of alteration experienced fresh sludge samples could be characterized against the results of these samples. By characterizing the nature of sludge and how it changes over time (and other parameters), future pilot tests could focus more on how sludge characteristics would suit disposal options.

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INTRODUCTION

The Britannia Mine was a copper operation on the southwest coast of British Columbia between 1902 and 1974. The mine is located in Britannia Beach, B.C., on Highway 99 between Vancouver and Whistler, B.C. The mine's concentrator is now the site of a mining museum which is open to the public from May to October.

Mining was carried out underground using sublevel caving methods and from a number of open pits on surface in an area known as Jane Basin. The sublevel caving extended to surface so that today, the snowmelt and precipitation in Jane Basin, flows into the underground mine workings where it picks up acid. Almost all the ARD flows out the 4100 Level portal after installation of a plug at the 2200 level by The University of British Columbia, CERM3 program. The flow from the 4100 Level is piped to a submarine discharge in Howe Sound.

When the Britannia Mine ceased operations in 1974, the owners of the property closed the mine to a standard that satisfied the regulatory requirements of the time. Today, reclamation standards have changed and there is a greater expectation on the part of the public that mine sites will be reclaimed to a higher standard.

In 2001, the Province provided indemnification for environmental liabilities to the successor companies of the mine operators, in exchange for \$30 million. Using this money, the BC Ministry of Water, Land, and Air Protection office has begun studying the remediation of the Britannia Mine site.

Canadian Environmental and Metallurgical Inc. (CEMI) conducted HDS pilot tests on the Britannia mine water. Samples from these and other treatment runs were characterized chemically, physically and mineralogically.

METHODOLOGY

The sludge samples characterized in this study were generated during pilot scale testing of the High Density Sludge (HDS) process at the Britannia Mine site in British Columbia during February - March 2002. Canadian Environmental and Metallurgical Inc. (CEMI) of Vancouver carried out the study. Information regarding the treatment conditions under which each sample was produced can be found in the May 2002 report prepared for the BC Ministry of Water, Land and Air Protection, Contract No: CLM02-57 (CEMI, 2002). However, there are conditions that may affect the results that are more difficult to report and measure. While the report (CEMI, 2002) clearly documents the process conditions that produced the sludge the preservation, storage and handling condition of each sample post-treatment will affect the sludge properties and may not accurately represent the true characteristics shortly after treatment.

High Density Sludge (HDS) produced from the pilot scale tests conducted at the Britannia Beach Mine site in British Columbia during February and March 2002 were

obtained from two sources. Samples from Run 4 and Run 9 were shipped to CANMET-MMSL lab in Ottawa from Golder Associates Ltd. Received were, two (2) one-liter samples of Run 4 and two (2) one-liter samples of Run 9. Upon inspection, it was noted that the samples arrived as a paste and contained no overlying water thus indicating partial dehydration of the sludge samples. The effect that this may have on the sludge characteristics is unknown and may require further investigation. However, investigating this potential effect is not the focus of this report. Consideration is given to the dehydration effect for selection of samples to undergo detailed analysis. Every attempt was made to select samples that showed the least degree of dehydration to lessen uncertainty associated with the effect it may have.

In addition to the samples received from Golder Associates Ltd., several HDS samples were obtained from CEMI. These samples included HDS sludge from test Runs 2,3,8,9,10 and 13. Further inspection of these samples indicate that they were mostly concentrated sludge samples, meaning that most of the overlying water had been removed prior to storage of the sample. It appeared however, that the samples remained saturated with water indicated by small amount of overlying water. Also received from CEMI, were three (3) twenty (20)-liter pails of sludge. Identifiers on the pails indicated two (2) from Run 9 and one (1) from Run 13. Since these “bulk samples” were estimated to have 75 – 85% water any concern over dehydration was not an issue. However, it should be noted that age and storage conditions may contribute to differences in sludge characteristics.

Sample Selection Rationale

Of the seven (7) samples obtained, three (3) of the samples were selected for detailed analysis. The selection of these samples was based on a combination of factors: 1) samples most representative of the expected conditions of a “full scale” HDS treatment plant including water chemistry and operating conditions; 2) sample integrity, samples that are considered least affected from collection and storage procedures over the twelve (12) month period (i.e. Run 9 and Run 13, pails); and 3) time constraints for analysis.

Based on the above criteria, the decision was made to conduct detailed analysis on Run 4 obtained from Golder Associates Ltd. Run 4 was considered to be one of the test predicted to represent conditions expected from a full scale HDS treatment facility. In addition, Run 9 (bulk sample) obtained from CEMI was chosen to undergo detailed analysis. Run 9 was also thought to represent conditions expected in a full scale HDS treatment facility and considered to be one of the better preserved samples for the 12 month storage. A final sample (Run 13, bulk sample) was selected, as it appeared to be well preserved during the 12-month storage period and provided a comparison with the other samples selected.

Preparation for detailed analysis

Run 9 and 13 were received in 20-liter pails and due to the long storage period, they required homogenization prior to sub-sampling. Homogenization involved using a

lightning mixer with a low shear impeller at a speed between 500 – 800 rpm for approximately 60 minutes. Mixing of these samples proved to be challenging due to compaction over the long storage period, for this reason some manual mixing was required. Manual mixing was achieved by using a rod to dislodge “caked” sludge from the bottom that had not been readily suspended with the mixer. Once dislodged, increased speed of the mixer was sufficient to achieve homogenization. While mixing institute measurements of pH, ORP and conductivity were obtained. Sub-samples were collected for percent solids and freeze-drying prior to physical, chemical and mineralogical analyses.

Run 4 the paste sludge sample a decision was made not to measure pH, ORP, conductivity, viscosity and percent solids. It was felt that the information would not be accurate with so many unknowns. Table 1 summarizes the samples received and the analyses completed on each sample.

Table 1: Samples received and the analysis for each.

	Run 2	Run 3	Run 4	Run 8	Run 9 *	Run 10	Run 13 *
Physical							
Freeze dried	x	x	x	x	x	x	x
Particle size	x	x	x	x	x	x	x
% Solids					x		x
Viscosity					x		x
Chemical ¹							
	x	x	x	x	x	x	x
Mineralogy							
SEM			x		x		x
XRD			x		x		x

* - 20-liter pail sample

1 – Chemical analysis (Al, As, Ca, Cd, Co, CO₂, Cr, Cu, Fe, K, LOI, LOM, Mg, Mn, Na, Ni, Np, Pb, S, Si, SO₄, Zn)

Freeze-drying

All samples were freeze-dried and further homogenized prior to analyses. Freeze drying is thought to better ensure the integrity of the sample than other drying methods (Zinck et al, 1997). After the samples had been freeze-dried they were homogenized further in powder form, the samples were further divided and submitted for analysis. Including 1 – 2 g particle size analysis, ~ 1g for XRD, ~ 1g for SEM analysis, 1 – 5 g chemical analysis while the remainder of sample is archived.

Sample preparation

Run 2, 3, 8, and 10 were received in 250 mL glass jars. These samples were “concentrated solids” that remained saturated but had most of the water removed from the sample. One jar from each of these was selected at random, freeze-dried, homogenized as a dry powder and further sub sampled. The freeze-dried sample

submitted for particle size and chemical analysis, the remainder of the sample was archived.

Physical Measurements

Run 9 and 13, after ~60 minutes once sufficiently homogenized, institute measurements of pH, ORP and conductivity were obtained. In addition while maintaining mixing, three sub samples were collected to measure percent solids content of the slurry. The percent solids is measured by drying the samples in an oven at 60 degrees Celsius for 24 hours and recording the moisture loss of the sample (Zinck, 1993)

Viscosity readings were also taken on these samples by measuring 1 liter of the slurry and placing it in a cone equipped with a valve at the bottom. The valve is open the time it takes the sample to empty from the cone is related to the viscosity of the sample. (Aubé et al, 2002)

Particle size measurements were completed using a Horiba LA 920 particle size analyzer. All seven samples were measured using this instrument.

Chemical analysis

All seven samples were also analyzed chemically for Al, As, Ca, Cd, Co, CO₂, Cr, Cu, Fe, K, loss on ignition (LOI), loss of moisture (LOM), Mg, Mn, Na, Ni, Na, Pb, S, Si, SO₄, Zn. The parameters were measured using an ICP-MS, ICP-IRS and LECO analyzer.

Mineralogy

Both X-Ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) techniques were used to determine the mineral composition of the sludge samples. The SEM used was equipped with an Energy Dispersion X-Ray (EDX) analyzer.

RESULTS AND DISCUSSION

Physical Characterization

Viscosity

Viscosity of both samples measured Run 9 and Run 13 were between 18 -20 cP.

Particle size analysis

Particle size analysis was performed on all sludge samples received using a Horiba LA 920 Particle Size Analyzer. Detailed analysis report for each sample is appended (Appendix A). In general, Runs 2, 3, 10 and 13 displayed a bimodal particle size distribution, while the distribution for Run 4, 8, and 9 was tri-modal. Table 2

summarizes the particle size median, mode and variance for each of the seven samples analyzed. Mean particle size for the samples analyzed ranged from 3.38 – 12.50 μm .

In order to examine the particle size distribution in more detail the analyses were grouped by feed composition. Therefore, a comparison of Run 2,3 and 4 is possible because they all received feed from the 4100 level portal as reported in the CEMI report.

When compared Run 2 and 3 have similar distribution patterns. However, particles from Run 3 were smaller than for Run 2. Mean particle size for Run 2 and 3 are 10.51 μm and 8.78 μm respectively (Table 2). Possible reasons for this shift in size between the two runs may be the process differences noted, such as the increased sludge recycle ratio or the decrease pH reported on the clarifier overflow. The particle size distribution of Run 4 was unique in comparison to the other sludges as it exhibited a tri-modal distribution pattern. While the mean particle size for Run 4 was 10.48 μm , it is unclear why the distribution pattern was different from Run 2 and 3. A possible explanation could be the increase in retention time along with the increase in pH set point noted during Run 4.

In contrast, to Run 2, 3 and 4, the sludge produced during Run 8, 9, 10 was produced from a blended feed. Distribution patterns for Run 8 and 9 are similar to one another and appear as tri-modal pattern. As well, the mean particle size of each are close and differ by only 0.8 μm . The same is true for the median, which differ by 0.04 μm . The process difference between Run 8 and 9 was simply the addition of the cadmium spike to Run 9. This appears that this had little or no effect on the size of the particle. However, Run 10 displayed a very different particle size distribution with a shift to a distinct bimodal distribution. In addition, there is an overall shift in particle size with the mean dropping to 3.38 μm . Run 10 also showed a much narrower distribution ranging from 0.2 – 20 μm , with two distinct peaks between 0.5 – 1 μm and 2 – 10 μm . A possible explanation for this change maybe related to the increased recycle ratio and short run time noted. The process may not of had enough time to reach equilibrium. Notably the decrease of particle size resulted in an increase of flocculants consumption. Particle size analysis of Run 13 revealed a bimodal distribution. The mean size of the particles was 8.33 μm . Currently there is no information on Run 13 test conditions under which this sample was produced.

Table 2: Particle size distribution

Sample	Median (μm)	Mean (μm)	Variance (μm^2)
Run 2	4.11	10.51	335.0
Run 3	3.77	8.778	226.5
Run 4	5.01	10.46	108.7
Run 8	6.41	13.35	335.3
Run 9	6.45	12.50	266.7
Run 10	2.67	3.38	9.03
Run 13	3.43	8.33	211.4

While, all the sludges were generated under HDS pilot plant conditions their particle size distributions, specifically mean particle size do not consistently agree with published trends regarding particle size, treatment process conditions and resultant sludge density. Figure 1 demonstrates that for the most part larger mean diameter particles are associated with low-density sludges while high-density sludges tend to have smaller, more uniform particle size distributions (Aubé and Zinck, 1999).

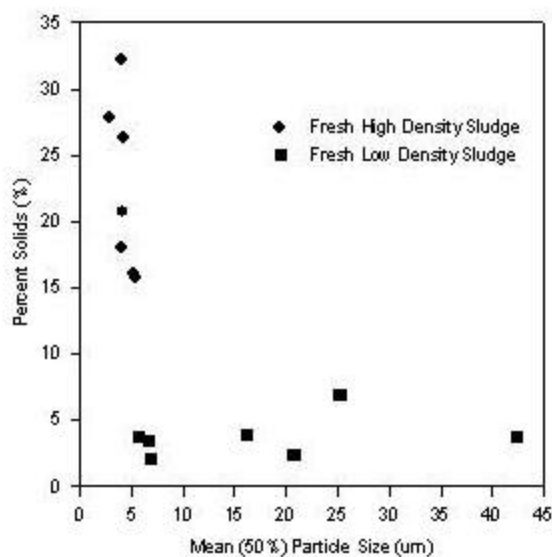


Figure 1: Relationship between particle size and sludge density

Percent Solids

Where possible percent solids were measured on samples received. However, due to many unknowns percent solid measurements reported in the CEMI report were not comparable to the measured values of the samples received. The measurement of percent solids content of Run 9 collected during homogenization of the sample is recorded to be 6.44 % and 15.28 % for Run 13 (Table 3). The low solids content of Run 9 differs from the 15.0% recorded in the CEMI report. This difference draws into question sample integrity, suggesting samples had been altered during storage and how

sludge characteristics may be affected. For Run 13 the percent solids measured on the bulk sample compare closely to values reported during pilot plant testing.

Table 3: Percent solids measurements.

	Trial 1 (%)	Trial 2 (%)	Trial 3 (%)	Average (%)
Run 9	6.46	6.46	6.41	6.44
Run 13	15.23	15.22	15.32	15.28

Chemical Characterization

The pH of the samples measured did not vary much between the two runs with Run 9 measuring 10.13 and Run 13 measuring 10.03. As compared to other sludges (Zinck, 1997), the sludge pH was high and suggests some degree of process inefficiency and over liming. Notably Run 9 reported the highest lime consumption rate.

The conductivity and ORP measurements of Run 9 were 1.97 mS and – 39 mV respectively and Run 13 measured at 1.77 mS and – 4 mV. .

A complete list of chemical analysis, of the sludge samples along with the results obtained by CANMET lab in February 2003 can be found in Appendix B. Table 4 summarizes the chemical analyses data obtained from the CEMI report and CANMET analyses.

Table 4: Chemical analysis of sludge solid samples

	Run 3			Run 4		Run 8		Run 9	
	CEMI	CEMI	CANMET	CEMI	CANMET	CEMI	CANMET	CEMI	CANMET
Al %	7.96	6.08	6.44	6.28	3.2	4.55	3.63	4.6	4.19
Cd %	0.021	0.021	0.0193	0.019	0.0117	0.008	0.0064	0.013	0.0135
Cu %	5.3	5.36	4.96	4.78	2.68	3.3	3.02	3.64	3.19
Fe %	2.56	2.6	3.1	2.51	3.59	3.8	3.95	4.59	4.11
Mg %	4.09	2.95	3.31	3.33	2.33	3.14	2.7	3.08	2.75
Zn %	4.51	4.7	4.35	4.16	3.29	3.92	3.63	4.25	3.77
S %	3.15	2.71	2.92	2.93	4.91	5.47	5.72	5.52	6.08

The major components in the samples were calcium, aluminum, copper, zinc, iron, magnesium, sulfur, silica and manganese. The samples analyzed also contained lower concentrations of sodium, cadmium, cobalt, chromium, and nickel. Overall, the analysis of the samples showed that the chemical composition of the sludge remained proportional to one another. However, some of the species appear to be more readily affected by process changes such as the case with Fe and S. Zn as well was affected by process changes but to a lesser degree than the Fe and S.

Overall Run 4 has the lower concentration for most of the major components present in the sludge sample among them were Al , Cu, Mg, Mn, Zn and Si. However, this is not

the case for the Fe and S concentrations, which varied from sample to sample. The high LOI and LOM values for Run 4 suggest that the sample may not have been completely dry and the apparent decrease in metal concentrations in the sludge were a result of dilution.

In most cases, the variation in the sludge composition is due to process related factors such as recycle ratio and feed composition. Aubé and Zinck (1999) studied the effect of various treatment processes and their impact on sludge characteristics. The process parameters an effect everything from the chemical composition of the sludge, the sludge stability, the particle size to degree of sludge crystallinity.

Due to the effects process changes has on the sludge characteristics. There may be potential to produce a sludge that meets certain specifications. Such as the case from a stability and sludge management point of view (i.e. objective to produce a stable dense sludge). Investigating the potential for engineering sludge for a specific purpose is possible and is an area that is largely overlooked.

Mineralogical Characterization

In general terms, except for the presence of abundant acicular grains the sludge samples appear largely as an amorphous material that is an agglomeration of smaller mineral grains. Appendix C presents the detailed mineralogical analyses and images.

Image 1-1 is a backscatter image of Run 13, the fibrous grains range 1 - 50 μ m. All samples analyzed were freeze-dried. Run 4, 9 and 13 were “dusted” onto an aluminum stub that contained a double-sided adhesive carbon disk. The sample had been carbon coated as is required for SEM analysis. The difference in gray scale seen in an image is an indication of differences in chemical composition (backscatter electron image (BEI)). However, in this case the difference in gray scale could be due to the uneven surface and charging from this mounting technique. A more detailed analysis of each sample was carried out on polished sections prepared to minimize the effect of an uneven surface.

The sludge from Run 4 appears as loose agglomeration of fine-grained particles seen in Image 1-2. Higher magnification of these agglomerated particles (Image 1-3) show that these agglomerates are made up of crystalline and non-crystalline fine-grained minerals often less than 20 μ m in size. The larger agglomerates measure upwards of 700 μ m. The chemical composition varies throughout the sample with the elements Ca, S, P, Al, Si, Mg, Mn, Zn, Cu, and Fe at different concentrations (Figure 1-1). Larger-grained minerals are present but tend to be much less common e.g. Image 1-4 and 1-5 showing calcite and gypsum, respectively. More often, these larger-grained minerals are hosted within the larger agglomerate such as the case with the quartz in Image 1-6.

X-ray diffraction analysis identified the presence of ettringite ($\text{Ca}_6\text{Al}_2(\text{SO}_4)_3(\text{OH})_{12} \cdot 26\text{H}_2\text{O}$) XRD diffractogram 1-1. Ettringite is confirmed with EDX analysis and can be seen as the acicular grains found throughout the sample in Image 1-1. Gypsum, also identified by XRD, may account for some of the acicular grains present but EDX analysis

of individual grains show the majority to be comprised of mainly Ca, S and Al suggesting ettringite (Figure 1-2). Because the peak intensity for each of these elements is similar, when analyzing each grain it is thought that ettringite is most likely. The evidence from EDX spectra acquired suggest that ettringite is associated with other elements adsorbed to the surface of the mineral or the analysis is influenced by the background elements close to the mineral. This would explain the high peaks for Mg, Fe, Cu, Zn, Si and Mn seen in the spectra acquired when comparing Figures 1-2 to 1-4 and also why peak intensities for these elements vary for each spot analysis acquired. Furthermore the ratio of Ca:S:Al remain the same suggesting each grain analyzed in this case are the same mineral. However, the change in peak intensities for the elements Mg, Fe, Cu, Zn, Si, and Mn can be explained by either adsorption or background interferences, as previously mentioned. The analysis of the matrix, Images 1-8 and 1-9, show a similar chemistry to the crystalline ettringite. However, EDX spectra Figures 1-5 & 1-6 respectively show higher Ca present than compared to Figures 1-2 to 1-4. This along with the lack of crystallinity of these areas suggests the matrix is something other than ettringite.

Crystalline calcite identified by XRD is found throughout the sample (Images 1-2 and 1-4). Calcite can occur as a single pure mineral as in Image 1-4 but more commonly is hosted in larger agglomerate as seen in Images 1- 2 & 1-9. EDX spectrum Figure 1-7 shows a typical calcite spectrum as seen in Image 1-9.

Run 9 (Image 1-10) shows the sample is comprised predominantly of acicular grains 1 to 50 μ m in length. EDX analysis of these grains shows chemically they contain Ca, S, and Al as seen in spectrum in Figure 1-8. X-Ray Diffraction analysis identified crystalline minerals calcite, gypsum, quartz and aragonite, along with amorphous minerals (XRD-Diffractogram 1-2). Similar to Run 4, this sample has varying amounts of Fe, Cu, Zn, Si, Mn and Mg throughout. In addition to the acicular grains of Image 1-10 it shows the presence of rounded mineral grains also. The rounded grains tend to be smaller, often less than 10 μ m in diameter. EDX analysis in Figure 1-9 of these grains shows they contain mostly Ca, as well as minor amounts of other elements such as Al, Si, S, Fe, Cu, Zn, Mn, and Mg. Also present in both Image 1-10 and Image 1-11 are tabular gypsum grains.

Image 1-12 of Run 9 shows that the sample is actually a loose agglomeration of smaller individual grains. SEM/EDX observations showed that many of these grains contain calcium as the major component. However, there is not enough information available to differentiate, which grains are calcite and which are aragonite. However, based on the XRD information it is thought that calcite is more abundant in the sample. Quartz, in both Images 1-15 & 1-16 produce a strong EDX peak for Si (Figure 1-13). Gypsum seen in Images 1-10 & 1-11 are present as tabular grains. The abundant acicular grains throughout the sample are believed to be both gypsum and ettringite similar to Run 4. EDX analysis of the sample shows that individual grains contain many different elements such as Ca, Mg, Al, Si, S, Fe, Cu, Zn and Mn such as the case with Image 1-15 (Figure 1-14). However, as previously mentioned the elements present may be due to background or adsorption, this would explain the similarities between Figure 1-14 & 1-15 spectra. While both spectrum have been acquired from a different location, Figure 1-14 from

image 1-15 acicular grain and Figure 1-15 from Image 1-16 Matrix, both spectra display similar chemistry.

Run 13 X-Ray diffractogram (XRD 1-3) showed calcite, gypsum, quartz and ettringite. Similar to Run 4 and 9 this sample is a loose agglomeration of smaller mineral grains pictured in Image 1-17. EDX analysis confirms the presence of calcite and quartz as indicated. A higher magnification of the sample (Image 1-18) show rounded grains and acicular grains throughout the sample. Comparing the EDX analysis between the two Figures 1- 16 & 1-17 indicates similar chemistry. However, the peak intensity for the different elements varies, suggesting the amount of the particular element is different.

In each of the 3 samples analyzed crystalline minerals of ettringite, calcite, gypsum & quartz are present. The crystalline minerals are of particular interest from a stability and disposal point of view. In addition these minerals may be equally of interest to a potential end user. Again it is important to note that engineering HDS sludge for a particular use is largely an under explored area.

CONCLUSIONS

1. Characterization of the sludge was based on condition of samples received. Any effective of age, storage and/or preservation techniques of samples is undetermined.
2. Compared to other sludges, the sludge pH was high and suggests some degree of process inefficiency and over liming.
3. The major chemical components in the sludge samples were calcium, aluminum, copper, zinc, iron, magnesium, sulfur, silica and manganese. The samples also contained lower concentrations of sodium, cadmium, cobalt, chromium, and nickel.
4. Except for the presence of abundant acicular grains, the sludge samples appear largely as an amorphous material that is an agglomeration of smaller mineral grains. This amorphous mass contained most of the metals (Fe, Zn, Cu, Al).
5. The crystalline components of the sludge consisted of calcite, gypsum, quartz and ettringite. These crystalline components occurred as a single pure mineral but more commonly is hosted in larger agglomerate

RECOMMENDATIONS

1. Fresh sludge samples from HDS pilot plant testing should be compared to the characterization of these aged sludges to ascertain the degree of alteration experienced.
2. Optimization of the HDS processes in terms of lime consumption should be conducted.
3. Changes in the HDS process should be investigated in terms of producing sludge favorable for alternative uses (i.e., Supplementary Cementing Materials).
4. Additional pilot tests should be performed with a greater focus on sludge characteristics to aid in disposal options.

ACKNOWLEDGMENTS

The authors wish to acknowledge the contribution of CANMET's Analytical Services Group and John Kwong for reviewing the mineralogical portion of this report. The authors would also like to thank Golder Associates and Canadian Environmental and Metallurgical Inc. for supplying CANMET with the sample characterized in this study. The authors would also like to thank province of British Columbia, Ministry of Water Land and Air Protection for assistance and cooperation obtaining samples.

REFERENCES

CEMI, 2002, Pilot Scale Testing of the High Density Sludge Process, Report prepared for BC Ministry of Water, Land, and Air Protection, Contract CLM 02-057, May 2002.

Aubé, B.A., Zinck, J.M., Griffith, W.F., and Black, C., 2002, Pilot Treatment of Mine Water from {Client Confidential, MMSL Report 02-065(CR).

Aubé, B.A. and Zinck, J.M. 1999. Comparison of AMD Treatment Processes and Their Impact on Sludge Characteristics. Paper presented at *Mining and the Environment II, Sudbury '99*. September 12-16, 1999 at Sudbury, Ontario.

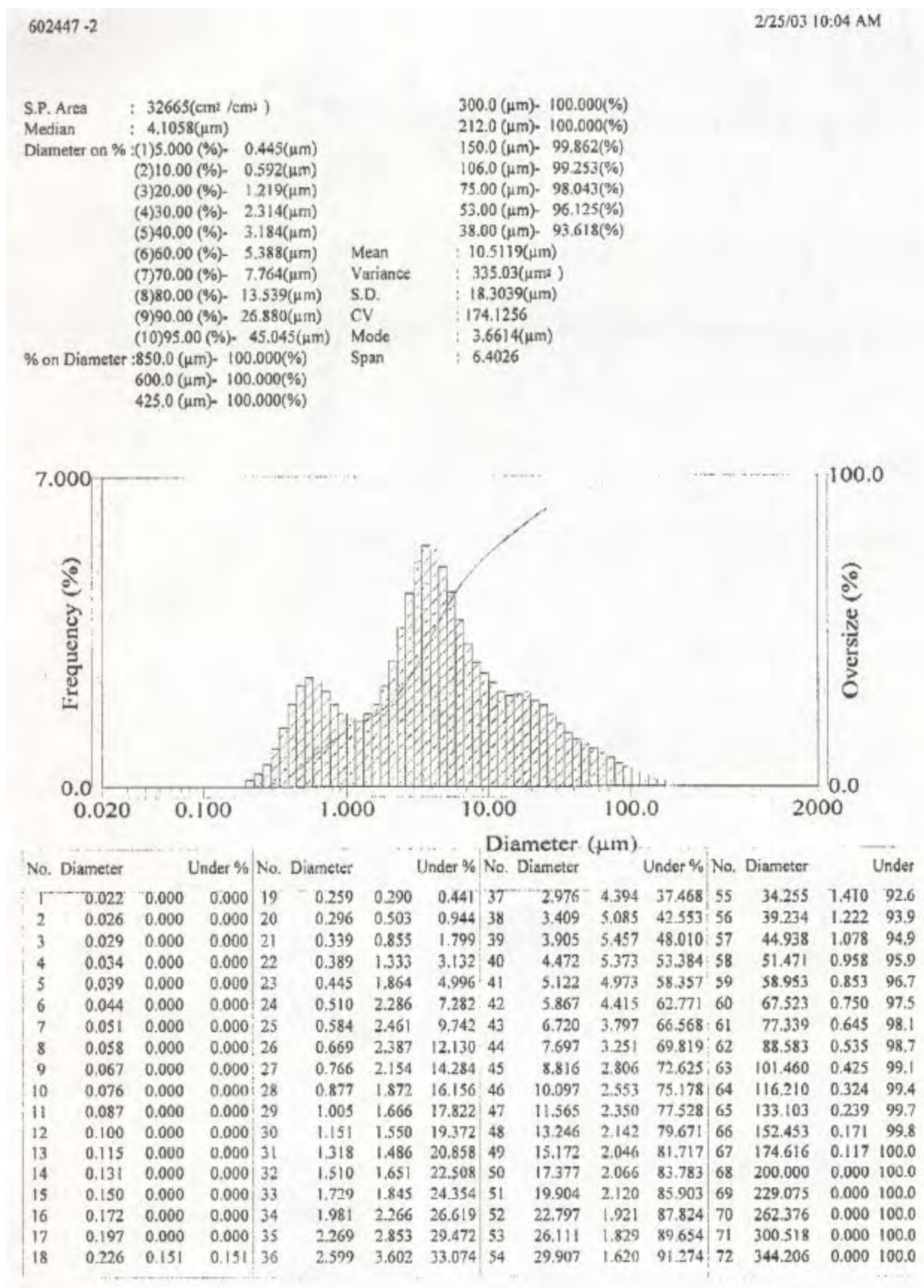
Zinck, J.M., 1993, An investigation into the hydrolytic precipitation of Fe(III) from sulphate-bearing effluents. M.Eng., McGill University

Zinck, J.M., 1997. Acid Mine Drainage Sludges in the Canadian Mineral Industry: Physical, Chemical, Mineralogical and Leaching Characteristics. Paper presented at *the Fourth International Conference on Acid Rock Drainage*, May 31 - June 6, 1997, Vancouver, British Columbia

Zinck, J.M., Wilson, L, Chen, T.T., Mikhail, S., Turcotte, A.M., and Griffith, W.F., MEND, 1997. Characterization and Stability of Acid Mine Drainage Sludges. MEND Report 3.42.2, May 1997.

APPENDIX A: Detailed Particle Size

Run 2 Particle size analysis

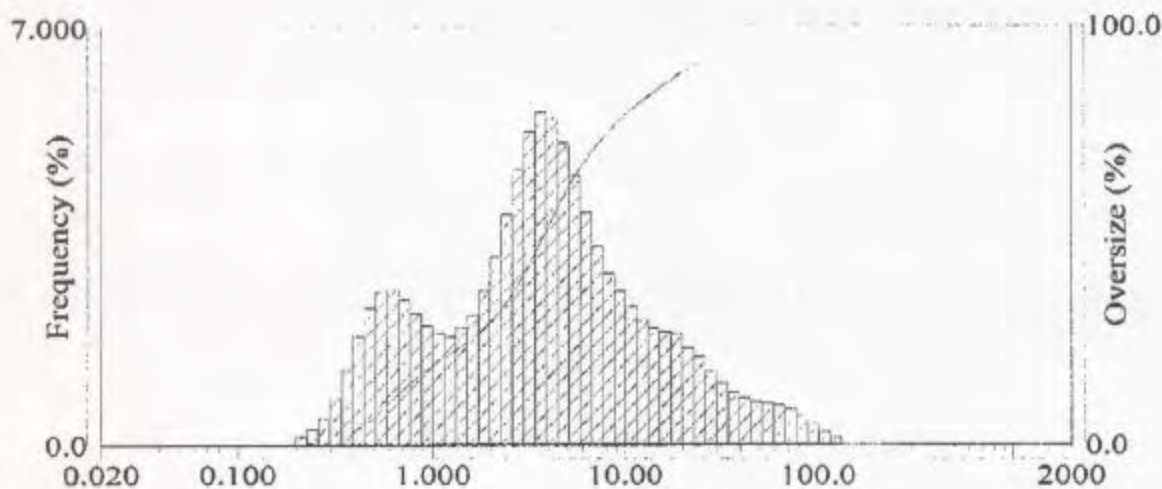


Run 3 Particle size analysis

602447 -3

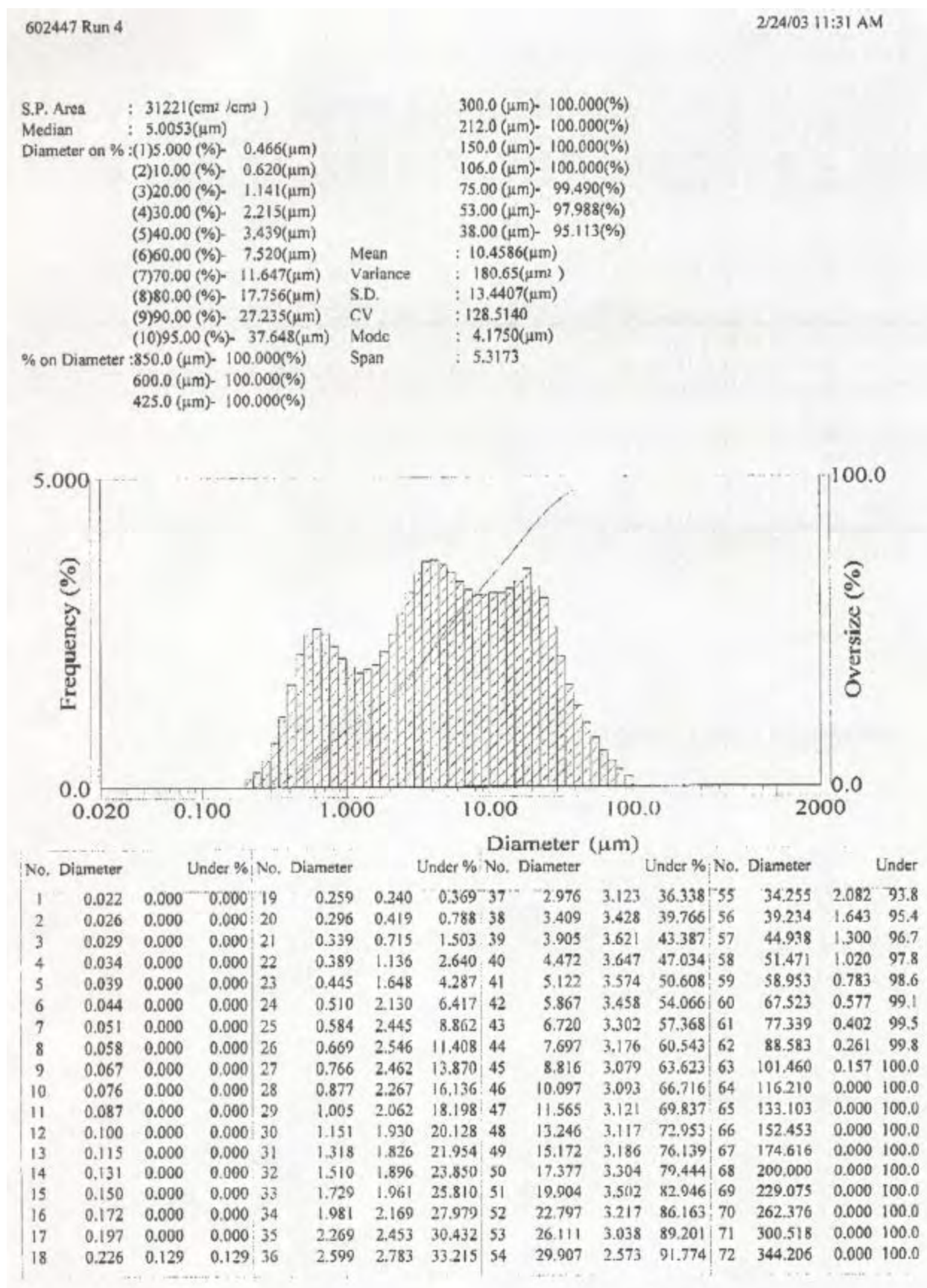
2/25/03 9:05 AM

S.P. Area : 33867($\text{cm}^2 / \text{cm}^3$)
 Median : 3.7713(μm)
 Diameter on % : (1)5.000 (%) - 0.454(μm)
 (2)10.00 (%) - 0.599(μm)
 (3)20.00 (%) - 1.106(μm)
 (4)30.00 (%) - 2.069(μm)
 (5)40.00 (%) - 2.924(μm)
 (6)60.00 (%) - 4.859(μm)
 (7)70.00 (%) - 6.634(μm)
 (8)80.00 (%) - 10.635(μm)
 (9)90.00 (%) - 21.390(μm)
 (10)95.00 (%) - 37.212(μm)
 % on Diameter : 850.0 (μm) - 100.000(%)
 600.0 (μm) - 100.000(%)
 425.0 (μm) - 100.000(%)
 300.0 (μm) - 100.000(%)
 212.0 (μm) - 100.000(%)
 150.0 (μm) - 100.000(%)
 106.0 (μm) - 99.720(%)
 75.00 (μm) - 98.660(%)
 53.00 (μm) - 96.990(%)
 38.00 (μm) - 95.134(%)
 Mean : 8.7766(μm)
 Variance : 226.47(μm^2)
 S.D. : 15.0491(μm)
 CV : 171.4685
 Mode : 3.6583(μm)
 Span : 5.5129

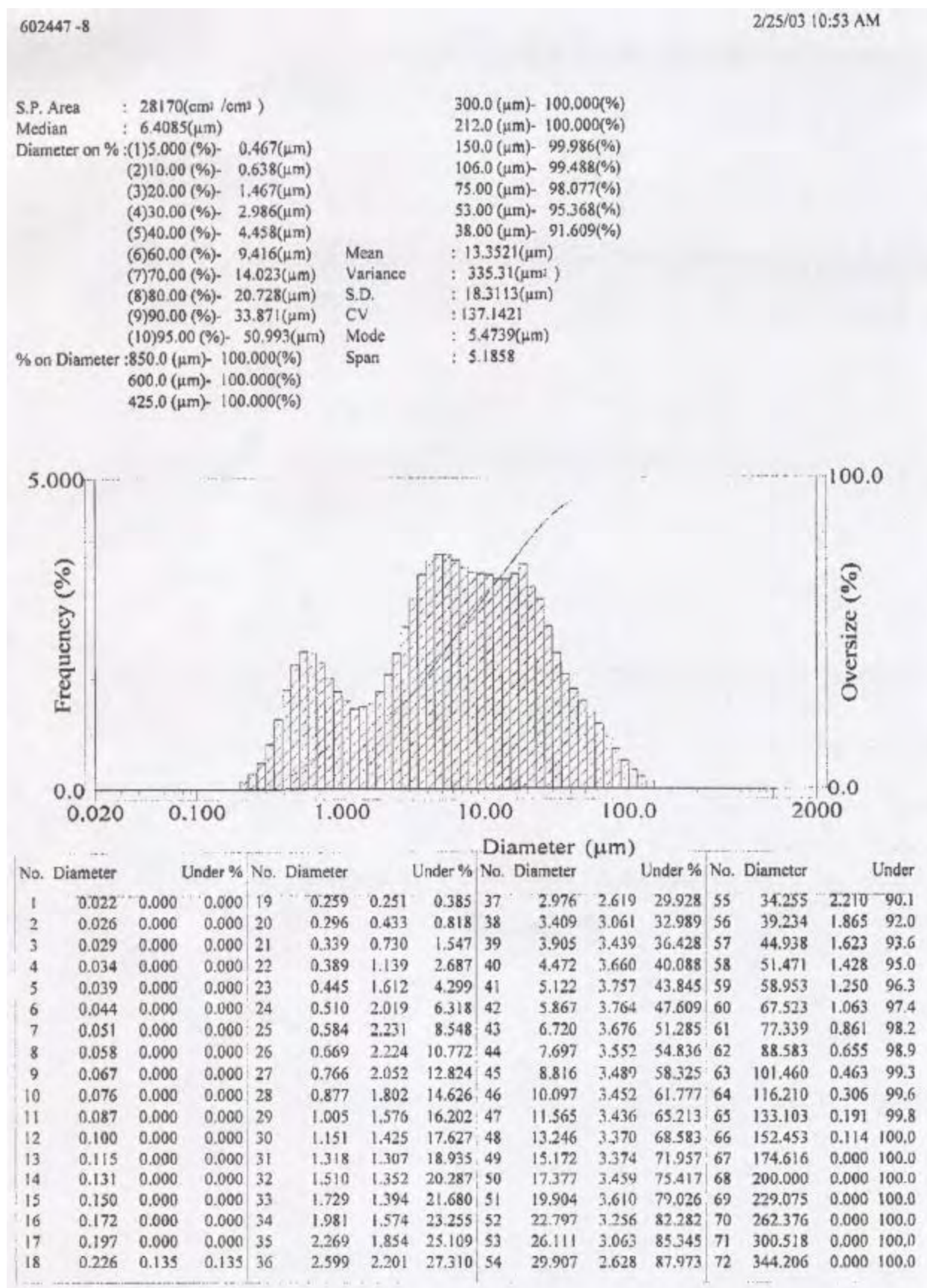


Diameter (μm)							
No.	Diameter	Under %		No.	Diameter	Under %	
1	0.022	0.000	0.000	19	0.259	0.257	0.393
2	0.026	0.000	0.000	20	0.296	0.453	0.846
3	0.029	0.000	0.000	21	0.339	0.779	1.626
4	0.034	0.000	0.000	22	0.389	1.242	2.867
5	0.039	0.000	0.000	23	0.445	1.791	4.658
6	0.044	0.000	0.000	24	0.510	2.281	6.939
7	0.051	0.000	0.000	25	0.584	2.560	9.499
8	0.058	0.000	0.000	26	0.669	2.591	12.090
9	0.067	0.000	0.000	27	0.766	2.433	14.523
10	0.076	0.000	0.000	28	0.877	2.184	16.707
11	0.087	0.000	0.000	29	1.005	1.977	18.683
12	0.100	0.000	0.000	30	1.151	1.858	20.541
13	0.115	0.000	0.000	31	1.318	1.787	22.328
14	0.131	0.000	0.000	32	1.510	1.954	24.282
15	0.150	0.000	0.000	33	1.729	2.145	26.427
16	0.172	0.000	0.000	34	1.981	2.564	28.991
17	0.197	0.000	0.000	35	2.269	3.139	32.131
18	0.226	0.136	0.136	36	2.599	3.859	35.989
				37	2.976	4.609	40.598
				38	3.409	5.250	45.848
				39	3.905	5.579	51.427
				40	4.472	5.474	56.901
				41	5.122	5.070	61.971
				42	5.867	4.512	66.483
				43	6.720	3.887	70.370
				44	7.697	3.317	73.687
				45	8.816	2.864	76.552
				46	10.097	2.563	79.115
				47	11.565	2.317	81.432
				48	13.246	2.069	83.501
				49	15.172	1.923	85.424
				50	17.377	1.872	87.296
				51	19.904	1.851	89.147
				52	22.797	1.607	90.754
				53	26.111	1.460	92.214
				54	29.907	1.230	93.444
				55	34.255	1.026	94.4
				56	39.234	0.869	95.3
				57	44.938	0.778	96.1
				58	51.471	0.724	96.8
				59	58.953	0.692	97.5
				60	67.523	0.661	98.1
				61	77.339	0.603	98.7
				62	88.583	0.497	99.2
				63	101.460	0.355	99.6
				64	116.210	0.222	99.8
				65	133.103	0.129	100.0
				66	152.453	0.000	100.0
				67	174.616	0.000	100.0
				68	200.000	0.000	100.0
				69	229.075	0.000	100.0
				70	262.376	0.000	100.0
				71	300.518	0.000	100.0
				72	344.206	0.000	100.0

Run 4 Particle size analysis



Run 8 Particle size analysis

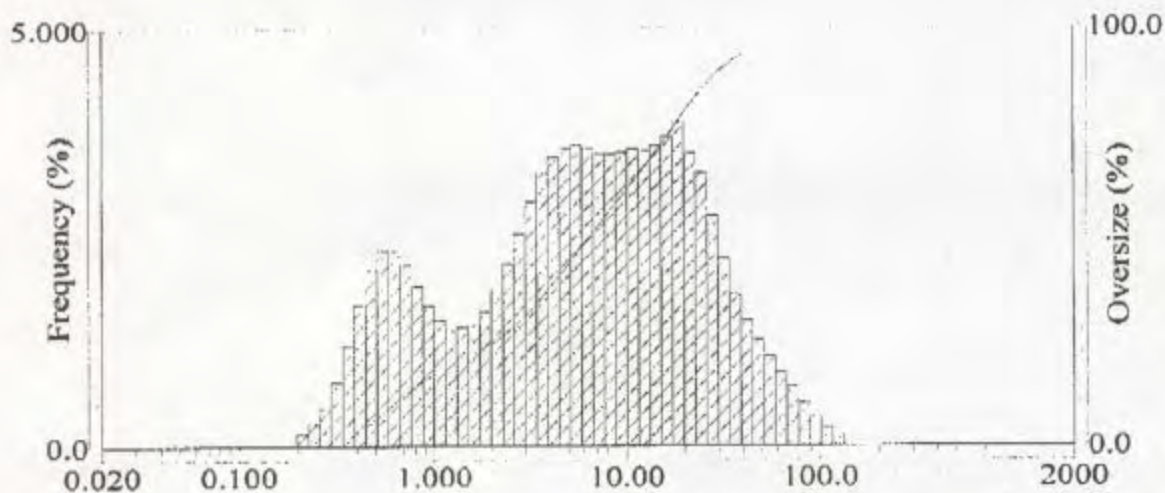


Run 9 Particle size analysis

602447-9

2/24/03 10:38 AM

S.P. Area : 28894(cm^2/cm^3)
Median : 6.4478(μm)
Diameter on % : (1)5.000 (%) - 0.461(μm)
(2)10.00 (%) - 0.624(μm)
(3)20.00 (%) - 1.353(μm)
(4)30.00 (%) - 2.870(μm)
(5)40.00 (%) - 4.414(μm)
(6)60.00 (%) - 9.485(μm)
(7)70.00 (%) - 13.890(μm)
(8)80.00 (%) - 19.953(μm)
(9)90.00 (%) - 30.885(μm)
(10)95.00 (%) - 45.082(μm)
% on Diameter : 850.0 (μm) - 100.000(%)
600.0 (μm) - 100.000(%)
425.0 (μm) - 100.000(%)
300.0 (μm) - 100.000(%)
212.0 (μm) - 100.000(%)
150.0 (μm) - 100.000(%)
106.0 (μm) - 99.732(%)
75.00 (μm) - 98.664(%)
53.00 (μm) - 96.440(%)
38.00 (μm) - 93.070(%)
Mean : 12.5017(μm)
Variance : 266.67(μm^2)
S.D. : 16.3300(μm)
CV : 130.6216
Modc : 18.5339(μm)
Span : 4.6932



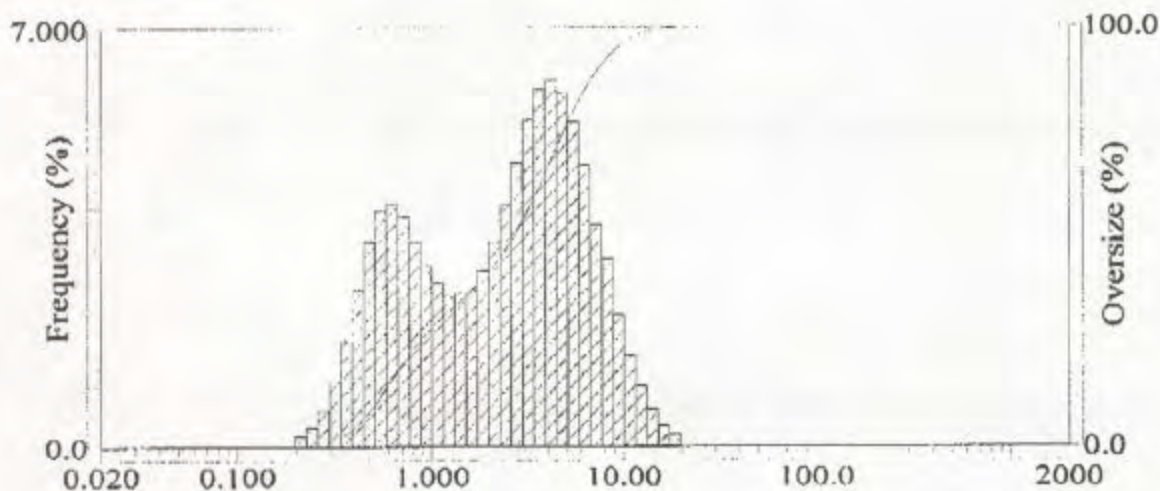
No.	Diameter	Under %	No.	Diameter	Under %	No.	Diameter	Under %	No.	Diameter	Under %
1	0.022	0.000	19	0.259	0.259	37	2.976	2.530	55	34.255	2.230
2	0.026	0.000	20	0.296	0.447	38	3.409	2.924	56	39.234	1.790
3	0.029	0.000	21	0.339	0.754	39	3.905	3.263	57	44.938	1.480
4	0.034	0.000	22	0.389	1.179	40	4.472	3.467	58	51.471	1.243
5	0.039	0.000	23	0.445	1.670	41	5.122	3.572	59	58.953	1.050
6	0.044	0.000	24	0.510	2.095	42	5.867	3.610	60	67.523	0.869
7	0.051	0.000	25	0.584	2.321	43	6.720	3.572	61	77.339	0.687
8	0.058	0.000	26	0.669	2.320	44	7.697	3.509	62	88.583	0.505
9	0.067	0.000	27	0.766	2.147	45	8.816	3.502	63	101.460	0.339
10	0.076	0.000	28	0.877	1.891	46	10.097	3.525	64	116.210	0.211
11	0.087	0.000	29	1.005	1.653	47	11.565	3.564	65	133.103	0.125
12	0.100	0.000	30	1.151	1.492	48	13.246	3.552	66	152.453	0.000
13	0.115	0.000	31	1.318	1.365	49	15.172	3.605	67	174.616	0.000
14	0.131	0.000	32	1.510	1.399	50	17.377	3.706	68	200.000	0.000
15	0.150	0.000	33	1.729	1.428	51	19.904	3.886	69	229.075	0.000
16	0.172	0.000	34	1.981	1.591	52	22.797	3.509	70	262.376	0.000
17	0.197	0.000	35	2.269	1.846	53	26.111	3.275	71	300.518	0.000
18	0.226	0.139	36	2.599	2.156	54	29.907	2.751	72	344.206	0.000

Run 10 Particle size analysis

602447 -10

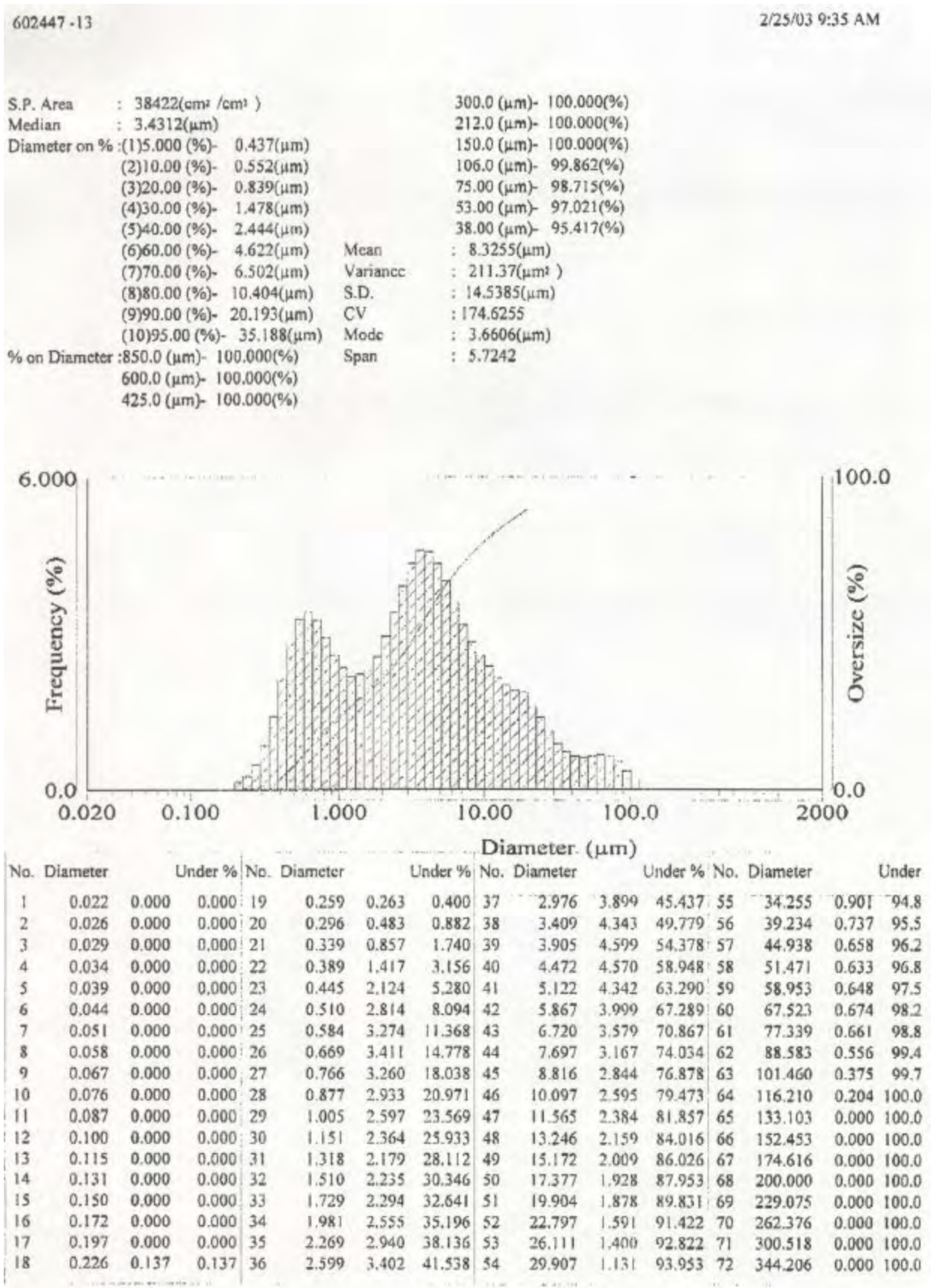
2/25/03 8:53 AM

S.P. Area : 45781(cm^2/cm^3)
 Median : 2.6722(μm)
 Diameter on % : (1)5.000 (%) - 0.410(μm)
 (2)10.00 (%) - 0.510(μm)
 (3)20.00 (%) - 0.719(μm)
 (4)30.00 (%) - 1.096(μm)
 (5)40.00 (%) - 1.841(μm)
 (6)60.00 (%) - 3.471(μm)
 (7)70.00 (%) - 4.344(μm)
 (8)80.00 (%) - 5.489(μm)
 (9)90.00 (%) - 7.407(μm)
 (10)95.00 (%) - 9.281(μm)
 % on Diameter : 850.0 (μm) - 100.000(%)
 600.0 (μm) - 100.000(%)
 425.0 (μm) - 100.000(%)
 300.0 (μm) - 100.000(%)
 212.0 (μm) - 100.000(%)
 150.0 (μm) - 100.000(%)
 106.0 (μm) - 100.000(%)
 75.00 (μm) - 100.000(%)
 53.00 (μm) - 100.000(%)
 38.00 (μm) - 100.000(%)
 Mean : 3.3809(μm)
 Variance : 9.0279(μm^2)
 S.D. : 3.0046(μm)
 CV : 88.8717
 Mode : 4.1761(μm)
 Span : 2.5811



Diameter (μm)			Diameter (μm)			Diameter (μm)			Diameter (μm)		
No.	Diameter	Under %	No.	Diameter	Under %	No.	Diameter	Under %	No.	Diameter	Under %
1	0.022	0.000	19	0.259	0.334	37	2.976	4.737	55	34.255	0.000
2	0.026	0.000	20	0.296	0.607	38	3.409	5.451	56	39.234	0.000
3	0.029	0.000	21	0.339	1.073	39	3.905	5.977	57	44.938	0.000
4	0.034	0.000	22	0.389	1.758	40	4.472	6.120	58	51.471	0.000
5	0.039	0.000	23	0.445	2.610	41	5.122	5.921	59	58.953	0.000
6	0.044	0.000	24	0.510	3.419	42	5.867	5.434	60	67.523	0.000
7	0.051	0.000	25	0.584	3.933	43	6.720	4.684	61	77.339	0.000
8	0.058	0.000	26	0.669	4.056	44	7.697	3.697	62	88.583	0.000
9	0.067	0.000	27	0.766	3.840	45	8.816	3.127	63	101.460	0.000
10	0.076	0.000	28	0.877	3.427	46	10.097	2.190	64	116.210	0.000
11	0.087	0.000	29	1.005	3.014	47	11.565	1.500	65	133.103	0.000
12	0.100	0.000	30	1.151	2.728	48	13.246	0.989	66	152.453	0.000
13	0.115	0.000	31	1.318	2.494	49	15.172	0.624	67	174.616	0.000
14	0.131	0.000	32	1.510	2.559	50	17.377	0.339	68	200.000	0.000
15	0.150	0.000	33	1.729	2.618	51	19.904	0.187	69	229.075	0.000
16	0.172	0.000	34	1.981	2.932	52	22.797	0.000	70	262.376	0.000
17	0.197	0.000	35	2.269	3.420	53	26.111	0.000	71	300.518	0.000
18	0.226	0.173	36	2.599	4.026	54	29.907	0.000	72	344.206	0.000

Run 13 Particle size analysis



APPENDIX B: Detailed Chemical Analyses

*Characterization of High Density sludge from Britannia Beach
November 2003*

Chemical analysis of High Density Sludge (HDS) produced during Pilot Scale ARD treatment in
February 2002 - March 2002 in Britannia Beach, BC, Chemical analysis by CANMET Laboratories in Ottawa, ON

Sample	% Al	% As	% Ca	% Cd	% Co	% CO2	% Cr	% Cu	% Fe	% K	% LOI	% LOM
Run 2	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Run 3	6.44	0.004944	11.4	0.0193	0.0136	nd	0.0018	4.96	3.1	0.0528	30.2	8.2
Run 4	3.2	0.001899	14.3	0.0117	0.0043	7.78	0.0031	2.68	3.59	<.037	40.1	21.1
Run 8	3.63	0.001452	16.4	0.0064	0.0048	nd	0.0033	3.02	3.95	<.039	29.9	8
Run 9	4.19	0.004222	16.1	0.0135	0.0052	nd	0.0034	3.19	4.11	<.038	27.8	5.4
Run 10	5.17	0.002319	14	0.0158	0.0103	nd	0.0016	3.93	1.48	<.039	34.6	11.2
Run 13	5.75	0.000598	14.2	0.0172	0.113	10.4	0.0024	4.29	1.82	<.037	29.9	5

Sample	% Mg	% Mn	% Na	% Ni	tonns Np	% Pb	% S	% Si	% SO4	% Zn	Totals %
Run 2	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	
Run 3	3.31	0.769	0.04	0.0098	311	<.024	2.92	4.45	2.9	4.35	83.121644
Run 4	2.33	0.276	0.02	0.0076	311	<.024	4.91	1.67	4.9	3.29	110.1924
Run 8	2.7	0.299	0.02	0.0084	396	<.025	5.72	1.9	5.7	3.63	84.927352
Run 9	2.75	0.333	0.02	0.0087	nd	<.024	6.08	n/a	6.1	3.77	79.857622
Run 10	3.11	0.634	0.05	0.0077	382	<.025	3.37	3.27	3.4	3.61	87.858319
Run 13	3.43	0.696	0.04	0.0084	369	<.024	3.43	3.71	3.4	3.99	90.235098

Whole Rock analysis of HDS from Britannia Beach, BC February 2002 - March 2002

Data Supplied by Environment Canada.

Certificate Number	Sample Name	WRA SiO2 %	WRA Al2O3 %	WRA Fe2O3 %	WRA CaO %	WRA MgO %	WRA K2O %	WRA TiO2 %	WRA MnO %	WRA P2O5 %	WRA Sr ppm	WRA LOI %	WRA Total %
2V0137PA	ClarU/F Feb10/02 23	8.89	12.35	2.80	17.26	5.45	0.05	0.02	0.93	0.28	460.00	30.23	78.30
2V0137PA	ClarU/F Feb15/02 11	9.29	12.69	4.21	16.55	5.39	0.05	0.02	0.94	0.37	460.00	29.98	79.52
2V0137PA	ClarU/F Feb18/02 11	7.82	10.93	3.22	16.27	4.90	<0.01	0.02	0.82	0.28	410.00	34.26	78.55
2V0137PA	ClarU/F Mar04/02 23	4.37	7.79	5.01	25.36	4.76	<0.01	0.01	0.39	0.18	480.00	28.82	76.73
2V0137PA	ClarU/F Mar09/02 23	4.18	7.71	5.94	25.79	4.55	<0.01	0.01	0.41	0.18	500.00	27.96	76.78
2V0137PA	*DUP ClarU/F Feb10/02 23	8.52	12.20	2.66	16.63	5.25	<0.01	0.02	0.89	0.29	440.00	30.33	76.84
2V0137PA	*STD 93-1	53.41	10.17	9.53	15.81	3.57	2.43	0.40	0.29	0.35	400.00	3.67	99.68
2V0137PA	*STD SY-4	53.28	22.31	6.59	8.53	0.56	1.77	0.32	0.11	0.15	1270.00	4.56	98.31
2V0137PA	*BLANK	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<10		

Pilot Plant Testing of High Density Sludge Process

Britannia Mine AMD Treatment

January to March 2002

Data Supplied by Environment Canada

Clarifier Underflow Sludge Analysis by ICP-OES

Sample Name:		Clar. U/F Feb. 10, 2002 Run #3	Clar. U/F Feb. 15, 2002 Run #4	Clar. U/F Feb. 18, 2002 Run #4	Clar. U/F Mar. 4, 2002 Run #8	Clar. U/F Mar. 9, 2002 Run #9
Ag	ppm	1.6	1.6	1.4	1.0	0.8
Al	%	6.36	6.4	5.67	3.95	3.94
As	ppm	5	10	10	<5	5
Ba	ppm	20	30	20	<10	10
Be	ppm	3	3	2.5	2	2
Bi	ppm	<5	<5	<5	<5	<5
Ca	%	11.67	10.91	11.26	14.96	>15
Cd	ppm	>100	>100	96	38	64
Co	ppm	115	117	103	44	44
Cr	ppm	11	12	10	21	27
Cu	ppm	>10000	>10000	>10000	>10000	>10000
Fe	%	2.08	3.04	2.44	3.59	4.25
K	%	0.01	0.01	0.01	0.01	0.01
Mg	%	3.39	3.29	3.1	2.87	2.79
Mn	ppm	6720	6705	6070	2760	2925
Mo	ppm	<2	<2	<2	<2	<2
Na	%	0.01	0.01	0.01	0.01	0.01
Ni	ppm	94	96	85	79	90
P	ppm	5690	6000	5330	3500	3750
Pb	ppm	134	112	96	52	52
Sb	ppm	15	10	10	10	10
Sc	ppm	5	5	4	2	2
Sn	ppm	<10	<10	<10	<10	<10
Sr	ppm	540	530	491	543	553

Ti	%	0.0	0.01	0.01	<0.01	<0.01
V	ppm	14	16	14	22	24
W	ppm	820	840	760	710	760
Y	ppm	120	121	107	42	43
Zn	ppm	>10000	>10000	>10000	>10000	>10000
Zr	ppm	2	3	2	3	3

Pilot Plant Testing of High Density Sludge Process

Britannia Mine AMD Treatment

January to March 2002

Data Supplied by Environment Canada

Clarifier Underflow Sludge Analysis by AA Assay

Sample Name:	Clar. U/F	Clar. U/F	Clar. U/F	Clar. U/F	Clar. U/F	Clar. U/F	
	Feb. 10, 2002	Feb. 15, 2002	Feb. 18, 2002	Mar. 4, 2002	Mar. 9, 2002	Feb. 10, 2002	
	Run #3	Run #4	Run #4	Run #8	Run #9	Run #3-Duplicate	
Al	%	7.96	6.08	6.28	4.55	4.60	7.17
Cd	%	0.021	0.021	0.019	0.008	0.013	0.021
Cu	%	5.30	5.36	4.78	3.30	3.64	5.32
Fe	%	2.56	2.60	2.51	3.80	4.59	2.14
Mg	%	4.09	2.95	3.33	3.14	3.08	3.68
Zn	%	4.51	4.70	4.16	3.92	4.25	4.48
S	%	3.15	2.71	2.93	5.47	5.52	3.13
		27.59	24.42	24.01	24.19	25.69	25.94

Note: S% is sulfur total as sulfate

Run 4 Data

Table B-1: Feed data; overflow data and solids data for Run 4

	Feed (page 11- CEMI)	Out-over quality (Table3.3- CEMI)	Sludge analysis (Table3.7 – CEMI)	Sludge analysis (CANMET)
			Feb 18/02	Feb 27/03
pH	3.9 – 4.4	9.5		
Retention time	58 min	58 min		
Al	22 mg/L	0.45 mg/L	6.28 %	3.2 %
Cu	19 mg/L	<0.005 mg/L	4.78 %	2.68 %
Fe	0.3 mg/L	N/A	2.51 %	3.59 %
Mg	53 mg/L	N/A	3.33 %	2.33 %
Mn	3 mg/L	0.104 mg/L	N/A	0.28 %
Zn	16 mg/L	0.016 mg/L	4.16 %	3.29 %
Cd			0.019 %	0.0117 %
S			3.15 %	4.91 %

Run 9 Data

Table B-2: Feed data; overflow data and solids data for Run 9

	Feed (page 14- CEMI)	Out-over quality (Table3.4- CEMI)	Sludge analysis (Table3.7 – CEMI)	Sludge analysis (CANMET)
			Mar 9/02	Feb/03
pH	2.4	9.5		10.13 *
Conductivity				1.97 mS *
ORP				-39 *
% Solids			15.0 %	6.4433 *
Retention time	56 min	56 min		
Al	45 mg/L	0.52 mg/L	4.6 %	4.19 %
Cu	40 mg/L	0.025 mg/L	3.64 %	3.19 %
Fe	47 mg/L	N/A	4.59 %	4.11 %
Mg	110 mg/L	N/A	3.08 %	2.75 %
Mn	ND	0.109 mg/L	N/A	0.33 %
Zn	47 mg/L	0.046 mg/L	4.25 %	3.77 %
Cd	0.31 mg/L	<0.002	0.013 %	0.0135 %
S			5.52 %	6.08 %

*- Measurement obtained on slurry

N/A –Not available

ND – Not detected

Run 13 Data – Maybe Run 12

Table B-3: Feed data; overflow data and solids data for Run 13

	Feed (page 14- CEMI)	Out-over quality (Table3.4- CEMI)	Sludge analysis (Table3.7 – CEMI)	Sludge analysis (CANMET)
				Feb/03
pH	5.3	9.5	N/A	10.03 *
Conductivity			N/A	1.77 mS *
ORP			N/A	- 4 *
% Solids			N/A	15.2806 *
Retention time	56 min	56 min		
Al	22 mg/L	0.55 mg/L	N/A	5.75 %
Cu	19 mg/L	0.017 mg/L	N/A	4.29 %
Fe	0.3 mg/L	N/A	N/A	1.82 %
Mg	53 mg/L	N/A	N/A	3.43 %
Mn	3 mg/L	0.306 mg/L	N/A	0.7 %
Zn	16 mg/L	0.026 mg/L	N/A	3.99 %
Cd			N/A	0.0172 %
S			N/A	3.43 %

APPENDIX C: Detailed Mineralogical Analyses

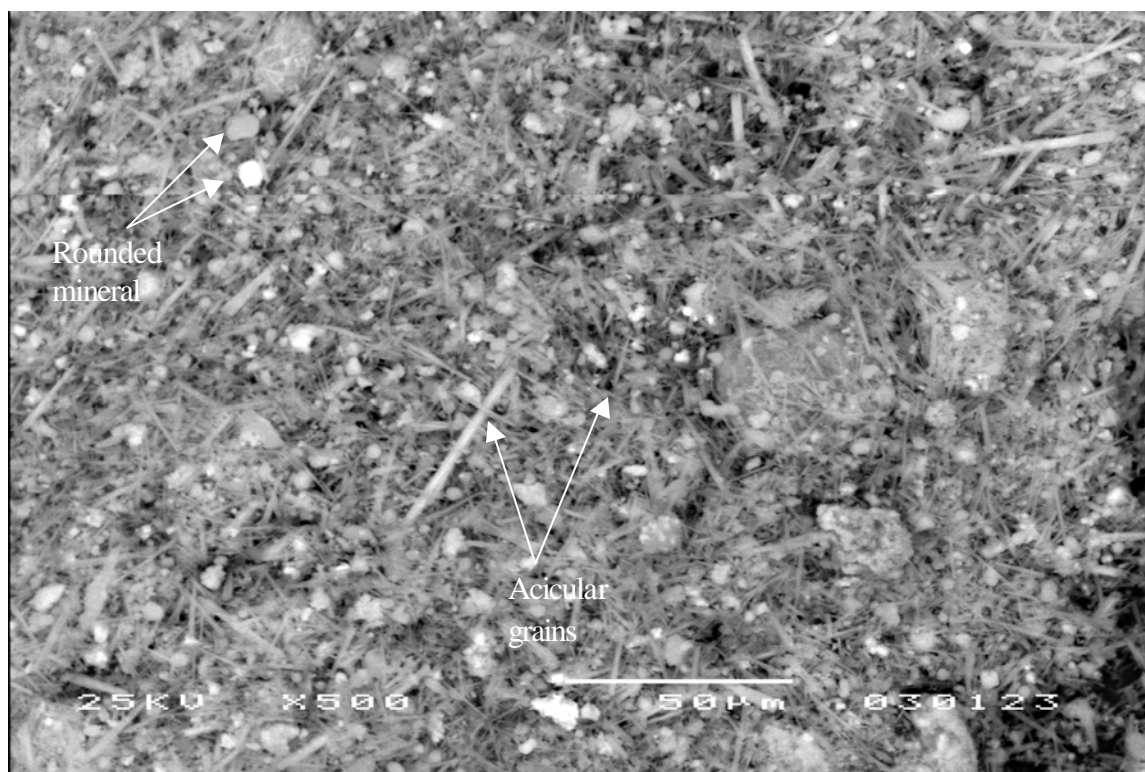


Image 1-1: Backscatter image of Run 13

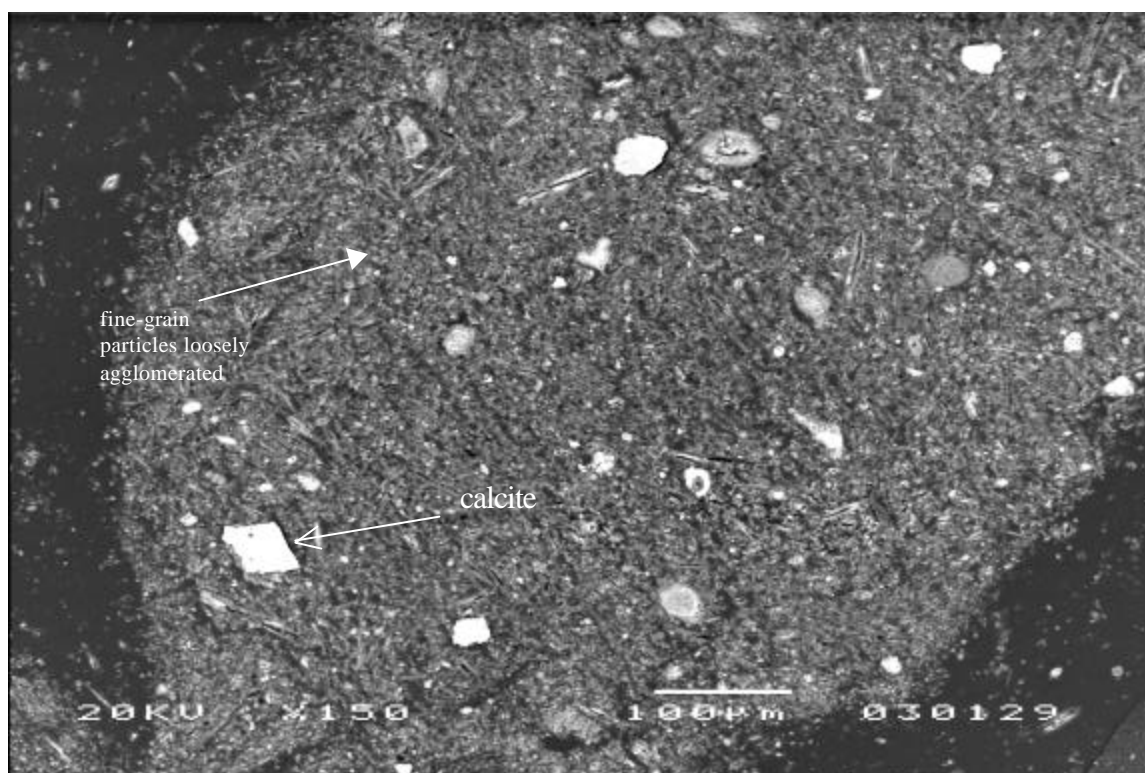


Image 1-2: BEI image of Run 4 showing loose agglomeration of fine mineral grains.

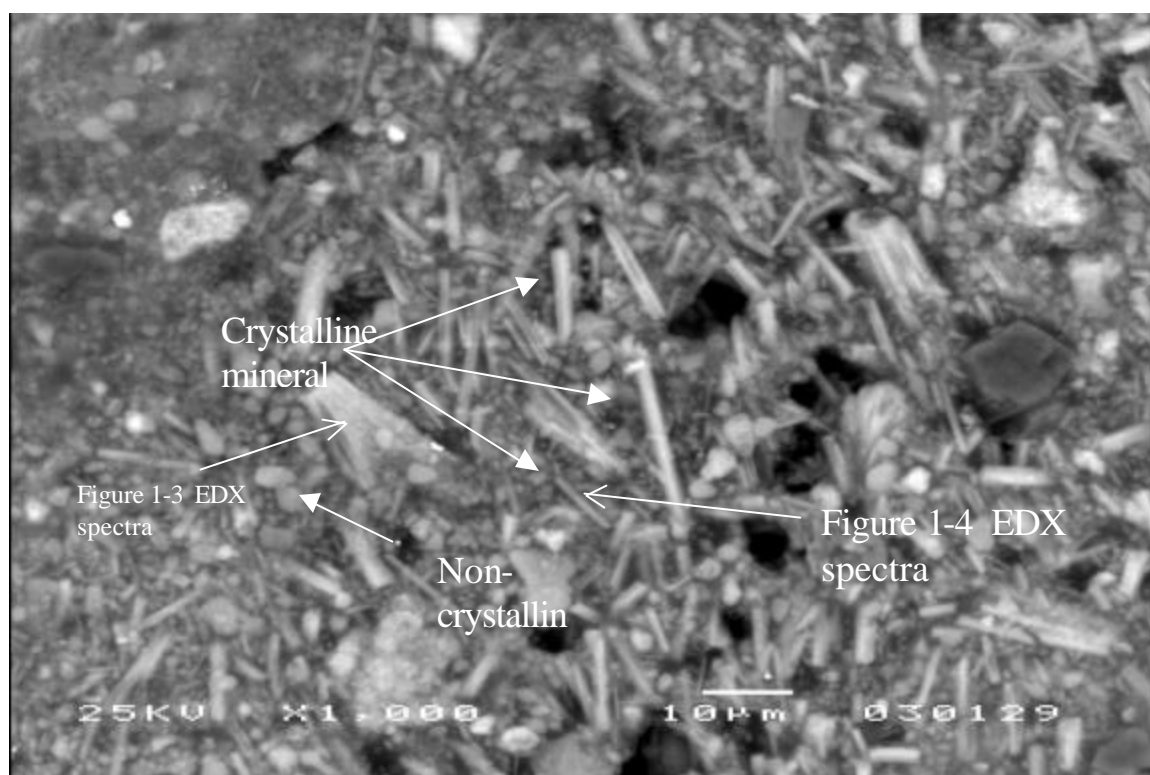


Image 1-3: Run 4 BEI showing crystalline and non-crystalline grains

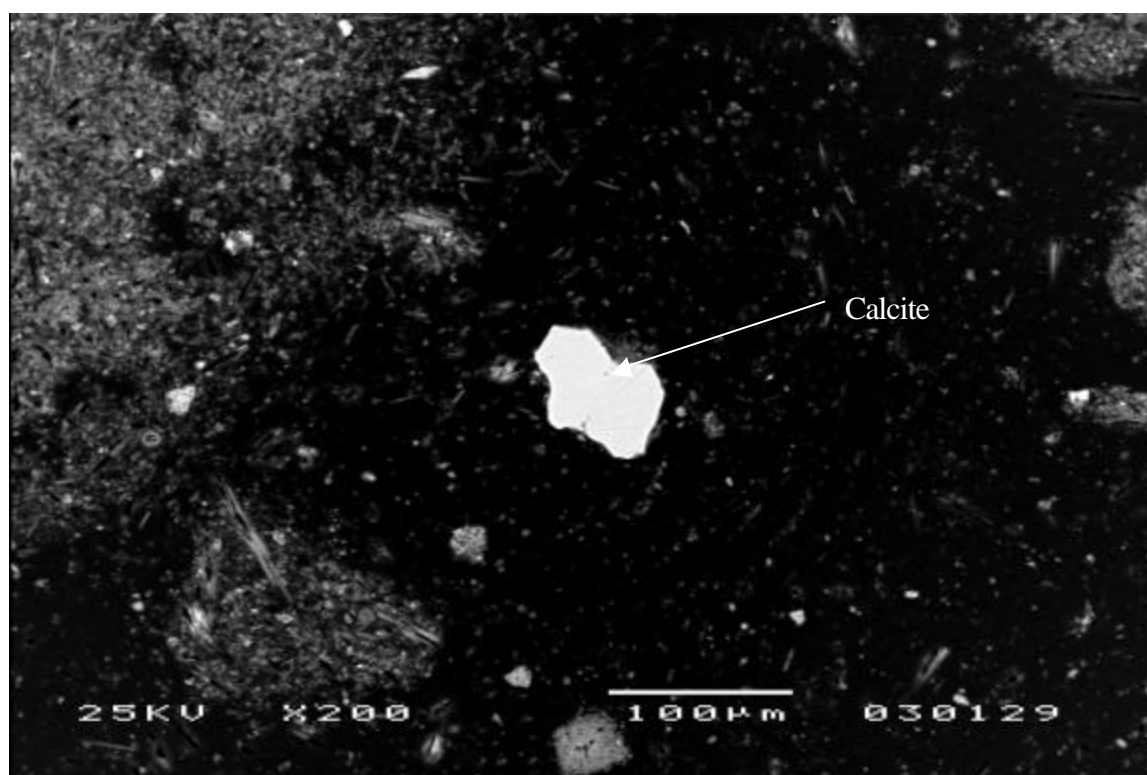


Image 1-4 : Run 4 BEI showing pure calcite grain

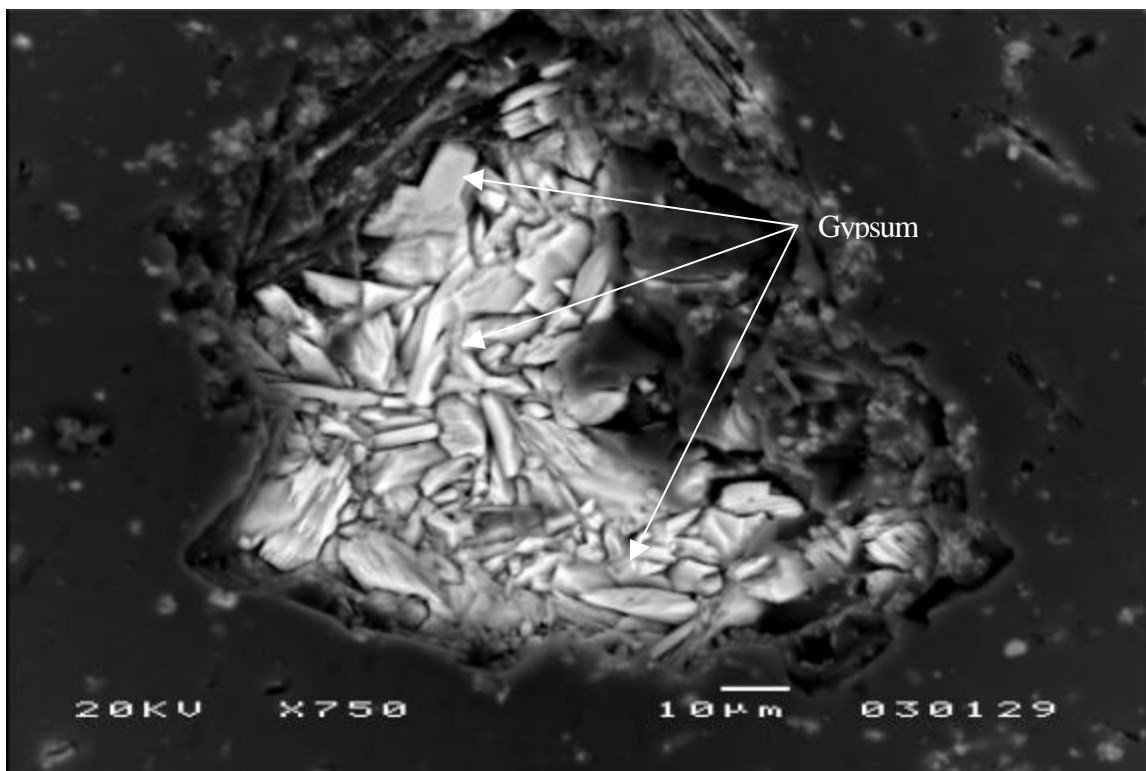


Image 1-5 : Run 4 BEI showing pure gypsum

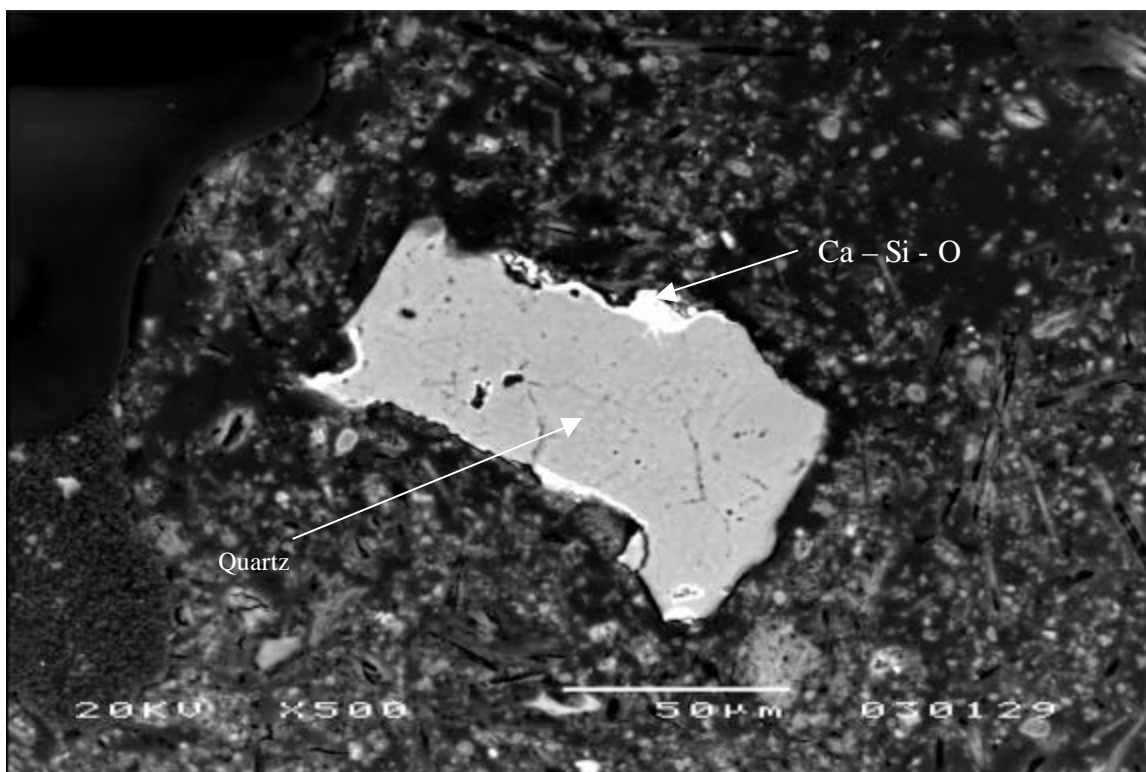


Image 1-6: Run 4 BEI showing large quartz crystal hosted within larger agglomerate

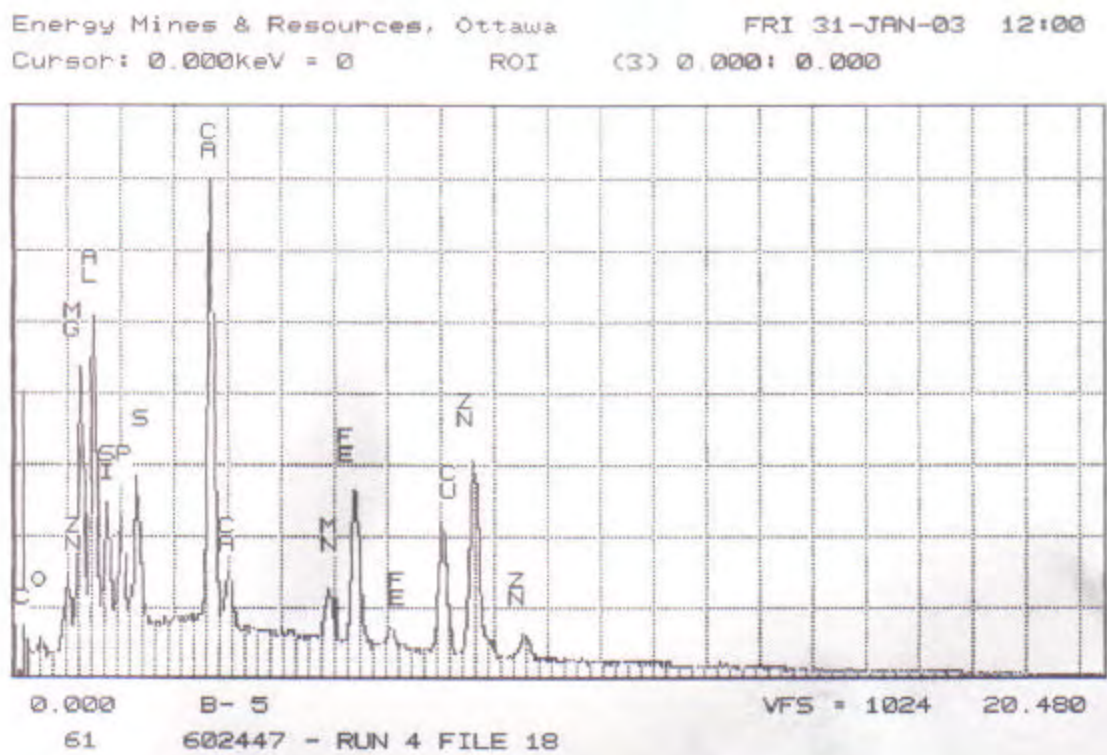


Figure 1-1: EDX spectrum showing typical composition of sample: the elements Ca, S, P, Si, Mg, Mn, Zn, Cu, and Fe are identified.

Energy Mines & Resources, Ottawa FRI 31-JAN-03 11:59
 Cursor: 0.000keV = 0 ROI (3) 0.000: 0.000

Ca₆
 S₃
 Al₂
 Background

0.000 B-5 VFS = 2048 20.480

C-6

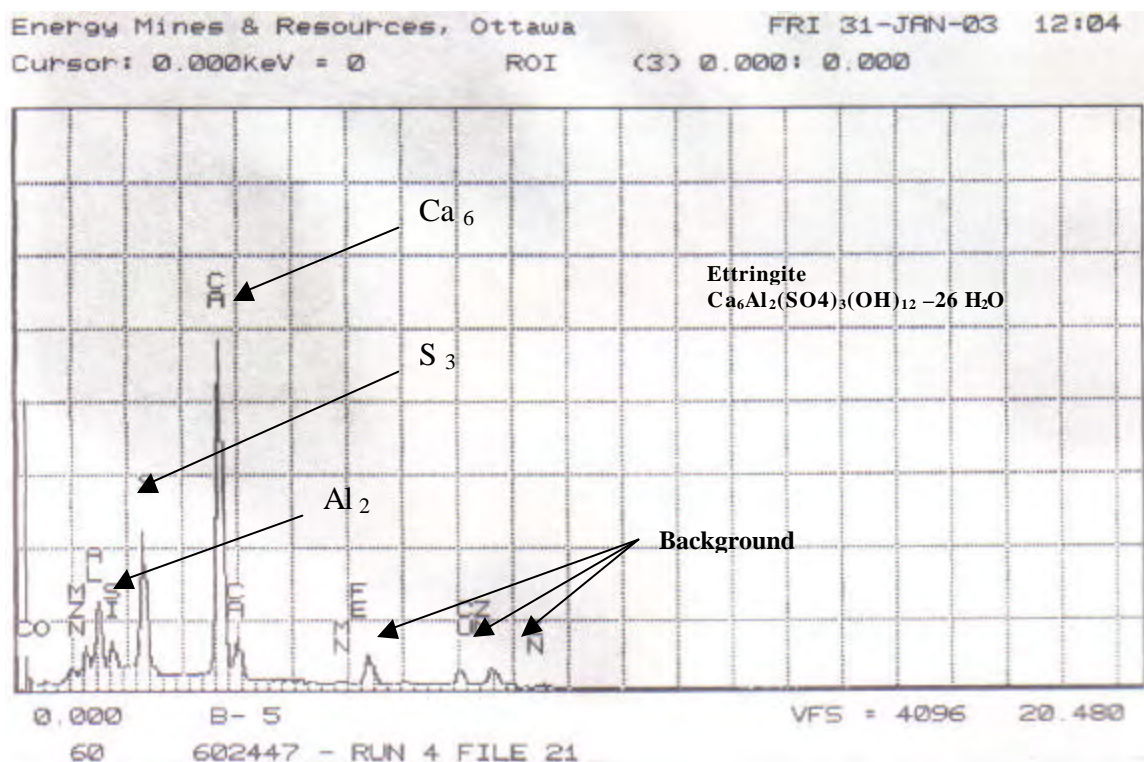


Figure 1-3: Ca:S:Al with background Fe, Cu, Zn and Mn (Image 1-3)

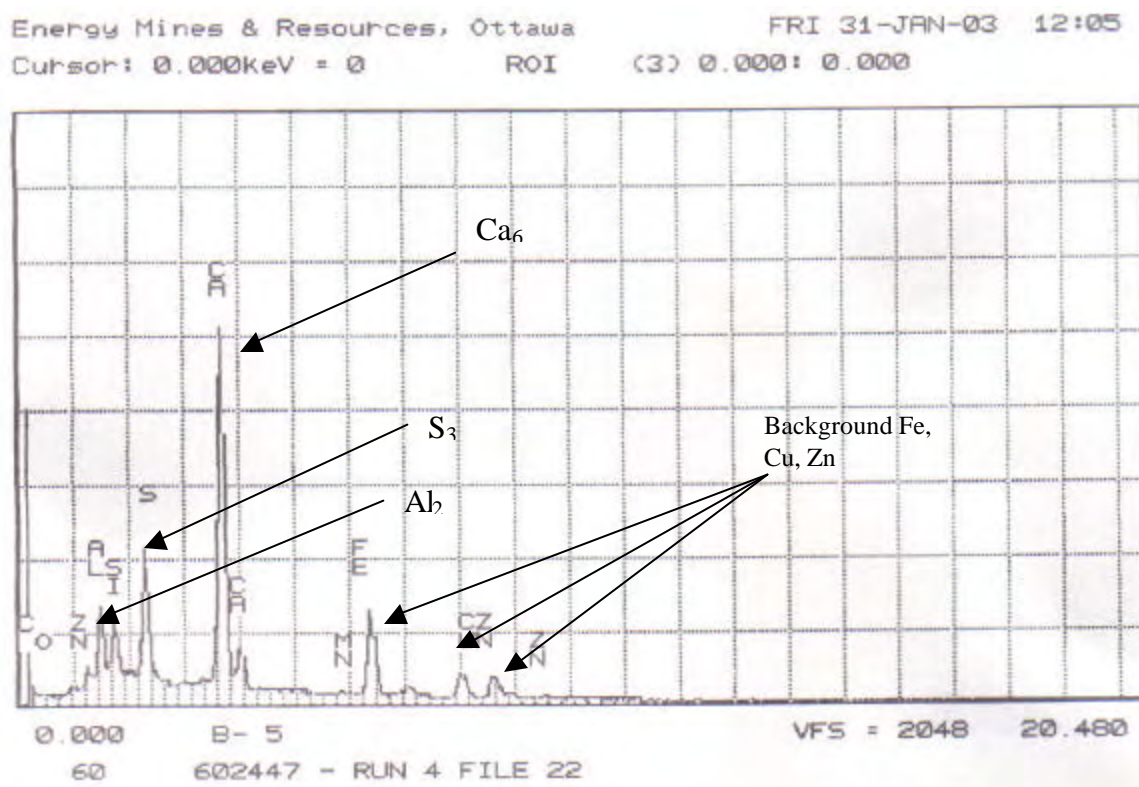


Figure 1-4: Ca : S : Al ratio similar to Figure 1-2 & 1-3. Background elements Fe, Cu and Zn differ between samples. (Image 1-3)

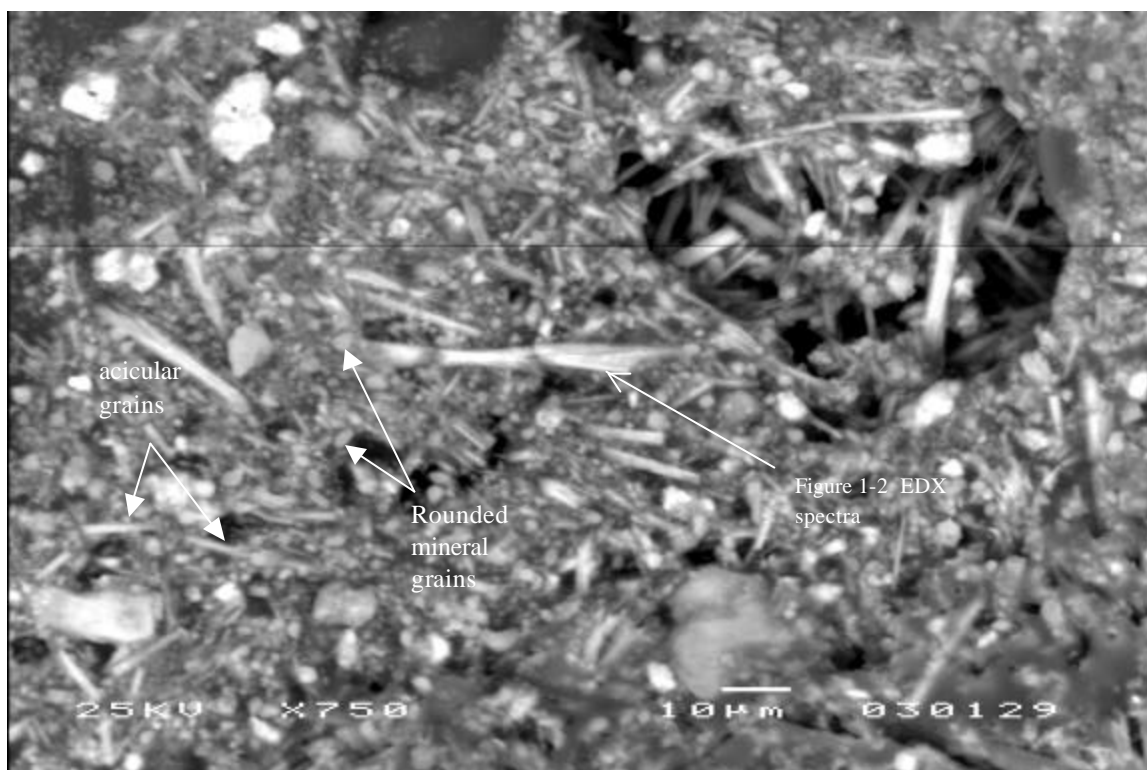


Image 1- 7 : BEI of Run 4 showing acicular and rounded mineral grains. Indicated is the EDX spot analysis location.

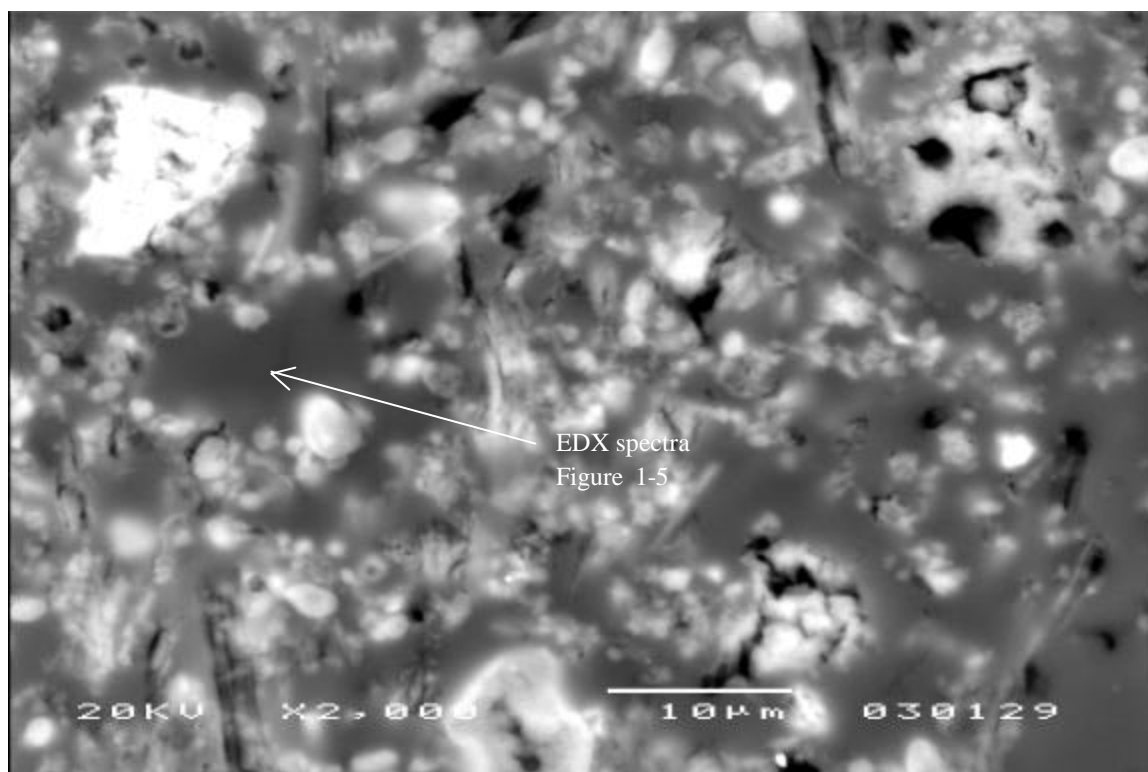


Image 1- 8: Typical appearance of the matrix. Indicated is the EDX spot analysis location.

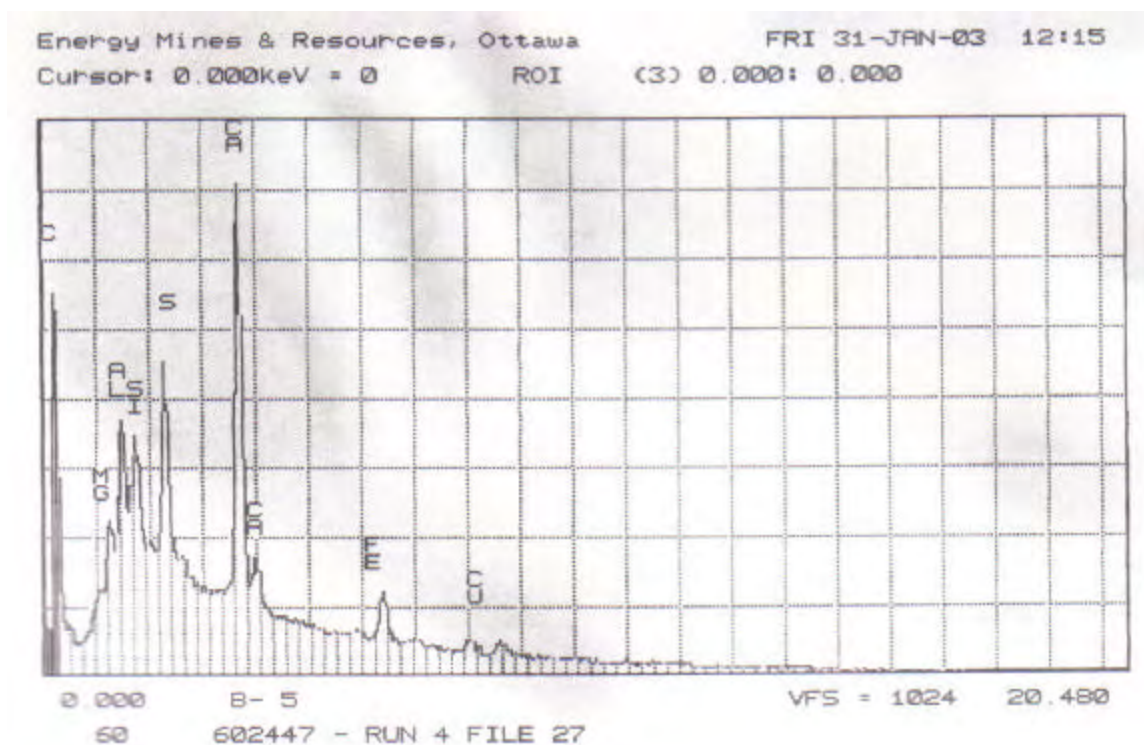


Figure 1-5: EDX spectrum from Image 1-8 illustrating chemical composition of matrix

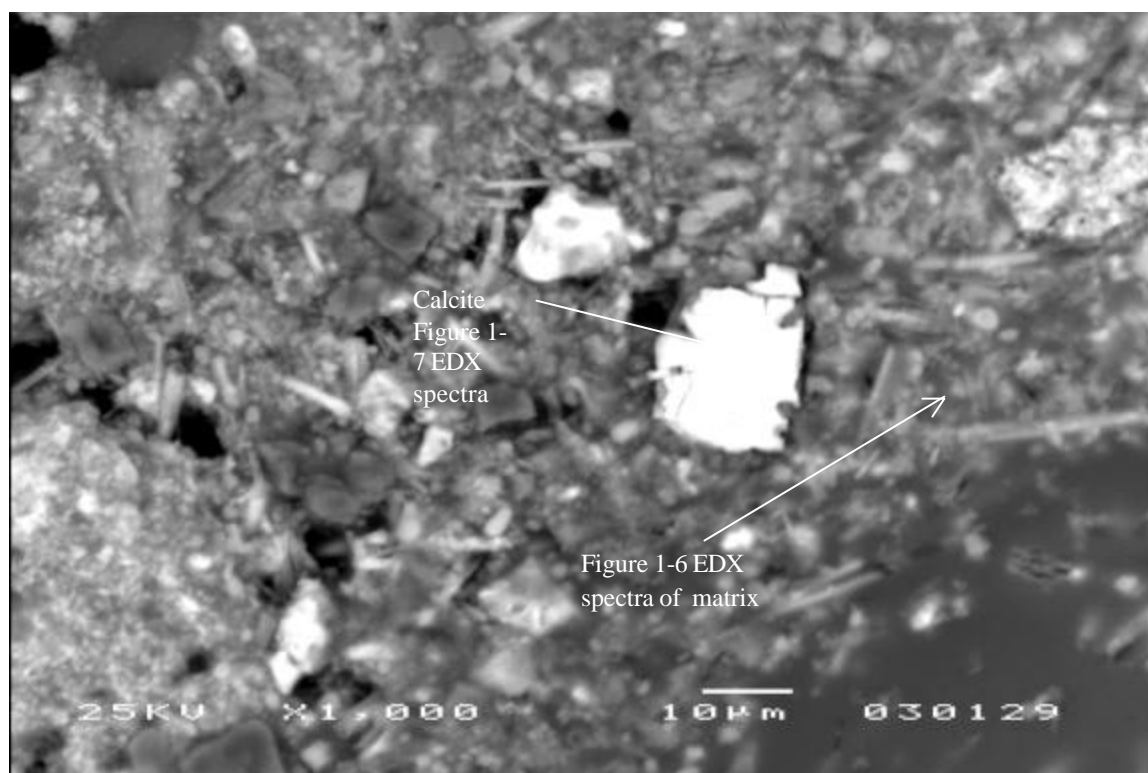


Image 1-9 : Calcite as part of agglomerate. EDX spot analysis for Figure 1-6 & 1-7

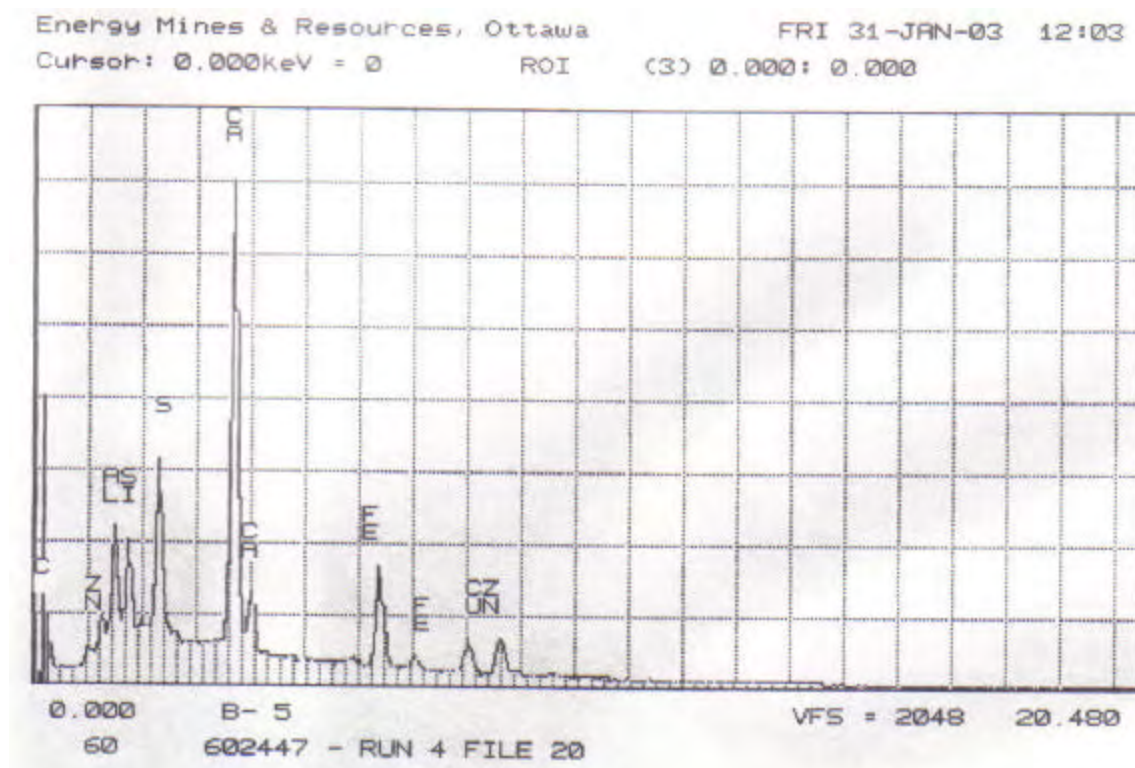


Figure 1-6: Spectrum from Image 1-9 showing chemical composition of the matrix

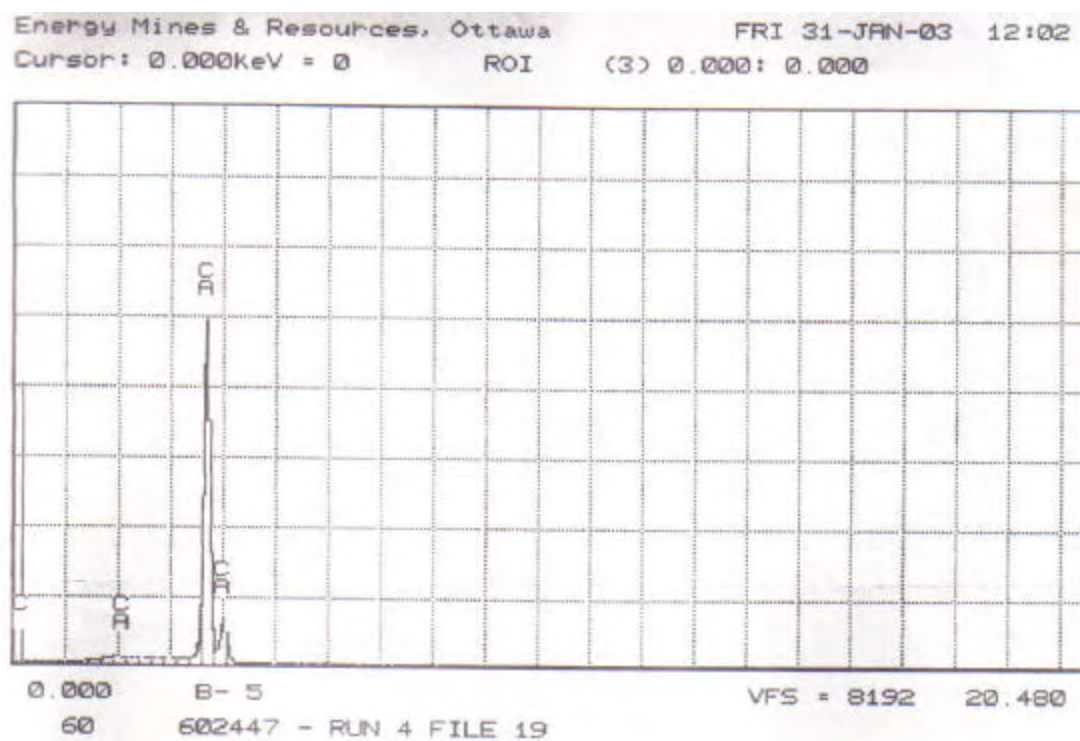


Figure 1-7: EDX spectrum of calcite in Image 1-9



Image 1-10: BEI Run 9 showing the presence of acicular grains, rounded mineral grains and tabular mineral grains.

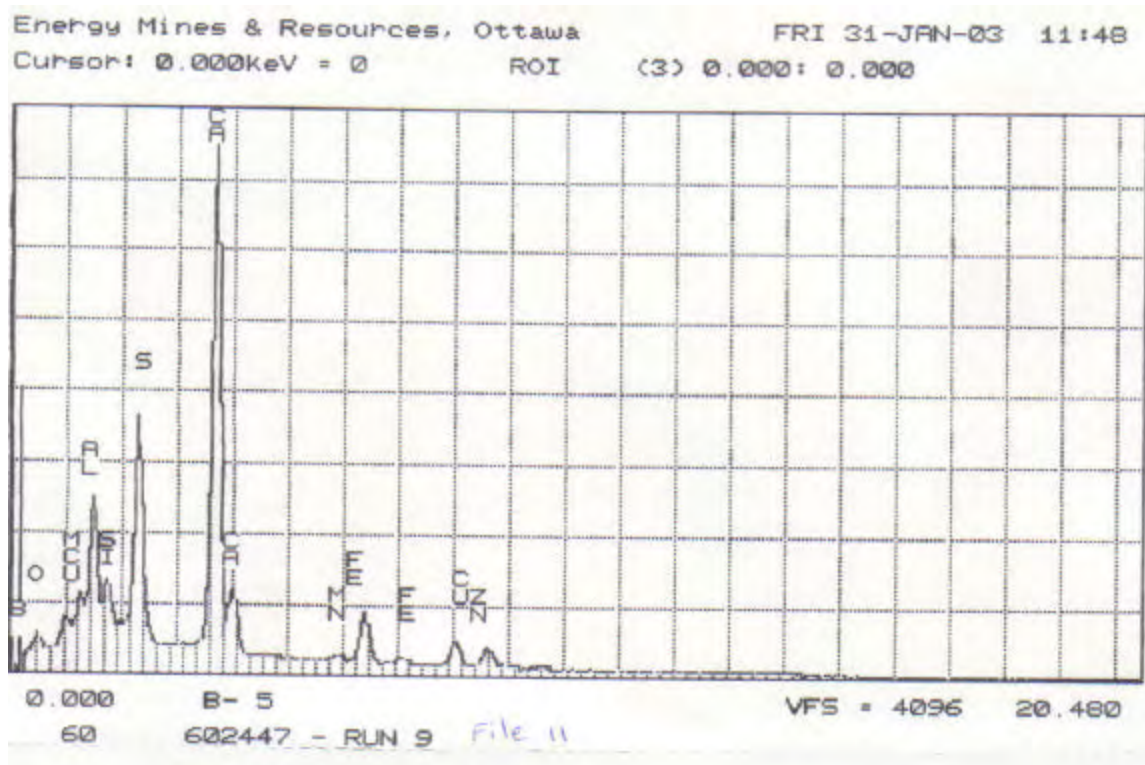
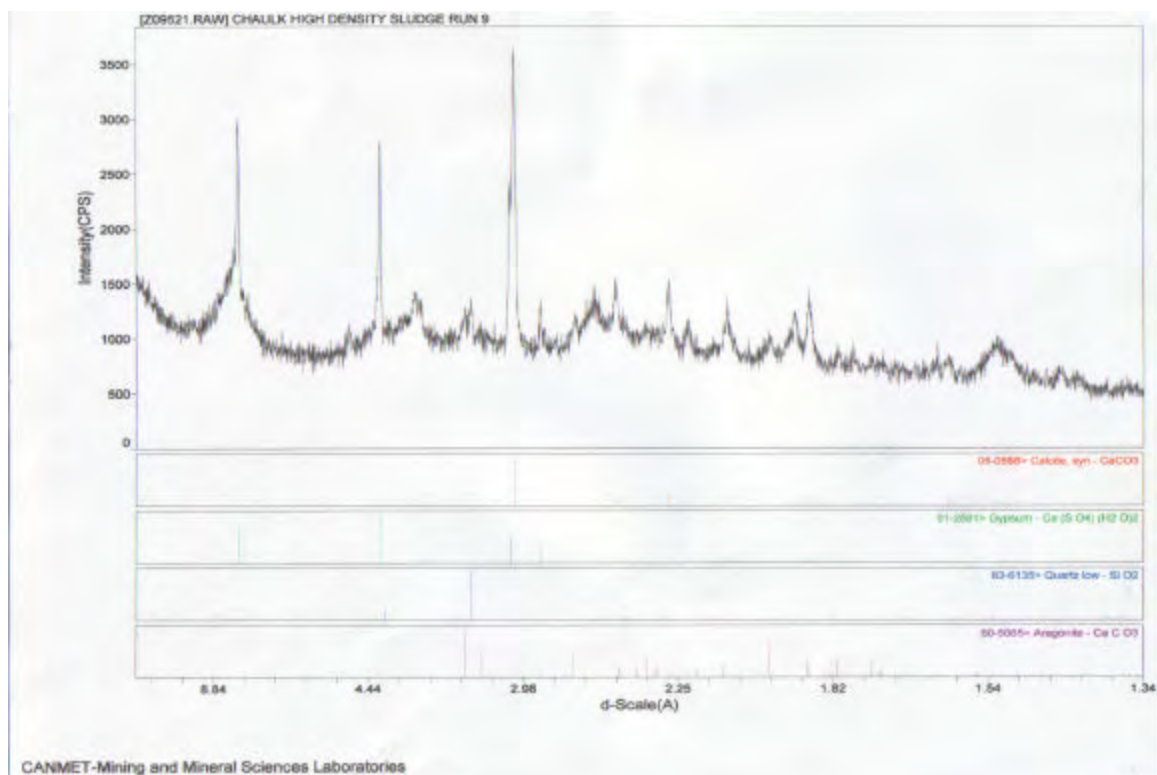


FIGURE 1-8: EDX SPECTRUM SHOWING COMPOSITION OF ACICULAR



XRD 1-2: Run 9 X- Ray diffractogram. Calcite, gypsum, quartz and aragonite are identified.

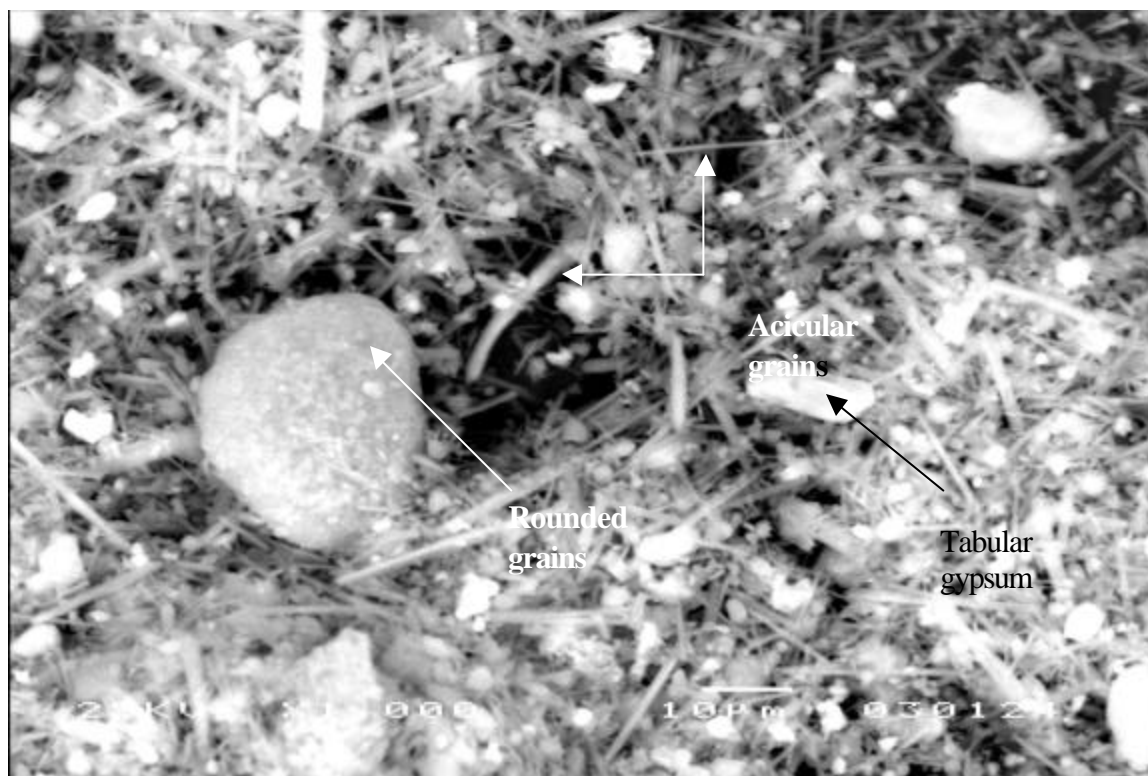


Image 1-11: Rounded mineral grains and acicular grains, also visible tabular gypsum

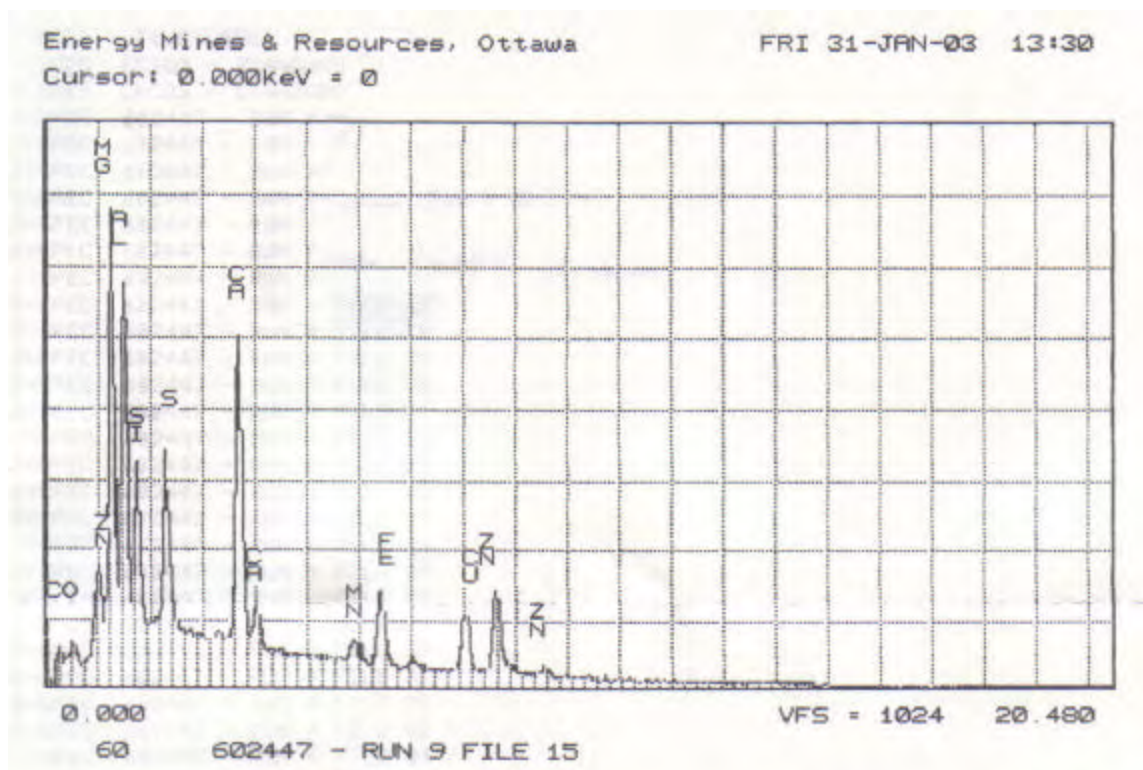


Figure 1-9: Spectrum of rounded minerals, Image 1-11

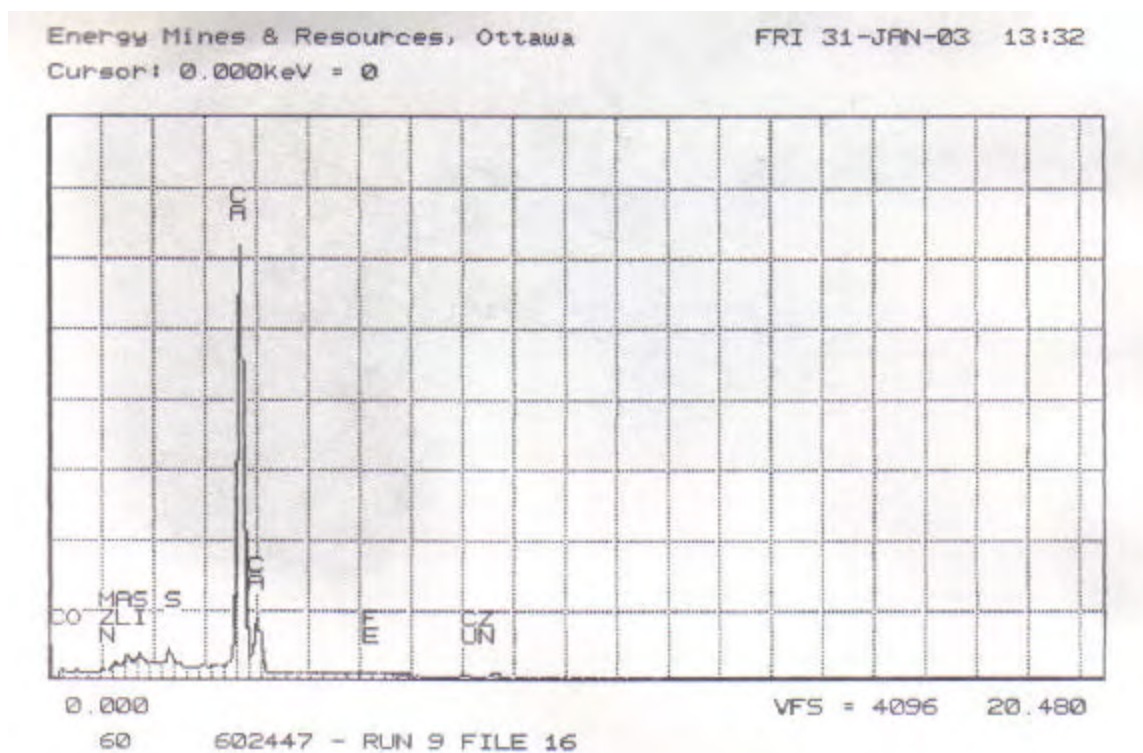


Figure 1-10: Spectrum of acicular grains, Image 1-11

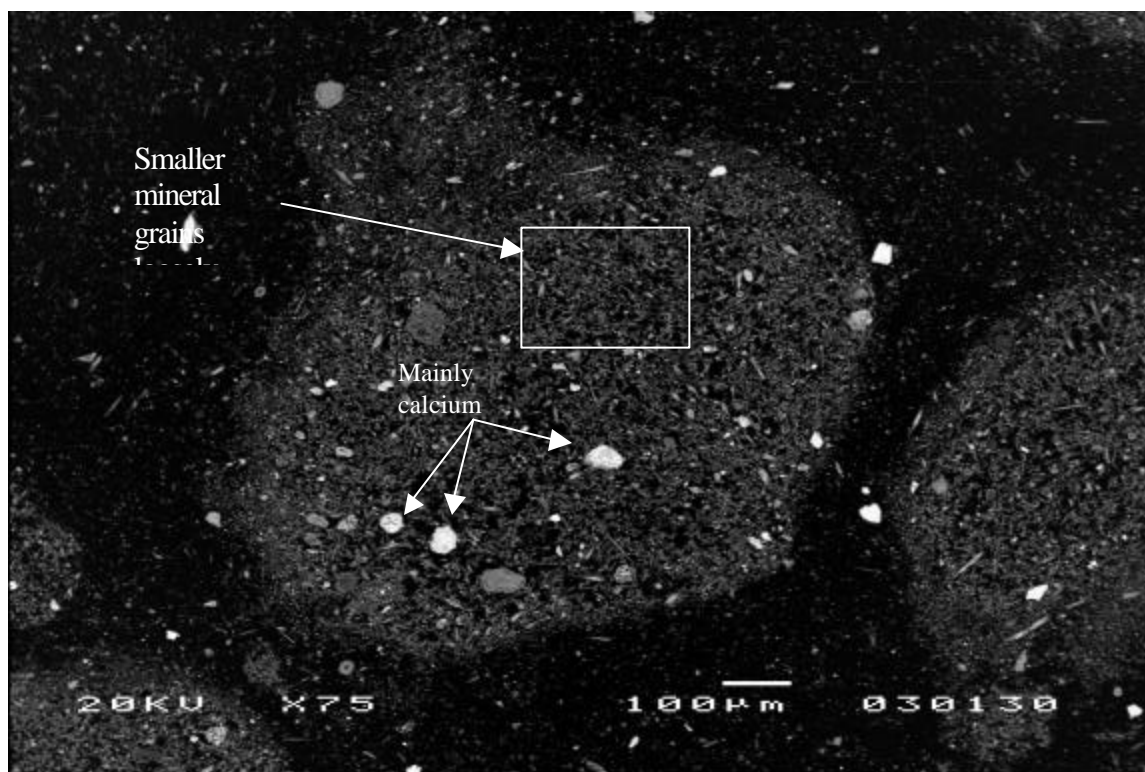


Image 1-12: Run 9 loose agglomeration of smaller individual mineral grains

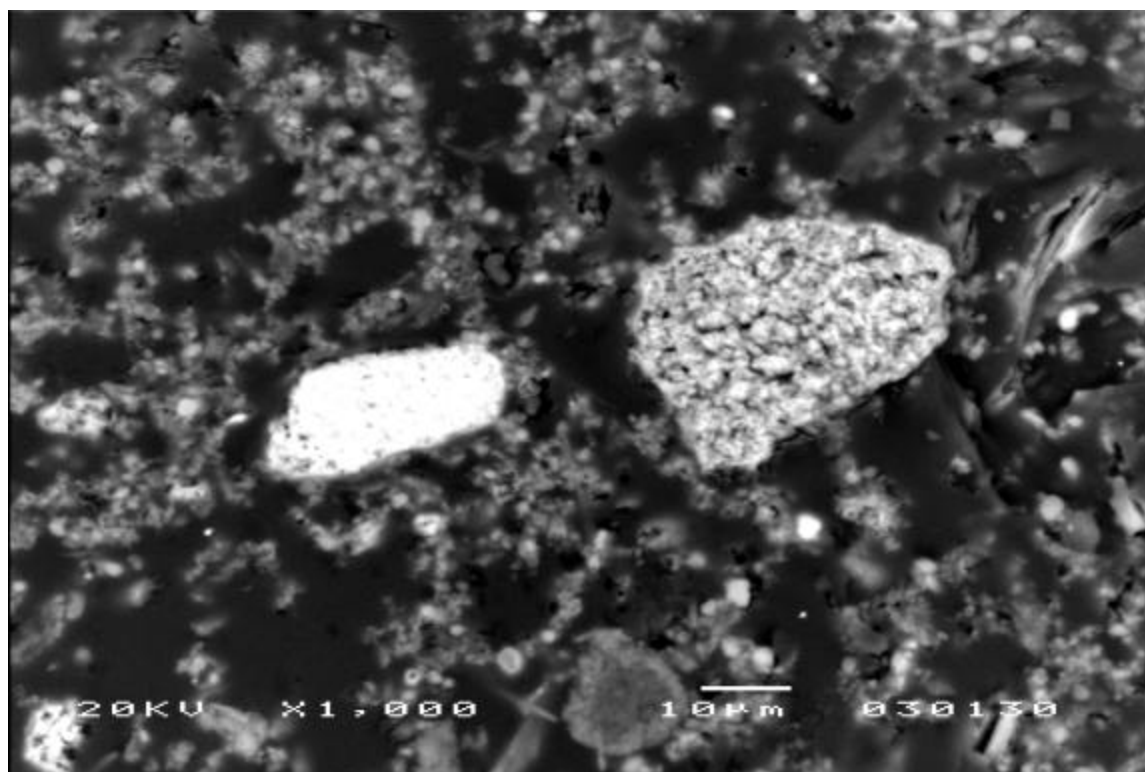


Image 1-13 : Run 9 showing two structures with mainly calcium see Figure 1-12

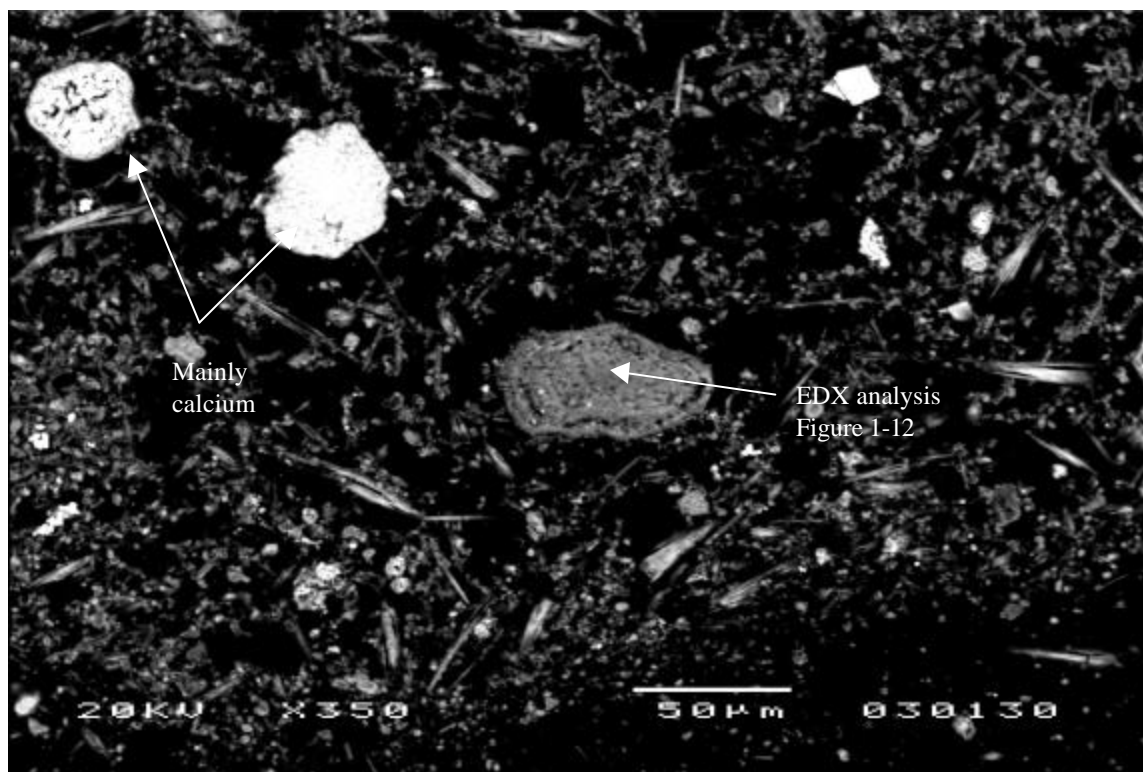


Image 1-14: Run 9 structures containing calcium as the main component.

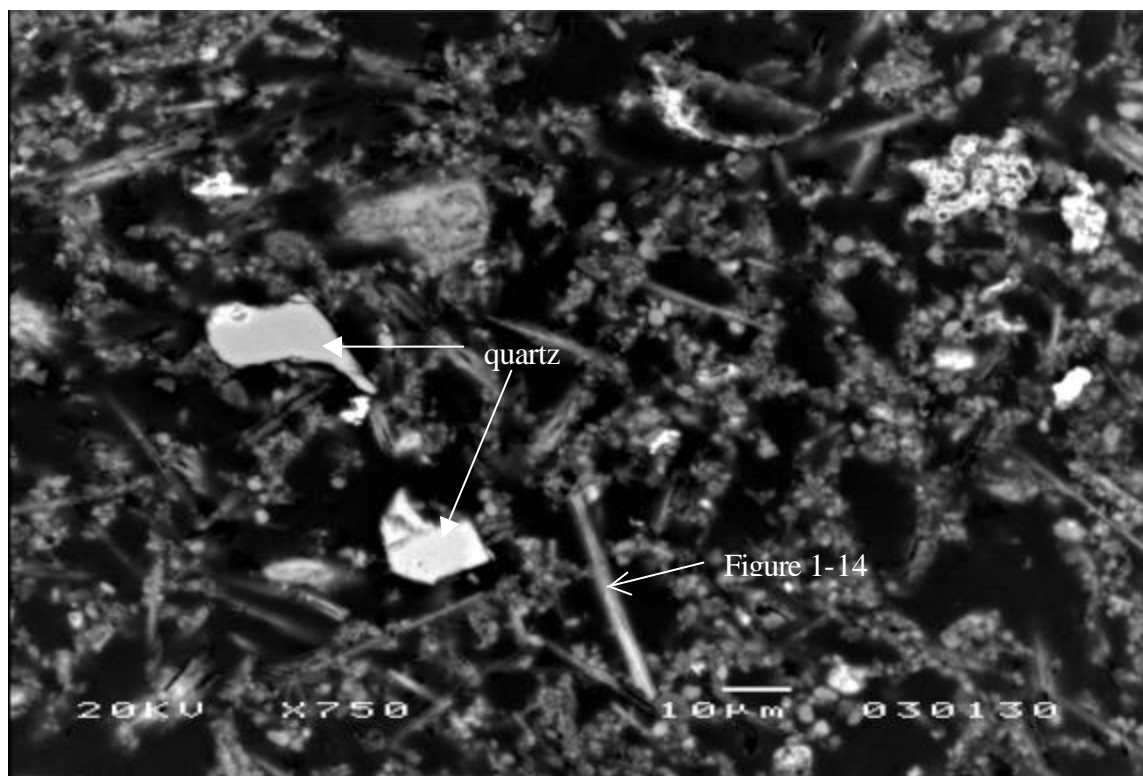


Image 1-15: Run 9 quartz (Figure 1-13)

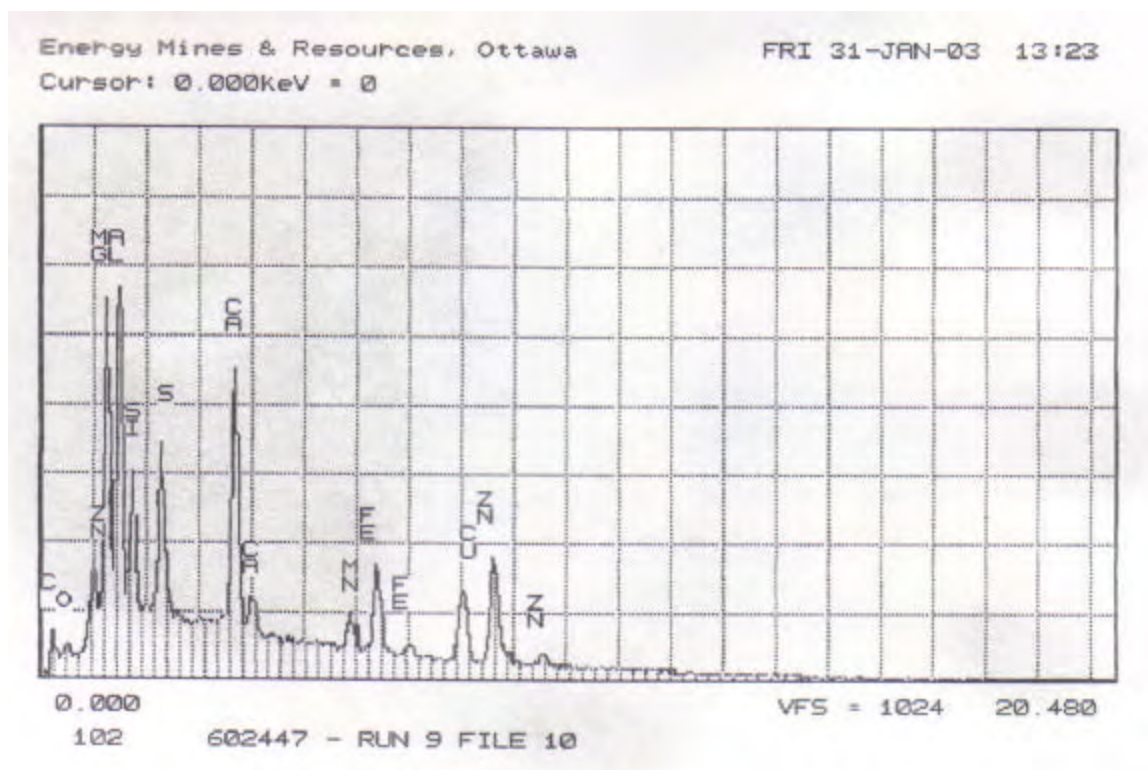


Figure 1-11: Spot analysis from Image 1-14

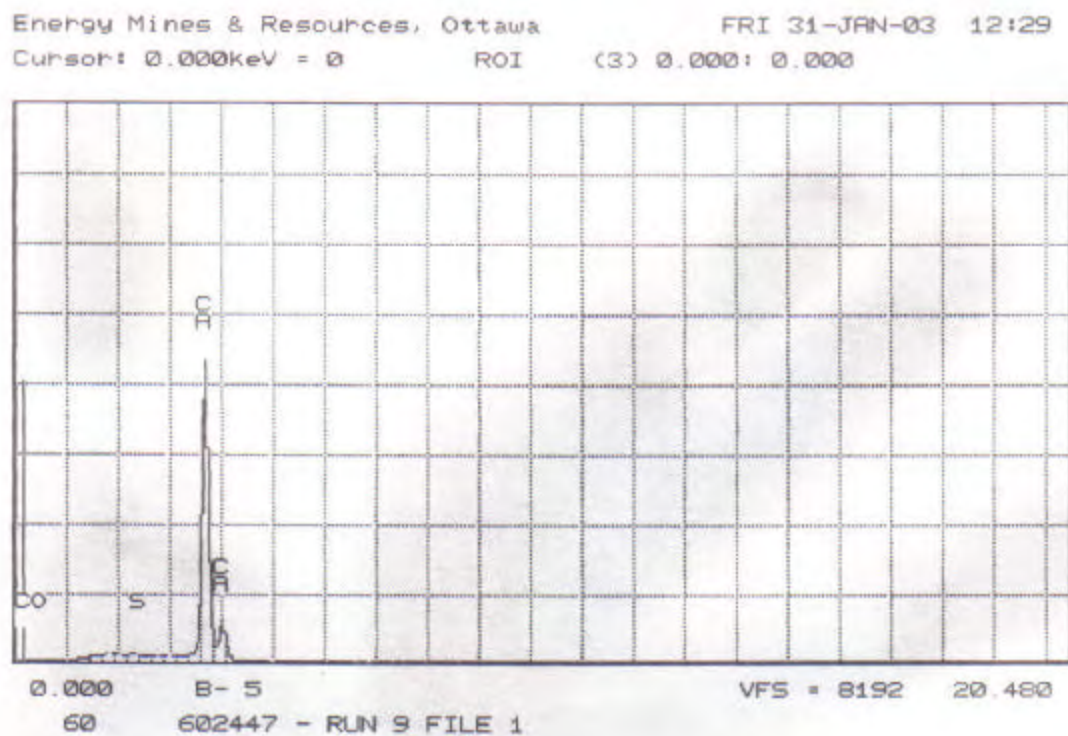


Figure 1-12: EDX spot analysis shown in Image 1-15

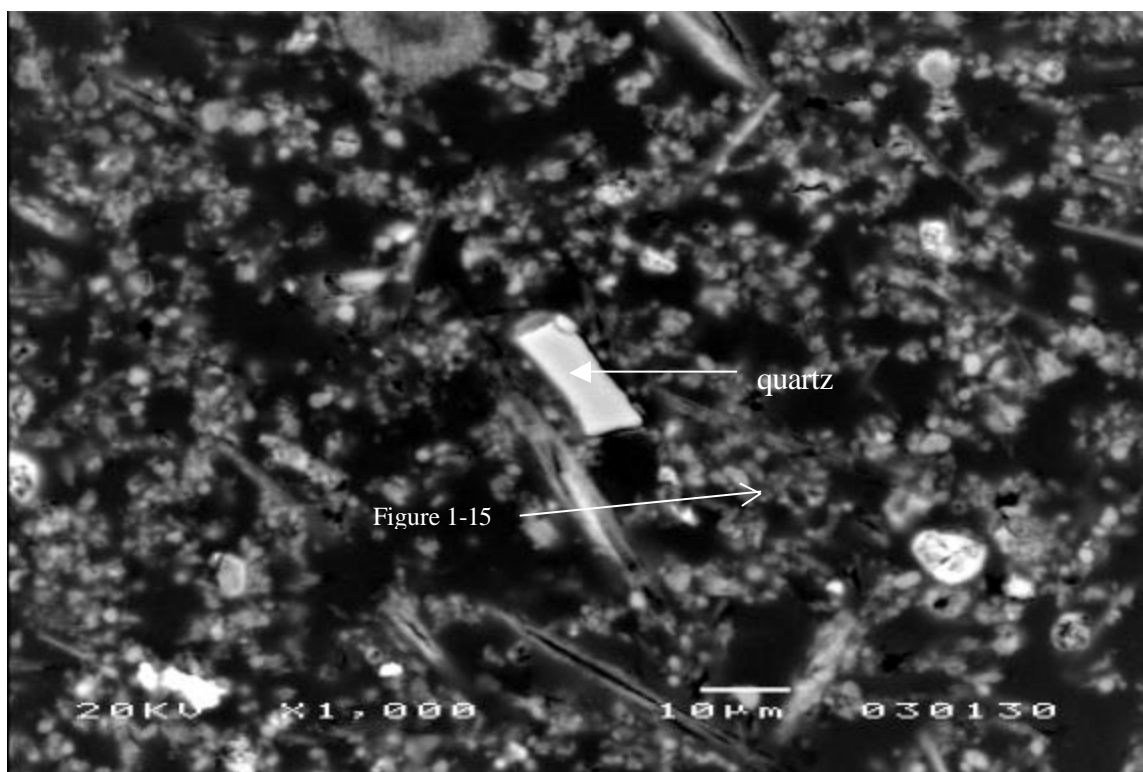


Image 1-16 : Run 9 shows quartz present in sample

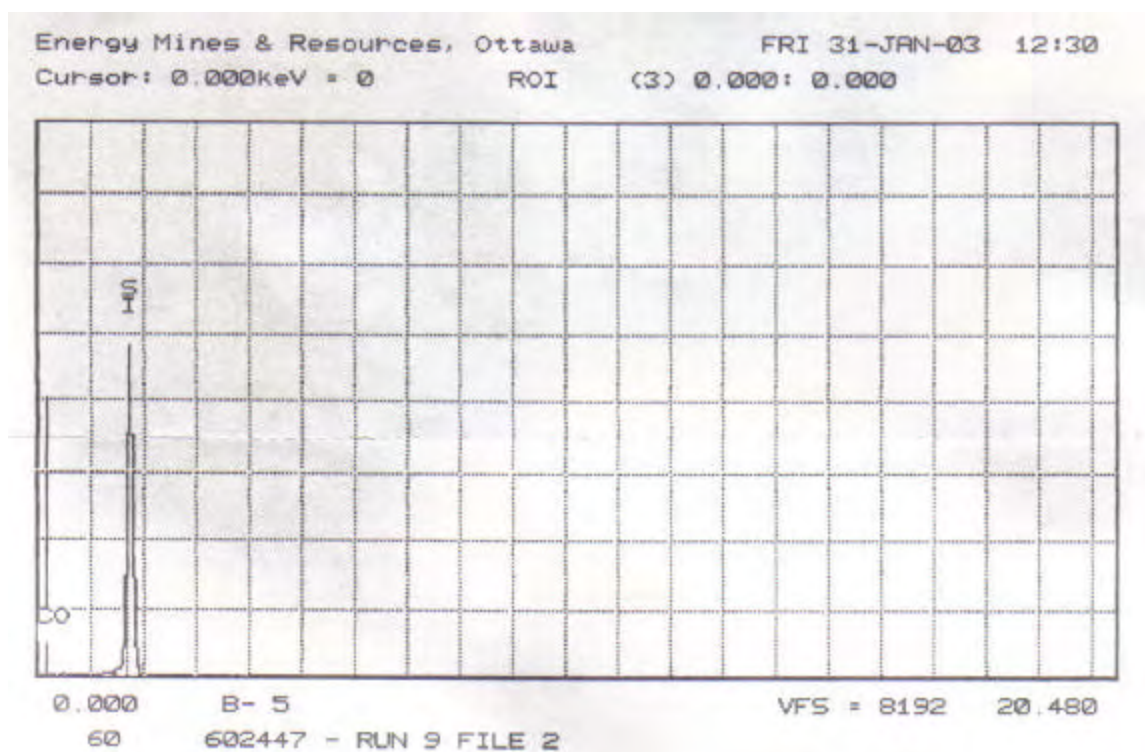


Figure 1-13: EDX spectrum of the quartz pictured in Images 1-15 &1-16

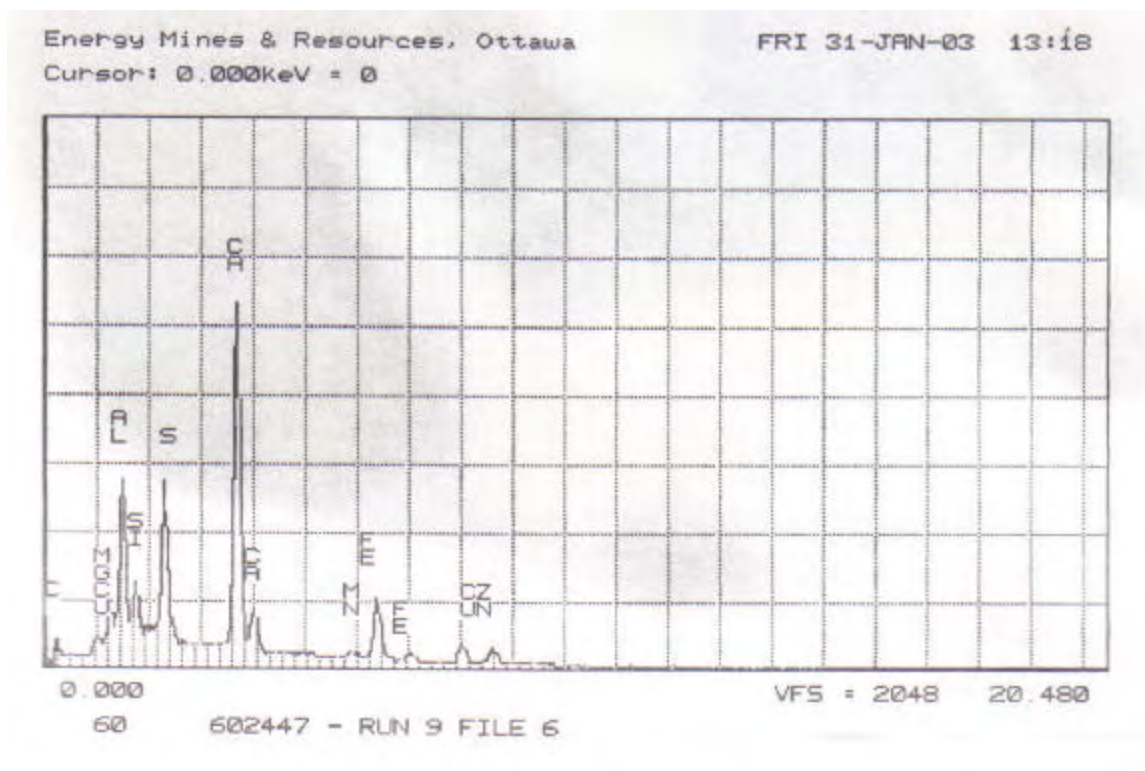


Figure 1-14: EDX spot analysis Image 1-15

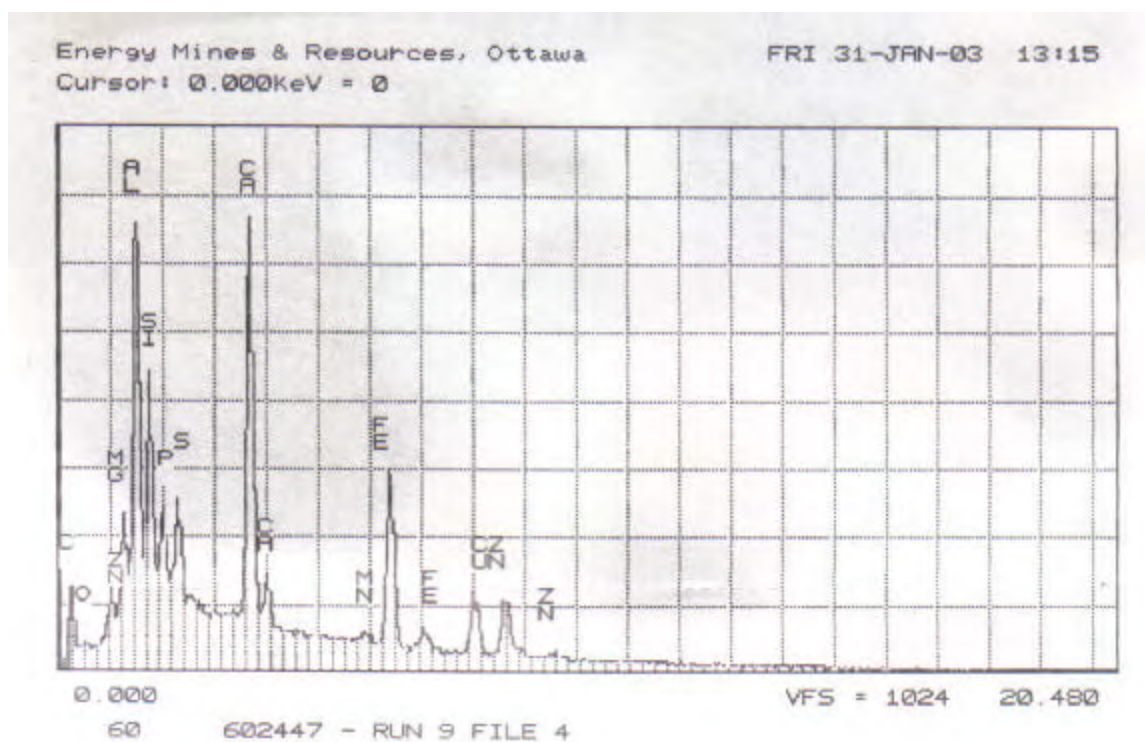


Figure 1-15: EDX spot analysis Image 1-16

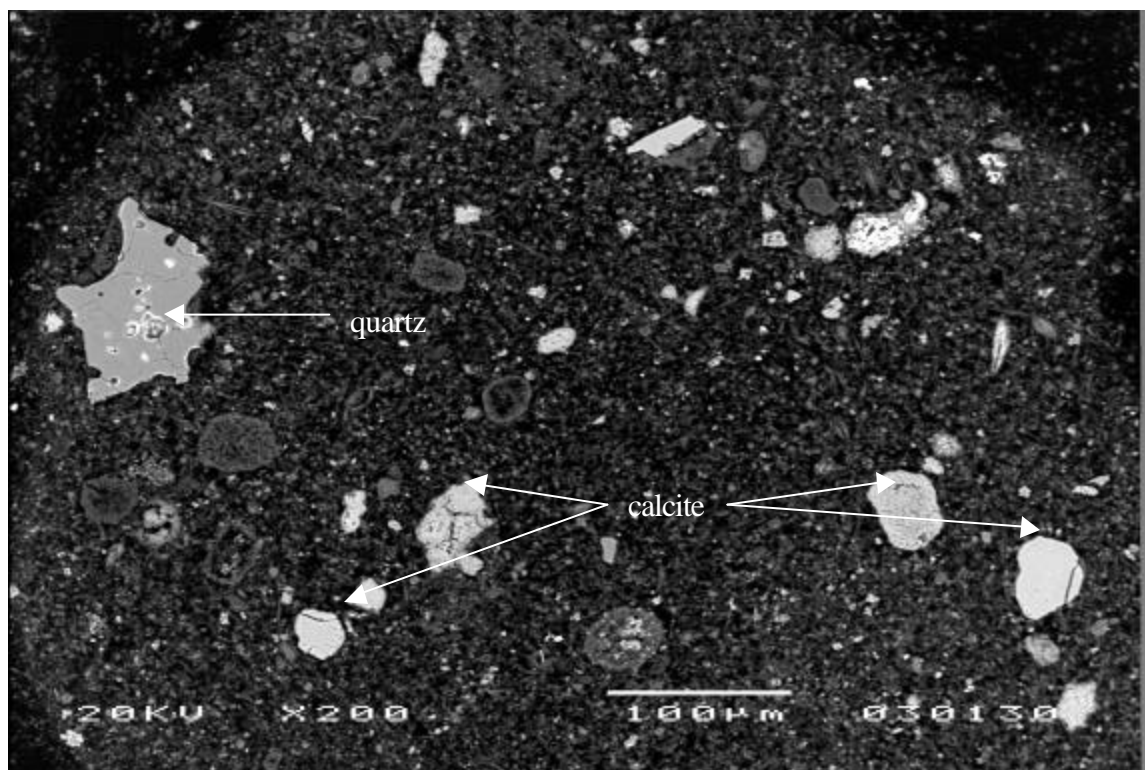


Image 1-17: Run 13 loose agglomeration of smaller mineral grains. Pictured are calcite and quartz grains

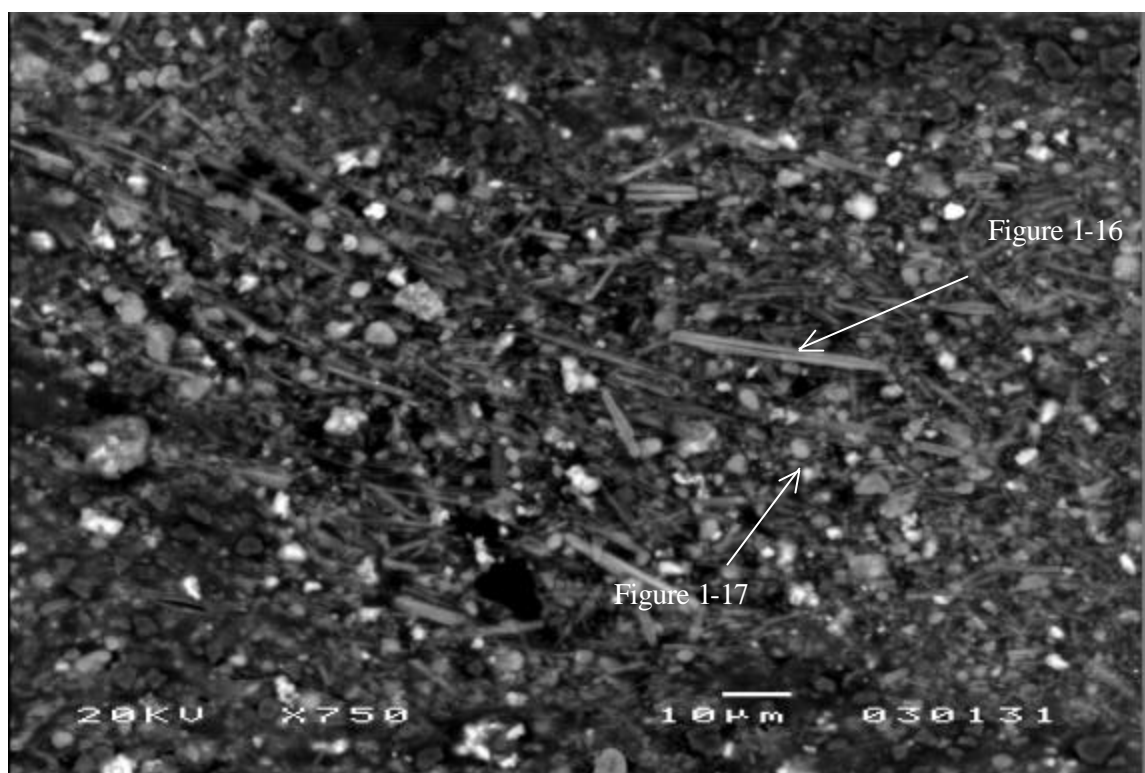


Image 1-18: Acicular grains and rounded grains. Also indicated are locations of Figure 1-16 & 1-17 EDX spot analysis.

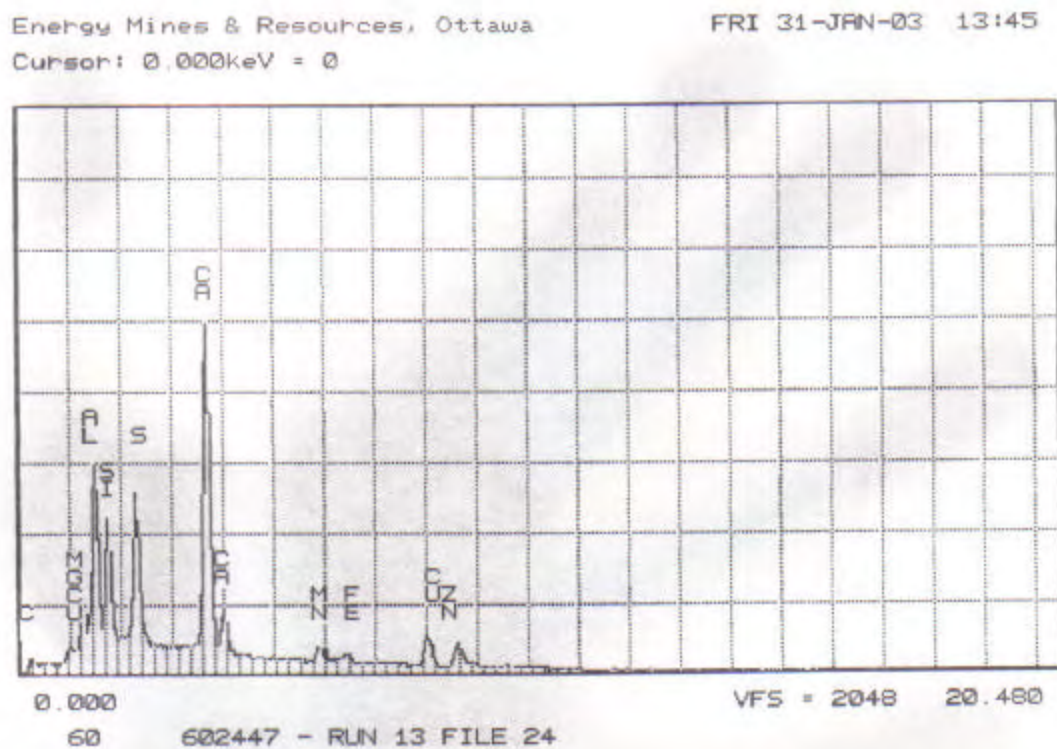


Figure 1-16: EDX spectrum of acicular grain shown in Image 1-18

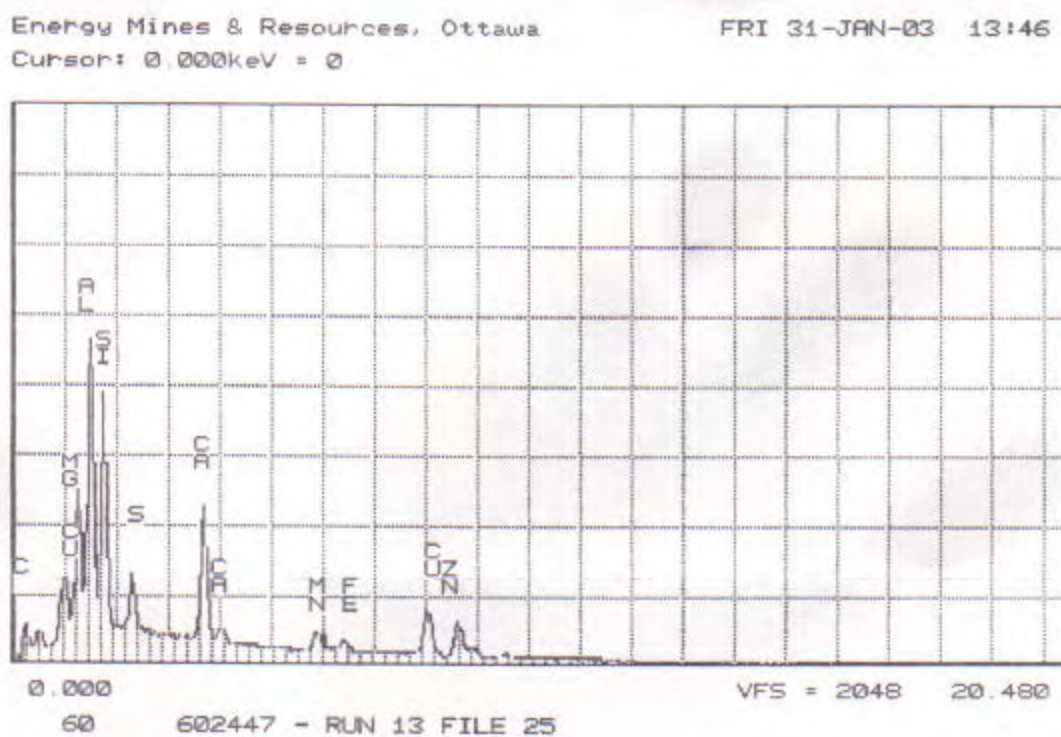
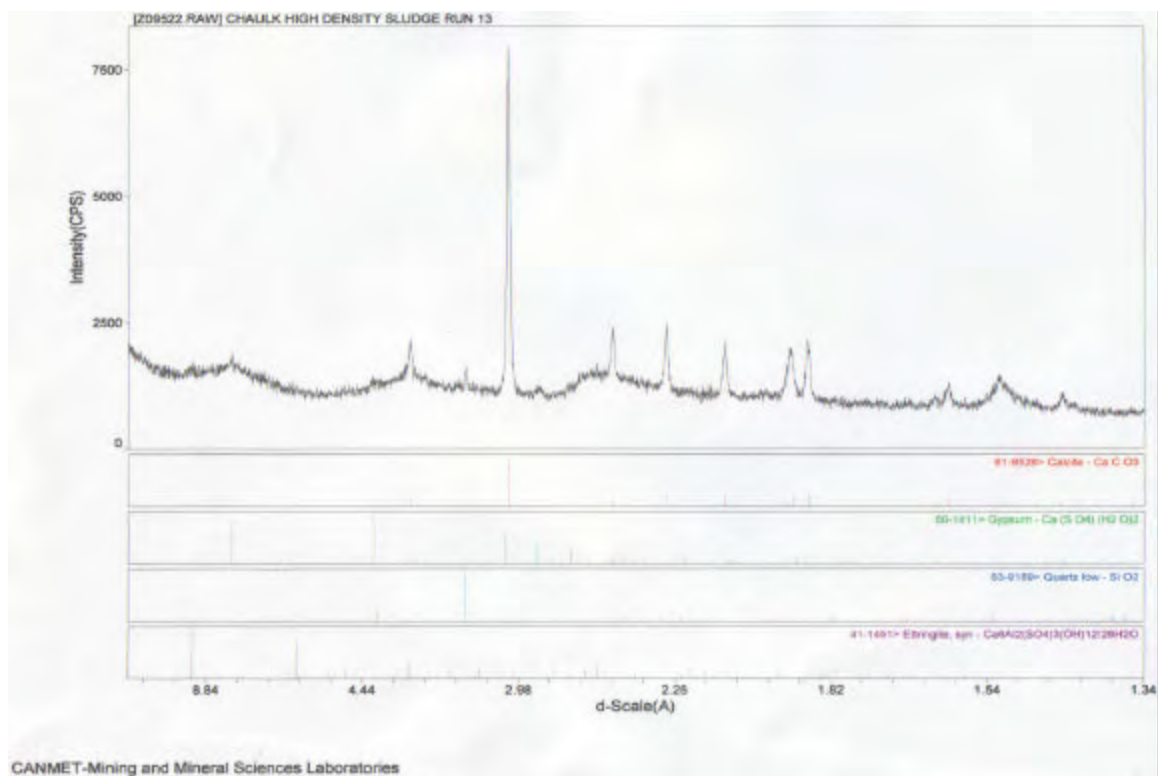


Figure 1-17: EDX spectrum of rounded grain shown in Image 1-18



XRD 1-3: Run 13 diffractogram