CHARACTERIZATION AND STABILITY OF ACID MINE DRAINAGE TREATMENT SLUDGES

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MINING AND MINERAL SCIENCES LABORATORIES
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EXECUTIVE SUMMARY

Acid mine drainage (AMD) and other acidic metalliferous effluents are commonly treated by the mining and metallurgical industries by lime neutralization. Upon neutralization, metals precipitate out of the effluent as hydroxides. This neutralization produces voluminous hydroxide sludges with low solids content (frequently < 5%). Despite recent improvements to the traditional neutralization method, it is estimated that as much as 6.7 million cubic metres of sludge are produced annually in Canada. In addition, the Canadian mineral industry is faced with questions related to the long term stability of AMD treatment sludges, and their environmentally acceptable disposal.

There is a need to develop standard sampling, handling and characterization protocols for AMD treatment sludges. A systematic method of assessing sludges with respect to their chemical, physical and leaching characteristics is necessary for estimating the sludge stability and for making informed decisions for disposal.

This report summarizes work that has been carried out in three areas:

☐ a site survey and sampling campaign of AMD treatment sludges at 11 Canadian mine sites;

☐ a detailed characterization of the collected sludges including physical, chemical, mineralogical and thermal analyses; and

☐ the leaching of the sludge samples using two distinct tests in association with a review of current hazardous waste regulations.

This report provides a data bank of lime treatment sludge characteristics which has been applied here in the discussion of sludge stability. Furthermore, this information may be used to assist operators, researchers and regulators in the development of improved treatment processes, effective disposal methods and appropriate regulatory tests for lime treatment sludges. These data may also be used to compare treatment operations and to forecast sludge related issues arising at treatment plants.

Sludge sampling

Sludge samples were collected from 11 Canadian mine sites (seven base metal, two uranium, one gold and one coal) from December 1995 to March 1996. Background information on AMD production, sludge production and disposal as well as the overall treatment process was compiled for all sites. Wherever possible, both fresh (i.e., end of pipe) and aged (i.e., pond core samples at depth) sludge samples were collected to study the effects of natural sludge aging.

Sampling plans were developed prior to the collection of sludge cores to ensure a representative composite sample of aged sludge. The number of samples was based in large part on the volume of disposed sludge, while the sampling stations were defined after a review of site-specific characteristics including pond dimensions and patterns of disposal within the pond. A commercially available hand corer with extension was used to reach depths of up to 6 m and is recommended for collection of cores in shallow water and/or sampling through an ice cover in winter.

Sludge characterization

Physical characterization and leaching tests were performed on the wet samples. The remaining analyses (chemical, mineralogical and thermal) were done using the freeze-dried material.

The pH values for the sampled sludges were alkaline and ranged from 8.2 to 10.8. In most cases aged sludges showed a lower pH than their fresh counterparts. E_h values ranged from 58 to 315 mV with the aged sludges commonly recording the lower values.

Denser sludges, generally produced using the High Density Sludge (HDS) process, displayed both smaller median particle sizes and narrower particle size distributions. Many of the sludges produced from conventional or basic treatment processes exhibited bimodal particle size distributions. In all but one case, the measured particle size was greater for the aged sludge.

The solids content of the sludges ranged from 2.4% to 32.8%. In almost all cases at least a 25% increase in solids content was seen from the fresh to the aged material. Based on the samples tested, no correlation was observed between the degree of densification and either the age of the deposited sludge or the presence (or absence) of a water cover on the sludge pond.

Neutralization potential values for the sludges collected ranged from 108 to 725 tonnes $CaCO_3$ equivalent per 1000 tonnes sludge. While low NP values are attractive in terms of plant efficiency, sludges with high NPs have more neutralization capacity which directly impacts on long term sludge stability. Calcium content in the sludges varied from 3.8% to 27%; calcium is present in two main forms, as calcite or gypsum.

The metals content of sludge can be viewed as potential recoverable assets or a source of leachable metals. Zinc recovery may be possible for some sludges ([Zn]>14%). Zinc concentrations ranged from 0.019% to over 14.4%. The low concentrations observed for copper and nickel (generally less than 1%) do not justify their recovery. Aluminum ranged from 0.1% to 11%. Copper, arsenic, boron, cadmium, chromium, mercury, lead, and selenium occur only in trace amounts, generally less than 0.01%. Iron ranged from 1.5% to 28% in the sludges.

All the sludges contained sulphate, in some cases greater than 30%. The sulphate content correlated directly with the amount of total sulphur in most of the sludge samples, indicating that all the sulphur present in these samples occurs as sulphate.

Mineralogical analyses of all sludge samples showed a major amorphous phase. Readily leached metal species such as zinc were commonly associated with this phase, which appeared to be effective in scavenging metal species (Al, Cu, Fe, Mg, Na, Ni, Zn) during precipitation. Calcium is present as calcite, gypsum and bassanite; they occur both as individual grains and in the amorphous phase. The amount of calcite may indicate the degree of recrystallization and the increased stability of the sludges. Quartz, silicates, sulphides and iron oxide particles found in the sludges are detrital in origin.

Sludge leachability

AMD treatment sludge samples were leached using two protocols. The Ontario Leachate Extraction Procedure (LEP) uses an acetic acid solution as a leachant while the Modified LEP substitutes a synthetic acid rain for the acetic acid. Acetic acid mimics the organic acids expected to be present in a municipal landfill and assumes co-disposal of mineral processing and municipal wastes. On the other hand, the mixture of sulphuric and nitric acids better simulates the inorganic acids that are likely to come in contact, through acidic precipitation, with sludges

disposed in ponds. Generally, less metal was leached from the sludges when they were subjected to the Modified LEP as opposed to the Ontario LEP. Sludge leachability is strongly dependent upon the final leachant pH which is influenced by the choice of leachant and by the neutralizing potential of the sludge. Metal leachability increases with decreasing pH at pH less than about 9.5. The amount of metal leached is also related to the metal concentration in the sludge itself. In general, the aged sludge samples showed an increase in stability relative to the fresh samples as indicated by the leaching results and supported by the mineralogical data and particle size analyses.

AMD treatment sludges are waste products which may be subject to waste management regulations. A leachate extraction test may be used to evaluate if the waste is capable of yielding a leachate which exceeds regulated concentration limits for selected contaminants. When a waste fails the test in relation to the limits specified in a particular jurisdiction, the waste may be classified as hazardous. All but two of the sludge samples subjected to the Ontario LEP passed the test when the leachate concentrations for metals are compared to the regulated limits governing the classification of hazardous waste material in Canadian jurisdictions. A fresh sludge from a base metal operation failed on zinc and an aged sludge from a uranium mine failed on uranium. There are only three jurisdictions in Canada which have a regulated limit for zinc and this sludge would actually fail only in comparison to Québec's current regulation. It must be noted however, that Québec's proposed new regulated limits do not include zinc. None of the sludge samples failed when tested with the Modified LEP. The leachate concentrations from both tests were generally at least five times lower than the most stringent of the regulated limits.

Therefore, fresh AMD treatment sludges would not generally be classified as hazardous wastes based on current leaching protocols and regulated contaminant limits. Aged sludges are even less likely to be classified as hazardous wastes. Based on the samples tested, this work has underlined that while sludge stability is an issue, greater emphasis should be placed on sludge disposal and volume reduction.

Numerous leach protocols have been developed to test solid wastes. None of these leaching tests have been specifically designed for evaluating AMD treatment sludge leachability. A thorough review of regulatory and research leach protocols from Canada and the United States

is provided. While regulatory leach tests for classification of hazardous wastes in Canada involve the use of acetic acid in the protocol, more appropriate tests, such as the Modified LEP, need to be considered for assessing the leachability of AMD treatment sludges for on-site disposal in a pond environment. Ultimately, the context within which sludge leachability (stability) is measured must be kept in mind when a leach test is applied.

In most provinces and territories, the testing of AMD treatment sludge and its storage/disposal is controlled by site-specific licences or permits based on appropriate legislation. A review of Canadian and American hazardous waste regulations, and other pertinent regulations and guidelines as they apply to the leachability testing of AMD treatment sludges is provided.

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1.0 INTRODUCTION

Lime neutralization of acidic metalliferous effluents is perhaps the oldest water pollution control technique practiced by the metallurgical and related industries. Upon neutralization, metals precipitate out of the effluent as hydroxides. This neutralization commonly produces voluminous hydroxide sludges with low solids content (frequently < 5%) which have the potential of dissolving and releasing metal ions into the environment over the long term. Despite recent improvements to the traditional neutralization method (Demopoulos et al. 1995; Dinardo et al. 1991; Flynn 1990; Kuit 1980; Kuyucak et al. 1991; Vachon et al. 1987), it is estimated that as much as 6.7 million cubic metres of lime treatment sludge is produced annually in Canada (section 2.4.1).

In addition, the Canadian mineral industry is faced with questions related to the long term stability of acid mine drainage (AMD) treatment sludges, and their environmentally acceptable disposal. To adequately assess disposal options, further information is required on the characteristics and stability of AMD treatment sludges. This study thoroughly characterizes sludge samples from a wide range of lime treatment operations. This data will serve as the baseline from which future disposal methods can be improved.

Reliable analytical data are very difficult to obtain from sludges. Sampling, preservation and analytical procedures have not been defined or understood for sludges. Consequently, what occurs in the field may be misrepresented through improper sampling and preservation techniques or misapplied analytical techniques. High variability is inherent in sludges as their composition is dependent on AMD composition and treatment process used, which in turn varies with source and season (Wilson 1994b). This variation has a significant impact on the reliability of the data generated and on the conclusions drawn. A standardized sampling protocol for sludges is required to ensure representative samples are collected and preserved for proper characterization. Without proper sampling and preservation techniques, characterization information is invalid.

There is increased concern about the long term stability of AMD treatment sludges. Information is not widely available with respect to sludge stability and the characteristics of the sludges generated. A 1987 study (GML 1987) examined representative treatment sludges from the Canadian mining industry. In this study the sludge samples were subjected to six different leaching procedures. The leach solution concentrations of Zn, Ni, Cd, Cu and Pd exceeded Québec regulatory levels for classification of a waste as hazardous in 50% of the sludge samples examined.

Provinces such as Ontario and British Columbia also have regulations governing hazardous waste which may apply to the disposal of treatment sludges. Other provinces such as New Brunswick refer to the provisions of the federal Transportation of Dangerous Goods Regulations for waste management control (Wilson 1994a). The leach tests associated with the above regulations may over predict contaminant mobility for AMD treatment sludges. Potential changes to waste management regulations in federal and provincial jurisdictions do not, however, address the issue of applicability of the existing regulatory leach tests to wastes which will not be disposed in municipal landfills. Appropriate leachability measurements can provide information on the stability of treatment sludges. Sludge stability is a primary concern of both industry and regulators in choosing an environmentally acceptable method of disposal.

The objectives of this project were twofold: to characterize the treatment sludges in terms of chemistry, mineralogy and environmental stability; and to investigate factors affecting sludge stability. This report covers the characterization phase of the project and summarizes work that has been carried out in three areas:

☐ A site survey and sampling campaign of AMD treatment sludges has been completed at 11 Canadian mining sites. The sampling campaigns are described and a general sampling protocol is recommended.

☐ A detailed characterization of the collected sludges has been completed, including physical, chemical, mineralogical and thermal analyses. Results for fresh sludges and previously deposited pond sludges are in turn presented to compare variation in sludge characteristics with time.

☐ The sludge samples were subjected to two distinct leach tests and the results documented. Within this context, current hazardous waste regulations and leach test protocols, as they apply to the stability and leachability of AMD treatment sludges, are reviewed.

2.0 PLANT SURVEY

The 11 sites taking part in this study were selected by MEND from an initial list of 15 sites derived from several information sources (GML 1987; Ritcey 1989; MEND 1995). A coding system was used to maintain confidentiality as to the characterization results and the plant information. The sites were selected with the goal of obtaining good representation of Canadian mines. To obtain the background information necessary to accurately characterize the lime sludge samples collected, a site questionnaire was completed by site personnel in collaboration with CANMET staff (Appendix A). This plant survey collected general site information, specific information on the AMD produced, sludge production and disposal data, and general information relating to the overall treatment process at the site.

2.1 Site Distribution

Sludges at a total of 11 mine sites were sampled. There were seven base metal sites, two uranium sites, one gold site, and one coal site. The sites are located in four provinces: New Brunswick, Québec, Ontario and Manitoba. Table 1 summarizes the general information for each of the 11 sites. Specific details of each site are described in Appendix B.

2.2 Acidic Drainage

The majority of the sites collected acidic drainage from tailings runoff. Other sources of AMD included waste rock and runoff from roadways. Several sites treated process water and smelter water with their acidic drainage. Table 2 presents the AMD data collected from the plant survey. Most sites report iron as the predominant metal species in the stream. Iron concentrations reported ranged from as little as 0.5 mg/L to as high as 774 mg/L. Copper and zinc were also present in significant concentrations at most sites. Sulphate concentrations when reported ranged from 950 mg/L to 4516 mg/L. The maximum sulphate concentrations were reported for drainage from waste rock piles associated with the gold mine. The pH values reported ranged from 2.5 to 10.8. The majority of the pH values were between 2.5 and 4.5. A

Canadian survey of acidic drainage characteristics can be found in MEND Report 3.22.1 (Wilson 1994b).

Table 1: General Site Information[1]

Site Code	Location	Operation	Local Geology	Facilities Present	Production Status
D-1	Ontario	Base Metal Ni, Cu		Smelter	Mining ceased 1991 Milling ceased 1988
Q-2	Manitoba	Base Metal Cu, Zn		Concentrator, zinc refinery, copper smelter	Producing
M-3	Ontario	Base Metal Cu, Ni, Co, Pt		Smelter, mill 2@130' dia. reactor clarifiers	Producing
R-4	Québec	Base Metal Cu		Lime neutralization plant	Closed 1962
F-5	NB	Coal	Plateau, lithology is green/grey; fine to coarse	Neutralization facility, sludge storage ponds	Closed 1986
S-6	Ontario	Base Metal Cu, Zn	Flat, underlined by clays	Smelter, refinery, purification leach plant, cell house	Producing
J-7	Québec	Gold	On the Cadillac fault	Mineral treatment	Producing
W-8	NB	Base Metal	Massive sulphide ore body	Underground mine/mill complex	Producing
N-9	Ontario	Uranium	Cdn. Shield, natural bedrock basins	Flooded tailings basin, 2 settling ponds	Closed 1990
B-10	Ontario	Base Metal Cu, Ni		Mill, smelter	Producing
T-11	Ontario	Uranium	Cdn. Shield, exposed bedrock	Elevated tailings, effluent treatment plant, sludge settling	Closed 1968

[1] Data compiled, as given, from site surveys (Appendix B)

2.3 Treatment Processes

Three basic lime treatment processes have been described in the literature (Kuit 1980; Vachon et al. 1987; GML 1987; MacDonald et al. 1989) and can be classified as follows:

Table 2: Acid Mine Drainage Characterization[1]

Site Code	Source	Chemical Composition (mg/L)															
		Al	As	Cd	Cr	Cu	Fe_t	Hg	Mg	Mn	Ni	Pb	Ra_{226}	U	Zn	SO_4^{-2}	pH
D-1	Tailings					5	<10				<10						2.5
Q-2	Tailings, process waters, residue pond decant			< 0.01		0.1-0.05	0.05-0.5			0.01-0.7	<0.01	<0.04			0.5-3		7-10.8
M-3	Tailings area and smelter	2.55	<0.0131	0.0021	0.0112	0.721	36.3			0.938	11.4	<0.03			0.16	1524	5.93
R-4	Tailings			0.016		1.0	75			4.0		<0.05			5.3	950	2.88
F-5	Tailings, sandstone overburden	126					40									1850	2.8-3.5
S-6	Tailings		0.09-<0.05	200-800		2-0.01	1-20	0.001			0.01-0.05	0.02			0.5-120	1200-2000	3.5-7.5
J-7	Waste rock						774									4516	
W-8	Tailings, waste rock, roadway, process waters					5	150					3			100	3500	4.5
N-9[2]	Beached uranium tailings					1.6	6.0			1.6	0.07	0.08	16 Bq/L	0.3	0.5	1580	4.5
B-10[3]	Tailings area and smelter																
T-11	Tailings, elevated uranium					0.05	487			2.8	0.07	<.1		0.05-2	0.57		3.8

[1] Data compiled, as given, from site surveys (Appendix B)

[2] 1987-1989 data

[3] No data available

Basic: Slaked lime is added directly to the effluent stream. Solid/liquid separation occurs in a sludge pond. (Percent solids 1-5%.)

Conventional: Mechanically or air agitated reactors; lime addition is controlled by pH. Process provides good effluent quality. Reactor discharge is sent to sludge settling ponds or tailings ponds for solid/liquid separation. (Percent solids 3-10%.)

HDS Process: The High Density Sludge Process recycles a portion of clarifier underflow sludge, which is blended with lime slurry required for neutralization prior to the main reactor vessel. Efficient solid/liquid separation is achieved using mechanical clarifiers. Sludge is disposed of either in tailings or sludge ponds. (Percent solids 15-30%.)

Of the 11 sludge sites sampled, almost half use a basic treatment system to treat their effluent (Table 3). Three sites have invested in the more sophisticated HDS sludge process, while the remaining three use simple reactor (conventional) treatment with or without the use of flocculants. Only one of the basic treatment operations uses flocculants and this occurs only during periods of seasonal turbidity in the sludge pond.

The majority of the conventional and HDS treatment plants use a flocculant to aid in settling. At the site where no flocculant is added, the percent solids of the sludge was only 3.3 %. Three different types of Percol flocculants were used in the sites surveyed, Percol 338, Percol 727 and Percol 90L. The most commonly used was Percol 338. Percol 338 and Percol 727 are granular powders while Percol 90L is the liquid dispersion equivalent of Percol E-10 another common flocculant used in lime precipitation. All three flocculants are very high molecular weight anionic polyacrylamide compounds.

Calcium hydroxide is used at all 11 sites to neutralize acidity and precipitate heavy metals. Generally the lime (CaO) is slaked either on site at the treatment plant or at the treatment location to produce a slurry of $Ca(OH)_2$. The uranium sites also add $BaCl_2$ to remove radium. Several sites sparge air into their reactors to facilitate the oxidation of ferrous iron to ferric iron. The addition of air also assists in mixing and discourages ice formation during the winter months.

The average flowrates treated at these facilities ranged from 4 m^3/min to 76 m^3/min. Higher flowrates are commonly associated with composite effluent streams (tailings, process

Table 3: Treatment Process[1]

Site Code	Effluents Treated	Reagents	Flocculant	Process	Flowrate (m^3/min)	Difficulties	Comments
D-1	AMD/smelter process water	$Ca(OH)_2$	None	Lime / air	27.3 (max)	Inefficient treatment process (54-85%)	Precipitation cells; 4.5 kg $Ca(OH)_2$/min
Q-2	Tailings/process water	Lime	On occasion	Settling with finishing pond		Seasonal influences; process upsets	
M-3	AMD, watershed, process waters	Slaked lime	Percol 338	Lime/air	76	Very large watershed	
R-4	AMD	Hydrated lime	Percol 90L	HDS/lime/ flocculant/air	6		
F-5	AMD	Slaked lime	None	Lime precipitation	11		By placing sludge on the waste rock, the acidity dropped over a # of yrs.
S-6	Tailings/process water	Slaked lime	None	Lime precipitation	6.94	Seasonal variability	Zn values change with mill values
J-7	Tailings/ process water	Slaked lime	Percol 338	HDS/lime/air	19		
W-8	AMD	Slaked lime	Percol 727	HDS/lime/air	60	Difficulties with Zn removal	
N-9	AMD and mine effluents	Slaked lime	None	Lime precipitation	7.8	Short circuiting in settling ponds	
B-10	AMD, watershed	Slaked lime	Percol 338	Lime precipitation	8	Slag dump water is higher in iron	Shut plant off in summer
T-11	AMD	Lime slurry	None	Lime precipitation	4	Short circuit from inlet to outlet	

[1] Information compiled, as given, from site surveys (Appendix B)

waters, runoff, etc.). Some sites indicated that the flowrates were expected to increase in the spring and fall and decline in the summer and winter. Information on the seasonal dependencies of flowrate can be found in the MEND survey on acid mine drainage (Wilson 1994b).

Several difficulties were identified in the treatment processes. A common issue was one of short circuiting in settling ponds. Some sites had made modifications to their ponds such as construction of berms to vary the flow pattern in the settling pond. Other difficulties encountered included seasonal variations and process upsets as well as process inefficiency (Table 3).

2.4 Sludge Management

2.4.1 Sludge production

Sludge production and disposal data collected are presented in Table 4. It was difficult to obtain accurate values for sludge production; estimated production values were obtained. At most sites, annual sludge production ranges from 10,000 to 70,000 m^3 per year and at one site sludge production was as much as 4,000,000 m^3 per year. The volume of sludge produced is directly dependent on the treatment process. High density sludge plants produce considerably less sludge (by volume) for the same flow treated.

An estimate of sludge production in Canada can be derived from information presented in Table 4 and from information provided by Senes (1994) and Feasby (1996). It is assumed that 50 sites in Canada produce sludge (7% solids; 1 g/cm^3 bulk density) from AMD treatment, with the average sludge production per site per year of 25,000 m^3. Assuming a disposal cost of \$4 per m^3 (Senes 1994), the following values were estimated:

Total sludge production annually in Canada

5,686,600 m^3 (8 sites, Table 4) + 25,000 m^3 * 42 sites

≈ 6,736,600 m^3 per year

Area occupied (assume 2 m depth) ≈ 3,350,000 m^2 ≈ 335 hectares

Disposal costs per year ≈ \$ 26,950,000

Dry weight ≈ 470,000 dry tonnes per year

Table 4: Sludge Management Practices[1]

Site Code	Sludge Production (m^3)		Disposal Scenario	Pond History	Difficulties	Needs and Options
	Annual	Total				
D-1	4,100	70,000	Settling pond	17 years sludge accumulation	Very little densification with aging	Sludge added to polishing pond to reduce lime required and improve sludge density
Q-2	40,000	800,000	Free settling	20 years sludge accumulation, pH final discharge at 10		
M-3	2,000,000	10-15 M	Co-disposal with tailings			Considerable revegetation initiative on tailings
R-4	2,500				Principal drainage ditch	Low cost maintenance
F-5	30,000	300,000	10 active sludge ponds, 3-6 ponds dredged every year	Last dredged in 1995	Cost of dredging; can't keep sludge on surface of waste rock	Automation is required for walk-away reclamation
S-6	3,500,000 - 4,000,000		Sludge impounded in tailings volume reduced by freeze/ thaw	25 years sludge accumulation; divided into 2 ponds in 1992	Low sludge density and poor effluent quality	Treat sludge at earlier stage to increase density
J-7	70,000	350,000	Sludge pond, moving to co-disposal in 1997	5 years sludge accumulation		Study stability of aged sludge
W-8	40,000		Co-deposit with mill tailings		Periodic turbidity	
N-9			Sludge to ponds	Pond #1 full, overflow to pond #2	Short circuiting in settling pond	Minimize the rate of acid generation by flooding
B-10			Sludge to ponds with sulphide thickener overflows	Sludge deposited in pond with thickener overflows; dredged 2-3 years, sludge is stockpiled		
T-11			Located in permanent location in bottom of lake	26 years sludge accumulation	Short circuit in winter from inlet to outlet	Sludge consolidation

[1] Information compiled from site survey data (Appendix B) and sampling plan information

2.4.2 Disposal environment

The most widely used sludge disposal methods are pond disposal and co-disposal with tailings. The majority of the sites surveyed dispose of their sludge in a sludge or settling pond either for short term dewatering or long term disposal (8 sites). Short term pond disposal is principally used to dewater the sludge before permanent disposal with tailings or on waste rock. Ponds used for short term disposal are commonly dredged every 2-3 years. Some sites use the ponds for permanent sludge disposal. At one site, the sludge has been deposited in this environment for over 25 years.

Co-disposal with mill tailings is practiced at three of the sites surveyed. The advantages of this method are reduction in solid waste storage area and containment of waste products to one principal location. This method is commonly used in situations where a thickener is present and solid/liquid separation is readily achievable. Disposal of the lime sludge in a natural lake environment is practiced at one of the surveyed sites.

At one site (F-5), the dewatered sludge is placed on top of the waste rock pile in an attempt to reduce acid generation. Site D-1 is examining the use of sludge to increase the alkalinity and sludge density in the polishing pond. Aged sludge from the sludge pond would be dredged every 2-3 years and added to the polishing pond. In-pit disposal, recycling back to the smelter and underground disposal are other disposal methods practiced by the industry (GML 1987; Senes 1994) but were not encountered in this survey.

Some sites have made modifications to traditional disposal methods to meet site requirements. One site (R-4) has constructed sand filtration beds under the sludge ponds to remove excess water, while another site (N-9) has installed a hypalon pond liner to reduce the risk of groundwater contamination.

Difficulties and concerns raised regarding sludge management included low sludge density, high turbidity levels in the discharge effluent, short circuiting in settling ponds, the cost of sludge dredging and sludge stability (Table 4). Many sites are examining alternatives to overcome these challenges.

3.0 SLUDGE SAMPLING METHODOLOGY

There is a need to develop standardized sampling, handling and characterization protocols for AMD treatment sludges due to their inherent variability. Since the composition of the AMD changes with source and season, the composition of the disposed sludge will vary throughout the sludge pond. This variation has a significant impact on the reliability of data generated and on conclusions drawn. Sludge samples must be collected following a sampling protocol that ensures that a representative sample is collected and properly preserved for characterization. Information on mine waste/sediment sampling from the literature was used to develop a 'working' sludge sampling protocol (Appendix C). Pilot site sludge sampling and characterization allowed for the refinement of the protocol to better meet project objectives. The sludge sampling protocol used in this study is outlined below. Sludge sampling and preservation techniques specific to this study are outlined in the experimental section.

3.1 Sludge Sampling Plans

Wherever possible, both fresh (i.e., end of pipe) and aged (i.e., pond core samples at depth) sludge samples were collected. Sampling plans were developed for each site in which cores were to be taken. The design of an accurate sampling plan is necessary in collecting appropriate samples from which to produce a representative composite sample of the disposed sludge. Before the sampling plan was designed, background information was first assembled. This information included:

- ✓ map/engineered drawing of sludge pond
- ✓ dimensions of pond
- ✓ sludge depth (depth profile if possible)
- ✓ location of inlet and discharge points
- ✓ variance in sludge composition within the pond (if available)

From this information the volume of sludge within the pond was determined. The number of cores required was based in large part on the volume of disposed sludge (Table 5), while the sampling stations were defined after a review of site specific characteristics including pond dimensions, pond morphology and flow patterns within the pond. One step in determining the number of sampling stations required should be an estimation of sludge variability. However, these sludge variability data were scarce and therefore this method was not applied in this study. Most of the site ponds sampled for this project were of average size, while a few ponds were large.

Table 5: Number of Samples to be Collected as a Function of Sludge Volume

Sludge Volume Disposed (m^3)	Number of Samples to be Collected
Very small (<10,000)	3-6[1]
Small (10,000 - 50,000)	7-12
Average (50,001 - 100,000)	13-18
Large (100,001 to 1,000,000)	19-40
Very Large (>1,000,000)	41 + (volume -1,000,000)/75,000[2]

[1] It is assumed that six samples are sufficient to obtain an acceptable level of confidence for a project of 10,000 m^3 (Ocean Chem Sciences Ltd. 1984).

[2] Atkinson (1985)

3.1.1 Selection and positioning sampling stations

The selection of sampling stations depends on the nature, dimensions and shape of the sludge pond as well as the flow patterns within the pond. Sampling stations were located on a transect down the long axis of the pond and along ray transects originating at the inlet point. When sludge depth profile information is available sampling stations are located across depth contours, with more samples taken in areas where the sludge depth is greater. An example of an actual sampling plan developed for a sludge pond is shown in Figure 1.

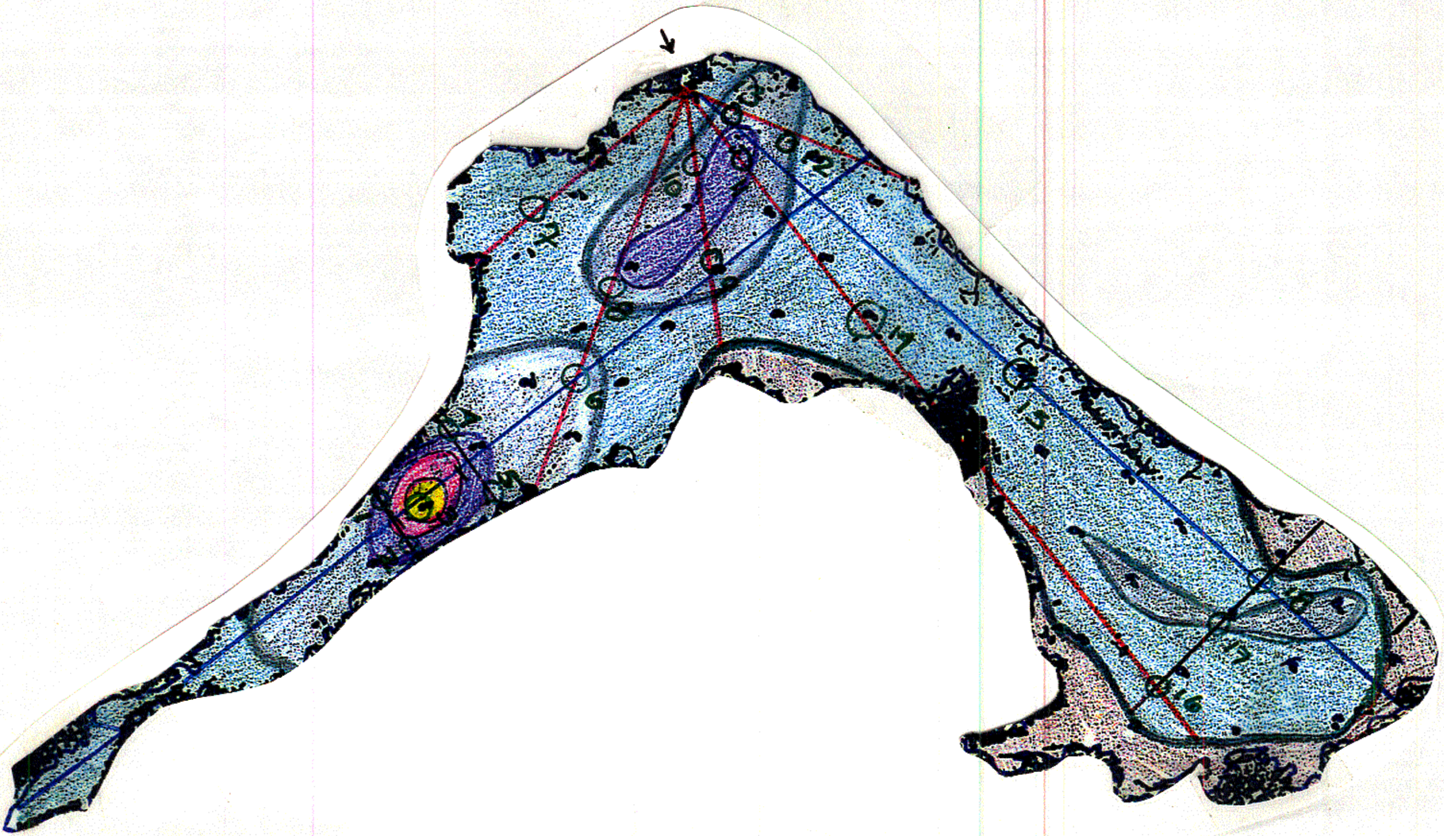


Figure 1: Example of a sampling plan for a sludge pond based on sludge depth, sludge volume, pond geometry and flow pattern. Colour contour lines mark the sludge depth profile on the pond. Numbers (①) indicate sampling stations and the arrow (←) marks the treatment plant discharge point.

Sample stations were located on the pond using the sampling plan (with marked sampling points), a compass and a 100 m tape measure. Applying a grid pattern over the sampling plan proved to be beneficial in locating the sampling stations during sampling.

3.1.2 Sampling equipment

Several different sampling devices are commercially available for grab and core sampling. Due to the fluid nature of the sludges, sampling equipment was chosen to eliminate the possibility of sludge loss during sampling. High-density polyethylene pails (20-L) were used to collect and store grab samples of fresh sludge. Commercially available corers can be used to collect core samples of sludge deposited in a pond environment. The hand corer shown in Figure 2 was most suitable for collecting cores during winter conditions after first opening a hole in the frozen pond with an ice auger (Figure 3). The hand corer is also useful for coring in shallow water environments. With an extension handle the corer could reach depths in excess of 5 m. The second type of corer for sludge sampling is the Ballchek corer (Figure 4). This type of corer is designed for deep water environments where a hand corer is not practical. The Ballchek corer has stabilizing fins which offer high strength and low drag characteristics. A practical feature of the Ballchek sampler is a simple positive sealing mechanism which acts as a check valve to retain the sludge in the core tube during raising operations.

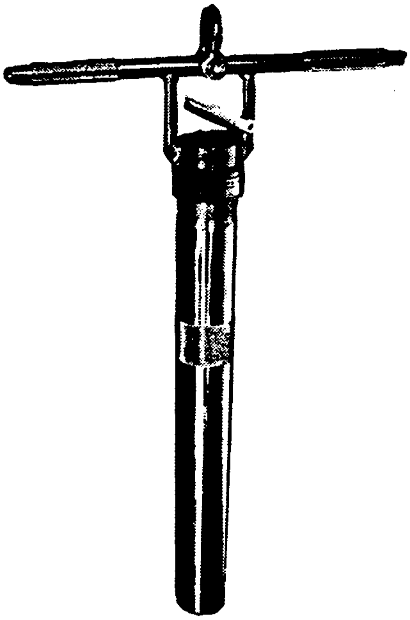


Figure 2: Hand corer.

Figure 3: Winter sludge sampling using a hand corer.

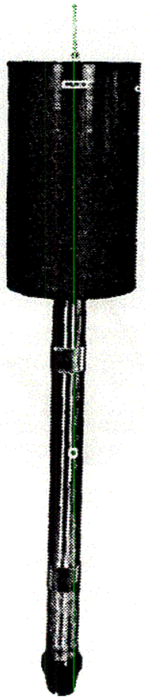


Figure 4: Ballchek corer.

Sterile acrylic coring tubes (50 cm x 5 cm (I.D.), hand corer; 75 cm x 5 cm (I.D.), Ballchek) were used to contain and store the sludge. Each tube was labelled with the following information:

- ✓ project number
- ✓ site, name and location
- ✓ sampling date
- ✓ station number
- ✓ sampling personnel
- ✓ sludge use

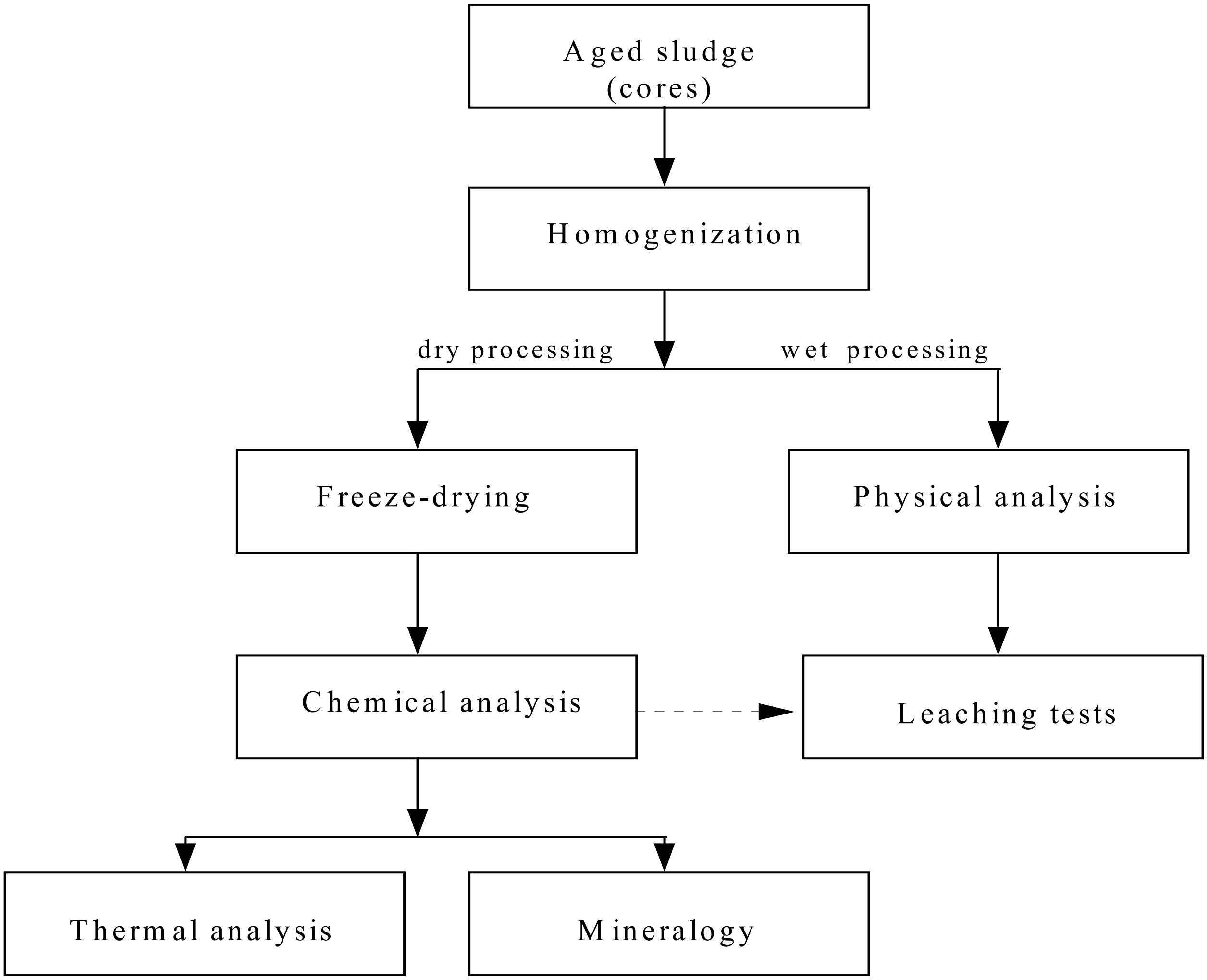
3.2 Handling, Preservation and Storage of Sludge Samples

Particle size distribution analyses, pH, E_h, percent solids and bulk density measurements were carried out on wet sludges. Samples for the wet analyses were stored at 4°C and never frozen. If ferrous iron is to be determined samples must be stored in airtight containers, under inert gas, and should be analyzed as soon as possible after collection. Drying, freezing and thawing of the sludges can cause irreversible aggregation of particles and should be avoided.

Three types of drying are commonly used to prepare solid samples prior to further analysis: air-drying, oven-drying, and freeze-drying. Freeze-drying was the drying technique selected for this study as it best maintained the integrity of the samples for further characterization. Further details on handling, storage and preservation techniques used in this study can be found in the experimental section.

4.0 EXPERIMENTAL PROCEDURES

The collected samples were characterized with respect to chemical, physical, mineralogical, leaching and thermal properties. Figure 5 summarizes the characterization process.



Fresh sludge processed in the same manner except without homogenization

Figure 5: Sludge characterization flowsheet.

The sludge samples were collected from 11 sites over a period from December 1995 to March 1996. Aged sludge samples were collected using coring equipment and following sampling plans developed specifically for each site. Whenever possible, samples of both fresh and aged sludge were collected from a site in an attempt to study the effects of natural sludge aging.

4.1 Sample Preparation

4.1.1 Preservation and storage

The sludge samples collected were stored in insulated coolers until their arrival at the laboratory. In the laboratory, the samples were stored at 4°C in a laboratory refrigerator until the analyses were undertaken. The homogenization, physical characterization and leaching analysis were performed on wet samples. The remaining analyses were conducted using homogenized samples which had been freeze-dried and vacuum sealed for long term preservation.

4.1.2 Homogenization

Sludge resulting from liming of AMD streams is a complex mixture of chemical species and thus a composite sample for each site was prepared. First the individual cores were inspected and a description/photograph of the core was taken (Appendix D). The cores were then extracted from the acrylic holding tubes with care to exclude any foreign material (sand, tailings, slag, etc.) from the composite sample. If necessary, the core sample was centrifuged at low speeds to separate the sludge from the heavier foreign material. A 10-mL sample of each core was collected for future reference. The cores were then combined in a 20-L pail and thoroughly mixed by hand, using a spatula, to ensure homogeneity. In the case of very viscous sludges, a low-speed mechanical mixer was used to homogenize the composite sample.

4.1.3 Freeze-drying

A Labconco Freeze Drier was used to dry the samples. Each sludge was first centrifuged to reduce the amount of water to be removed from the sludge by freeze drying. The sludge was then smeared on the inside surface of a 250-mL freeze-drying flask and freeze-dried for 24 hours (or longer if required). The freeze-dried sludge was split into several sections and vacuum sealed for long term preservation. Individual vacuum sealed packets were prepared for each of the characterization procedures.

4.2 Physical Characterization

Particle size determination was completed on the samples of fresh and aged sludges using a Microtrac X100 particle size analyzer and methanol as the measuring medium.

The percent solids measurements were determined through weight loss by oven drying at 110°C. For comparison, percent solids were determined by weight loss after oven drying at 60°C for 24 h (Zinck 1993) and using a Sartorius moisture balance.

The bulk density of the sludges was determined by weighing 5 cm^3 of sludge on an analytical balance. Larger size fractions can be used to increase accuracy.

4.3 Chemical Analyses

The sludge samples collected were analyzed chemically for the following components: Ag, Al, As, Au, B, Ba, Ca, Cd, Cr, Cu, Fe, Hg, Mg, Mn, Na, Ni, Pb, S, Se, Si, SO_4^{2-}, Zn (wt% content of each element). The pH, E_h, neutralization potential, water content (105-110°C), loss on ignition (1000°C) and CO_2 content were also measured. The analyses were carried out using ICP-MS, ICP-IRIS, ICP, AA, LECO Analyzer, ion chromatography, and gravimetric techniques. QA/QC protocols followed can be found in the ASG Quality Manual (1996-1997).

4.3.1 Neutralization potential

The neutralization potential was determined by treating a sample with excess standardized hydrochloric acid and heating to ensure complete reaction. A fizz test was employed to ensure that the consuming minerals present. The unconsumed acid was titrated with standardized base to pH 8.3 to allow calculation of calcium carbonate equivalent of the acid consumed (Lawrence and Marchant 1991). Net NP was calculated from sulphur, sulphate and NP determinations given in Table 8B.

4.4 Mineralogy

Mineralogical analysis of sludges is complicated by their high water content and amorphous nature. The sludges were examined using X-ray diffractometry (XRD) to supplement the information obtained using the scanning electron microscope (SEM) equipped with an energy dispersive X-ray analysis (EDX) system. Details of various mineralogical characterization techniques have been published (Chen and Dutrizac 1990).

4.5 Thermal Analysis

Thermal analysis provides information about the form and amount of moisture in the sludge sample as well as the different compounds present. The techniques used were thermogravimetry (TG), differential thermal analysis (DTA) and Fourier transform infrared (FTIR) spectroscopy. A simultaneous TG/DTA (SDT2960 system) was used with a Bomem MB100 FTIR for evolved gas analysis. A 15-mg sample and a 15-mg platinum foil reference were contained in alumina pans, and heated at 20ºC/min to 1200ºC in 50 mL/min dry air.

4.6 LEACHABILITY

Sludge samples were subjected to both the Ontario Leachate Extraction Procedure (LEP) (Government of Ontario 1994b) and a modified version of this leach test (Modified LEP). According to the Ontario LEP, if a sample contains distinct liquid and solid phases, it should be separated into its component phases using pressure filtration. This procedure removes only a portion of the liquid from the sludge samples, however, leaving the bulk of the liquid with the solids in a gel-like mass of questionable homogeneity. Therefore, samples taken from the well-slurried sludges were weighed and then extracted without phase separation. The percent solids content was used to determine the actual dry weight of the solids for extraction; and the volume of liquid present in the sample was subtracted from the volume of water added to provide a final liquid-to-solid ratio of 20:1. For many of the samples (especially those with low solids content) there was an insufficient quantity of sludge to provide the designated 50 grams of solids for the extraction. In those cases, the entire procedure was scaled to the calculated weight of solids to maintain the appropriate liquid-to-solid ratio.

The initial leachant used in the Ontario LEP is water. However, once the sample has been added, the pH of the leaching solution is monitored at set intervals during the course of the extraction and manually adjusted to pH 5.0 with acetic acid if the pH is greater than 5.2. Since there is a specified maximum acid addition, a pH of 5.0 may not be attained for highly alkaline samples. In the Modified LEP, a synthetic acid rain was used instead of acetic acid to adjust the pH of the leaching solution. The synthetic acid rain was a mixture of 60/40 weight percent sulphuric/nitric acid diluted to pH 4.5. This solution is similar to the leachant described in the Synthetic Precipitation Leaching Procedure (SPLP) (United States Environmental Protection Agency 1992). The leachates were analyzed for the following metals: Ag, As, B, Ba, Cd, Cr, Cu, Fe, Hg, Ni, Pb, Se and Zn.

5.0 SLUDGE CHARACTERIZATION

In order to obtain some information about the effects of aging on lime treatment sludge, samples of aged sludge were compared to fresh sludges from the same site and treatment process. Several assumptions are made when comparing the fresh material with the aged material. A true comparison can only be accomplished under controlled conditions where the starting material is fully characterized and allowed to age. This type of aging investigation in presently ongoing as part of the Phase II work of this project. Nevertheless, from the information presented in this report several trends were observed with respect the impact aging has on sludge composition and stability.

5.1 Physical Characterization

Table 6 summarizes the physical characterization, pH and E_h data collected for the sludge samples.

5.1.1 Colour

Some colour variation was observed with the sludges. The main colours noted were grey-brown, red-brown, dark brown and dark green (Table 6). This colour variation appears to depend on several factors including treatment process, metal composition and particle size. The colour of the sludges produced from the high density sludge process was more intense due to their higher metal content. The type and quantity of metals present also impact on the sludge colour. Sludges containing a high proportion of ferric iron are red-brown, whereas sludges with a high proportion of ferrous iron tend to be dark green. Other metals and calcium tend to give the sludge a greyer appearance. Colour is simply a descriptive tool and should not be used to replace any quantitative characterization technique.

5.1.2 Percent solids

The solids content in the sludges ranged from 2.4% to as high as 32.8% (Table 6). The type of treatment process strongly influenced the solids content in the sludge. The denser sludges

Table 6: Physical Characteristics, pH and E_h of Sludge Samples

Site Code	Sample	Physical Characteristics							pH	E_h (mV)
		Colour	Percent Solids (wt. %)	Particle size (μm, 50%)	Particle size (μm, ma)	Particle size (μm, mn)	Bulk density (g/cm^3)	Dry sludge density[2] (g/cm^3)		
D-1	fresh	grey-brown	3.7	5.74	5.35	3.21	1.05	2.48	9.45	161
	aged	grey-brown	7.2	7.96	24.01	3.96	1.09	2.39	9.51	315
Q-2	aged	grey-brown	2.14	6.78	3.95	0.930	1.05	3.10	11.41	239
	aged	green-brown	9.6	11.86	16.73	5.19	1.11	2.54	9.66	175
M-3	fresh	grey-brown	6.9	25.2	32.56	19.63	1.08	2.20	9.42	232
R-4	fresh	red-brown	18.0	3.96	5.36	2.83	1.15	1.87	8.90	112
	aged	red-brown	24.8	5.27	6.46	3.44	1.21	1.85	8.32	262
F-5	fresh	grey-brown	3.7	42.45	59.23	10.30	1.06	2.44	9.62	300
	aged	dark brown	12.5	13.62	21.2	30.10	1.12	1.89	8.20	296
S-6	fresh	grey-brown	3.4	6.67	4.14	7.63	1.08	3.24	10.85	239
	aged	brown	4.1	21.06	39.39	11.63	1.05	2.76	10.56	201
J-7	fresh	red-brown	26.3	4.18	17.88	2.85	1.19	1.83	9.22	121
	aged	red-brown	32.8	10.22	15.53	7.46	1.37	2.27	9.22	92
W-8[1]	fresh	dark brown	32.2	4.09	4.19	2.85	1.26	1.85	10.04	166
	aged	dark brown	10.4	21.25	20.16	7.46	1.10	2.03	10.66	60
N-9	aged	grey	7.6	23.76	40.42	9.59	1.09	2.16	9.36	212
B-10	fresh	grey-brown	3.9	16.11	23.54	11.93	1.06	3.04	10.54	252
T-11	fresh	dark green	2.4	20.72	53.26	8.55	1.05	3.34	9.34	58

[1] W-8: fresh sludge produced from HDS process, aged sludge produced from basic lime treatment. [2] Estimated

were produced from the high density sludge process involving both sludge recycling and mechanical thickening. The percent solids in the sludge produced from the conventional reactor process was not significantly higher than the simple basic treatment.

In general, the percent solids in the aged material was at least 25% higher than in the fresh sludge. Conventional and basic type treatment sludges demonstrated a greater degree of densification with aging than was shown by the high density sludges. However, the HDS material was initially much denser than sludge generated from the other processes and consequently would require less densification. Information on pond/sludge history is presented in Table 4 and Appendix B.

The degree of densification does not appear to be significantly affected by the aging duration nor the presence of a water cover. The effect of aging was most pronounced for the coal AMD lime sludge, i.e., a 250% increase in sludge density. It is unclear if the composition of the sludge and the AMD origin influence the sludge aging process. The Al:Fe ratio for coal sludge is much higher than observed for other sludge samples characterized. This suggests that Al compounds undergo densification with time more readily than do the Fe compounds. Based on these results, sand filtration beds do not appear to be more effective than traditional pond environments in densifying the sludge with age. In the case investigated (R-4), densification over time was found to increase by only 40%.

Percent solids determinations were also completed using the oven drying at 60°C moisture loss method. In most cases, the percent solids determinations were 5-10% (change, 1-5% absolute) lower than drying at 110°C. At 60°C, complete drying was not obtained. The sludges dried at 60°C were then heated to 110°C (in the oven) for a further 24 hours. The percent solids measurements decreased with the increased temperature. The percent solids measurement using the moisture balance compared closely to the oven drying method at 110°C.

5.1.3 Particle size

Detailed particle size analyses were completed in duplicate on all the sludge samples collected. Table 6 summarizes the particle size data in terms of 50% - median diameter, ma - mean diameter of the area distribution, and mn - mean particle diameter calculated from number frequency distribution. The full description of the particle size terminology is given in Appendix E. The complete analyses including particle size distribution histograms can be found in Appendix F.

This method of measuring the particle size assumes that the specific gravity of the components are equal. However, the specific gravity of the sludge components ranged from 2.32 for gypsum to over 3 for several of the hydrolysis products. Also, this method provides measurements for aggregated particles rather than true distinct particles. The absolute particle size for individually grown particles can only be ascertained through mineralogical analysis. However, useful information can be obtained on a comparative basis for the different samples.

In all cases except one, the particle size increased with aging. It is possible that through aging, particle dissolution and recrystallization occur, leading to particle growth and aggregation. Many of the sludges produced from conventional or basic treatment processes exhibit bimodal particle size distributions (Appendix F). The smaller size fraction is thought to represent the amorphous hydroxide mass produced during neutralization. Calcite formed from the carbonation of unreacted calcium hydroxide and detrital material likely accounts for the larger size fraction observed in the cases with bimodal distribution.

From the data, it appears that there is a weak inverse relationship between the median particle size of the sludge and the percent solids of the sludge. Denser sludges, primarily those produced from HDS processes, report a smaller median particle size on the order of 5-10μm . The skew to larger particle sizes with less sophisticated treatment methods may result from poor plant efficiency and the presence of calcite (from unreacted lime). However, when neutralization potential is plotted against particle size no relationship is observed.

The broadness of size distribution was also found to be weakly related to the type of treatment process. Generally, sludges produced using the HDS process displayed narrower particle size distributions than sludges produced from either conventional or basic types of treatment.

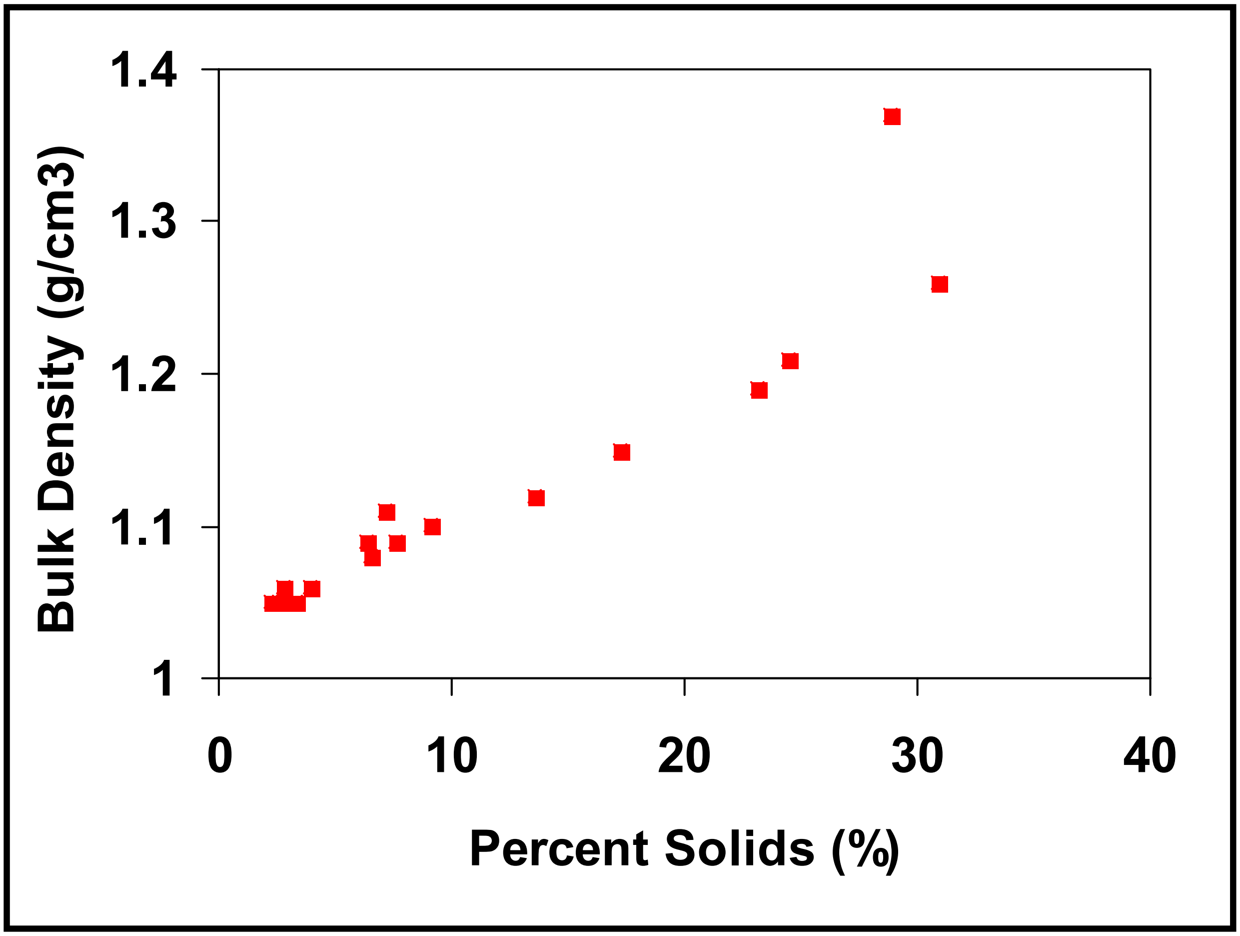


Figure 6: Relationship between bulk density and percent solids.

5.1.4 Bulk density

The bulk density is a determination of the weight of the sludge in a set volume fraction. Generally, the bulk density was found to correlate with the solids content in the sludge (Figure 6) and thus can be used as a rough and quick indicator of sludge density. However, the chemical composition of the sludges are different and as a result their bulk densities and more accurately, their dry precipitate densities will vary. These values were calculated from the percent solid and bulk density values and provide only an estimate of the dry precipitate (composite density). The dry precipitate (composite) density values range from 1.85 to 3.34 g/cm^3. This range is due to the variability in sludge composition between sites. This method gives estimated densities; more accurate measurements should be determined using a pycnometer. Table 7 lists the specific gravity values for several of the minerals found in the sludges.

Table 7: Specific Gravity of Sludge Minerals (HCP 1984; Hurlbut and Klein 1977)

Mineral	Specific Gravity (g/cm^3)
Gypsum	2.32
Ferric Oxyhydroxide	2.44-3.60
Ferrous Hydroxide	3.4
Zinc Hydroxide	3.05
Aluminum Hydroxide	2.42
Calcite	2.71
Albite	2.62
Muscovite	2.76-2.88
Chlorite - Clinoclore	2.6-3.3
Quartz	2.65
Ankerite	2.95-3.00
Pyrite	5.02
Talc	2.7-2.8
Barite	4.5

5.2 Chemical Analysis

The complete chemical analysis of the sludges collected from the 11 sites are presented in Tables 8A and 8B. Included in these tables are metal concentrations, carbon dioxide, sulphate, neutralization potential, loss on ignition and loss of moisture determinations. Measurements for sludge pH and E_h are presented in Table 6.

Table 8A: Chemical Composition

Site Code	Sample	Chemical Composition (%)												
		Al	Ag[1]	As	Ba	B	C	Cd	Ca	Cr	Cu	CO_2	Fe_t	Hg
D-1	fresh	0.1		<0.0016	0.012	0.0051	7.11	0.0002	26.6	<0.054	0.05	25.9	4.8	<0.0008
	aged	0.5	1.5	0.0023	0.005	<0.002	6.57	0.0009	22.9	<0.054	0.021	23.4	7.1	<0.0008
Q-2	fresh	1.0	10.40	0.011	0.004	0.009		0.059	6.9	<0.060	0.041	6.45	2.9	0.00011
	aged	1.8	6.6	0.0978	0.05	<0.002	3.29	0.0825	9.6	<0.054	0.25	11.6	12.3	<0.0008
M-3	fresh	1.5	19.5	0.0105	0.014	<0.002	1.97	0.0094	6.0	<0.054	0.27	6.45	14.7	<0.0008
R-4	fresh	2.8	1.0	<0.0016	0.020	<0.002	1.75	0.0029	8.3	<0.054	0.33	5.22	10.8	<0.0008
	aged	3.3	1.2	0.0025	0.005	0.0017	2.42	0.0025	7.4	<0.054	0.27	8.20	26.1	<0.0008
F-5	fresh	7.7	1.1	0.0018	0.005	0.29	1.34	0.0004	14.0	<0.052	0.0029	4.80	1.5	<0.0008
	aged	11.2	1.2	0.0020	0.005	0.16	0.98	0.0005	9.9	<0.052	0.010	3.41	6.2	<0.0008
S-6	fresh	1.3	7.3	0.0056	0.010	<0.002	4.57	0.0674	17.2	<0.052	0.15	16.1	2.3	<0.0008
	aged	0.6		0.0051	0.011	<0.002	2.40	0.1390	10.0	<0.054	0.20	8.66	3.0	<0.0008
J-7	fresh	4.3	4.7	<0.0016	0.005	0.0066	0.54	0.0002	13.0	<0.055	0.077	1.87	12.8	<0.0008
	aged	3.4	0.9	<0.0016	0.003	0.0049	0.30	0.0001	14.2	<0.055	0.054	0.95	13.4	<0.0008
W-8[2]	fresh	3.9	13.7	0.0248	0.014	0.0041	0.55	0.0137	3.8	<0.054	0.12	1.76	15.0	<0.0008
	aged	1.8	15.2	0.0487	0.022	<0.002	1.36	0.0057	11.6	<0.054	0.05	4.76	10.9	<0.0008
N-9	aged	4.9	1.1	0.0021	7.8	0.22	1.86	0.0014	5.0	<0.055	0.029	5.44	7.7	<0.0008
B-10	fresh	3.6	4.1	<0.0016	0.005	<0.002	2.52	0.0058	4.6	<0.054	1.48	7.73	22.2	<0.0008
T-11	fresh	0.6	1.8	<0.0016	0.011	<0.002	1.35	<0.0001	10.2	<0.055	0.001	4.80	28.1	<0.0008

[1] ppm [2] Fresh sludge was produced with HDS process; aged sludge was produced from basic lime treatment process.

Table 8B: Chemical Composition, Cont.

Site Code	Sample	Chemical Composition (%)															
		Mg	Mn	Na	Ni	Pb	Se	Si	S	U	Zn	SO_4	NNP	AP	NP	LOM	LOI
D-1	fresh	5.8	-	0.17	0.26	<0.43	<0.0009	1.1	1.43	-	0.067	3.70	NA	6	NA	3.69	38.3
	aged	6.3	-	0.07	1.06	<0.43	<0.0009	2.0	1.10	-	0.021	3.60	725	-	725	4.48	35.5
Q-2	fresh	18.1		0.24	0.005	<0.20		2.8	1.75	_	7.5	3.01	796	23	819	8.09	27.9
	aged	7.9	-	0.21	<0.11	<0.43	0.0296	5.2	3.18	-	5.9	3.22	359	66	425	6.19	21.8
M-3	fresh	7.3	-	0.21	4.19	<0.43	0.0148	4.5	3.00	-	0.98	5.95	293	32	325	14.5	23.0
R-4	fresh	5.5	-	0.04		<0.43	<0.0009	7.2	1.85	-	1.4	5.20	315	4	319	5.93	24.2
	aged	2.6	-	0.06	<0.11	<0.43	<0.0009	4.4	1.34	-	1.1	3.77	207	3	210	9.62	21.1
F-5	fresh	6.0	3.6	0.11	0.059	<0.43	<0.0009	1.7	7.46	-	0.14	18.80	335	38	372	6.85	23.7
	aged	1.2	2.1	0.07	0.059	<0.43	<0.0009	2.7	7.24	-	0.15	18.98	94	29	123	15.3	21.3
S-6	fresh	5.7	-	0.31	0.13	<0.42	0.0159	4.8	2.92	-	8.5	6.90	452	19	471	6.83	25.9
	aged	7.8	-	0.21	0.20	<0.43	0.0169	2.3	4.58	-	14.4	11.29	497	26	523	8.33	23.2
J-7	fresh	2.2	0.47	0.03	<0.03	<0.44	<0.0009	1.2	9.97	-	0.0031	26.71	84	34	118	14.1	16.1
	aged	1.7	0.24	0.03	<0.03	<0.44	<0.0009	0.8	11.3	-	0.019	30.71	74	34	108	12.8	14.1
W-8[1]	fresh	3.13	-	0.08	<0.11	<0.43	<0.0009	1.28	4.14	-	14.2	11.80	135	7	142	9.73	20.1
	aged	6.3	-	0.28	<0.11	<0.43	<0.0009	2.0	5.75	-	4.6	14.2	346	32	378	10.5	21.9
N-9	aged	3.8	1.1	0.15	<0.11	<0.45	0.0039	3.0	5.34	0.605	0.270	11.2	154	50	204	7.81	18.1
B-10	fresh	7.4	-	0.24	<0.11	<0.43	<0.0009	4.9	1.77	-	0.17	4.55	345	8	353	6.02	24.5
T-11	fresh	2.6	0.21	0.03	<0.11	<0.45	<0.0009	1.2	6.56	0.011	0.029	18.61	167	11	178	11.3	15.2

[1] Fresh sludge was produced with HDS process and aged sludge was produced from basic lime treatment process.

5.2.1 pH and E_h

As expected all the sludges studied are alkaline with pH values ranging from 8.2 to 11.41 Most of the sludges have pH values between 9 and 10. Two sludge samples have pH values below 8.5.

The F-5 aged sludge sample has a pH of 8.2, while the fresh sludge from the same site has a pH of 9.62. This may suggest, among other things, that the sludge alkalinity is being utilized in the disposal environment. In general, the pH of aged sludge samples was lower than that for the fresh sludge for the same site and process.

The oxidation-reduction potential was measured for all the sludges and provides an indication of the degree of oxidation during sludge precipitation. The E_h values ranged from 58 mV to 315 mV. The lowest value was reported for a sludge rich in ferrous hydroxide. No relation was found between aeration during lime treatment and higher potential values. In some HDS sludges, the oxidation potential was less than 100 mV suggesting incomplete oxidation. The aged sludges commonly recorded lower E_h values than the sludge collected directly from the treatment plant. This may be due to less available oxygen in the disposal environments examined.

5.2.2 Metal content

Concentrations of aluminum ranged from 0.1% to 11.2%. On average the concentration of aluminum was less than 5%. Higher values for aluminum were recorded for sludges produced from the treatment of coal AMD.

Iron is a major sludge component and was principally found in the ferric state. The iron content in the sludges ranged from 1.5% to 28.1%. Generally the iron content was roughly 10%.

Copper concentrations in the sludge samples were low. At all sites except B-10, the copper content in the collected sludge sample was less than 1%. Similarly, nickel concentrations were generally less than 1%. The low concentrations of copper and nickel do not justify metal recovery. At one site (M-3), the nickel content is 4.19% and may have potential for metal recovery.

Zinc recovery is possible from some sludges ([Zn] > 10%). The zinc content ranged from 0.019% to as high as 14.4%. In two samples the zinc content was in excess of 14%, making zinc recovery potentially feasible.

The silicon content ranged from 0.8% to 7.2%. The sources of silicon include quartz and other naturally occurring silicates. These silicates may originate from the natural environment and are collected with the AMD stream or silicates, particularly quartz, may be present in the lime source.

Arsenic, boron, cadmium, chromium, mercury, lead and selenium occurred in trace amounts. Concentrations of barium are generally less than 0.01% , except for sludge from N-9 which contains 7.8% barium in the form of barium sulphate.

5.2.3 Sulphate

As expected, all the sludges analyzed contained at least several percent sulphate. In some samples, the sulphate content is greater than 30%. The sulphate content correlated directly with total sulphur content in several of the sludge samples, indicating that all the sulphur present in these samples occurs as sulphate. In two sludge samples (Q-2, N-9) there is considerable excess sulphur present. In Q-2 the excess sulphur is present as pyrite, whereas in N-9 the apparent excess sulphur is barite that was not accounted for in the sulphate determinations.

5.2.4 Carbon and calcium content

The amount of total carbon, carbonate (reported as CO_2) and calcium was measured in the sludge samples. The total carbon content ranged from 0.30% to 7.11%. The amount of CO_2 (as carbonate) present ranges from less than 0.95% to 25.0%. While most of the carbon present is thought to be in the form of calcite, residual carbon (not present as calcite) is thought to result from flocculant usage. In some sludges the amount of residual carbon present is very low (less than 5% of total) while in other sludges there is as much as 25% additional carbon present which is not accounted for through calcite precipitation. Many of the sites with higher amounts of residual carbon in their sludges use flocculants.

The calcium content in the sludges varied from only 3.8% to 26.6%. Calcium is present in two forms, as calcite and as gypsum (or bassanite). A comparison of calcium concentration versus both carbon dioxide and sulphate content indicates in which form the calcium is reporting. For sludges D-1 (a), Q-2 (a), M-3 (f), R-4 (a), S-6 (f) and N-9 (a) the calcium is present as calcite as the molar ratio of Ca:CO_2 approaches unity. Whereas, gypsum is the principal Ca-bearing mineral for sludges F-5 (a), J-7 (f) and J-7 (a). In the remaining samples there is no apparent trend with respect to the Ca, CO_2 and SO_4 content suggesting a mixture of calcite and gypsum is present.

The origin of the calcite remains unclear. Several theories have been postulated with respect to the calcite source, including: carbonation as a result of aeration (Aubé 1997), the presence of unburnt lime in the quick lime (Bisceglia 1966) or the presence of carbonate from the use of soda ash in the process stream.

5.2.5 Neutralization potential

The neutralization potential is a measurement of the alkalinity in the sludge sample and is expressed in tonnes $CaCO_3$ equivalent per 1000 tonnes sludge. NP values for the sludges collected ranged from 108 to 819 tonnes $CaCO_3$ per 1000 tonnes sludge. The excess alkalinity is defined by the net neutralization potential (NNP). NNP values calculated ranged from 74 to 796 tonnes $CaCO_3$ equivalent per 1000 tonnes sludge. The acid potential (AP) is defined as the total sulphur minus the total sulphate sulphur then multiplied by a factor of 31.25. The AP values for the sludges were very low, yielding NNP values approaching NP values. As a result, the following discussions will refer to neutralization potential rather than net neutralization potential.

Four sludge samples have neutralization potentials below 150 tonnes. In three of these four sludges, the HDS process was used for AMD treatment. Neutralization potential can be used as a rough indicator of plant efficiency with respect to lime consumption. Poor lime utilization would result in a higher NP value. Sludge from site F-5 has a neutralization potential of 123 tonnes $CaCO_3$ per 1000 tonnes sludge. This value is low in comparison to other similar type treatment operations. While low NP values are attractive in terms of plant practice, sludges with high neutralization potentials have more neutralization capacity and may be more suited for particular disposal environments, such as co-disposal with tailings or used as a capping material.

Neutralization potentials correlate strongly with carbon dioxide values measured in the sludges. Most if not all the carbon dioxide present in the sludge is in the form of calcite; therefore the neutralization potential is an indirect measure of the CO_2 content in the sludge.

5.3 Mineralogy

A complete mineralogical examination was performed for all the sludge samples collected. Table 9 summarizes the mineralogical characterization, including both XRD and SEM results. The photomicrographs, with their descriptions, and X-ray diffractograms are included in Appendix G.

Mineralogical analyses of all sludge samples showed the presence of a major amorphous phase which is hydrated. Readily leached metal species are commonly associated with this phase, which appears to be effective in scavenging metal species (Al, Cu, Fe, Mg, Na, Ni, Zn) during precipitation. The leachability of the metal species therefore depends upon the stability of the amorphous phase. Carbonates and silica which are present serve to stabilize this phase.

Calcium occurs as calcite, gypsum and bassanite; they occur both as individual grains and in the amorphous phase. The calcite and gypsum grains are relatively pure, implying that the trace metals remain, concentrated, in the amorphous phase. Calcite, gypsum and barite present in the aged sludge samples show varying degrees of recrystallization. The aged sludge samples generally contain a smaller proportion of amorphous material as well as more recrystallized calcite and gypsum. The amount of calcite may indicate the degree of recrystallization and the increased stability of the sludges. Morphological evidence indicates that the calcite is chemical precipitated or recrystallized. No evidence of detrital (lime source) calcite was found. Mineralogical results indicate that the calcite and gypsum are the ‘final’ phases of the aged sludge. In the long term (millennia), the sludge will transform into a carbonate rock with a minor iron oxide phase. The ferric oxyhydroxide present in the sludge will slowly transform into hematite. The fate of metals such as zinc is yet unknown. However, it is expected that they will also be transform into stable

Table 9: Mineralogical Analysis (not in decreasing abundance)

Site Code	Sample	Mineralogy
D-1	fresh	Calcite ($CaCO_3$), amorphous phase (Ca,Mg,Fe,Si,C,S,O, trace Mn,Al)
	aged	Calcite ($CaCO_3$), trace Quartz (SiO_2), Gypsum ($CaSO_4\cdot 2H_2O$), Fe oxide, K-Al, Fe-Mg-Ca, Fe-Mg silicates amorphous phase (Ca,Mg,Fe,Si,C,S,O, trace Mn, Al)
Q-2	aged	Calcite ($CaCO_3$), Quartz (SiO_2), Clinochlore ($((Mg,Fe)_5Al(Si_3,Al)O_{10}(OH)_8)$, Pyrite ($FeS_2$), Talc ($Mg_3Si_4O_{10}(OH)_2$, Ankerite ($CaFe(CO_3)_2$), amorphous phase (Ca,Fe,Mg,CO_3, Si,SO_4,Zn,Na,Cl)
M-3	fresh	Calcite ($CaCO_3$), Quartz (SiO_2), amorphous phase (Fe,Mg,O,C,Si,Na,Ca,Zn,S,Ni,Al,Mn,Cl), $(Mg,Fe,Ca)CO_3$, Fe oxide, Pyrite (FeS_2), $ZrSiO_4$
R-4	fresh	Calcite ($CaCO_3$), amorphous phase (Mg,Al,Si,Fe,S,Ca,Zn,Mn), Mg-Al-Fe silicates, Quartz (SiO_2), Fe oxide, minor Gypsum ($CaSO_4\cdot 2H_2O$)
	aged	Calcite ($CaCO_3$), Quartz (SiO_2), amorphous phase (Fe,Mg,Al,Si,Ca,S,O,Zn,Mn), Fe-Ca-Mg-Al silicates, Na-Fe-Mg-Al silicates
F-5	fresh	Bassanite ($CaSO_4\cdot 0.5H_2O$), Gypsum ($CaSO_4\cdot 2H_2O$), Calcite($CaCO_3$), amorphous phase (Al,O, Fe,Ca,SO_4,Cu,Si,Mg,Mn,Na)
	aged	Gypsum ($CaSO_4\cdot 2H_2O$), Calcite ($CaCO_3$), Quartz (SiO_2), amorphous phase (Al,O, Fe,Ca,S,Cu,Si,Mg,Mn,Na), Fe oxide, Mn-Al oxide, K-Al silcates
S-6	fresh	Gypsum ($CaSO_4\cdot 2H_2O$), Calcite ($CaCO_3$), amorphous phase (Mg,C,O,Na,Ca,S,Zn,Si,Mn,Fe), silicates, Pyrite (FeS_2), Chalcopyrite ($CuFeS_2$), $(Ca,Zn,Mg)CO_3$
	aged	Calcite ($CaCO_3$), Gypsum ($CaSO_4\cdot 2H_2O$), Clinochlore ($((Mg,Fe)_5Al(Si_3\ Al)O_{10}(OH)_8)$, Muscovite ($KAl_2(AlSi_3)O_{10}(OH)_2$, amorphous phase (Mg,C,O,Zn,S,Si,Ca,Na, tr. Mn,Fe)
J-7	fresh	Gypsum ($CaSO_4\cdot 2H_2O$), amorphous phase (Fe,Ca,Al,Mg,,S,O,Si,Mn,Zn)
	aged	Gypsum ($CaSO_4\cdot 2H_2O$), amorphous phase (Fe,Ca,S,O,Al,Mg,Si,P,Mn,Zn), trace Fe oxide, Quartz, silicates
W-8	fresh	Calcite ($CaCO_3$), Bassanite ($CaSO_4\cdot 0.5H_2O$), Ettringite ($Ca_6Al_2(SO_4)_3(OH)_{12}\cdot 26H_2O$), Gypsum ($CaSO_4\cdot 2H_2O$), amorphous phase (Fe,Zn,Mn,Ca,O,Na,Mg,Al,Si), trace Quartz (SiO_2)
	aged	Gypsum ($CaSO_4\cdot 2H_2O$), Bassanite ($CaSO_4\cdot \frac{1}{2}H_2O$), Ettringite ($Ca_6Al_2\ (SO_4)_3(OH)_{12}\cdot 26H_2O$),trace Calcite ($CaCO_3$), amorphous phase (Fe,Ca,S,O,Zn,Al,Mg,Si,Mn,Na), trace Pyrite (FeS_2), trace Sphalerite (ZnS), trace Quartz (SiO_2), trace Fe oxide
N-9	aged	Barite ($BaSO_4$), Calcite ($CaCO_3$), amorphous phase (Mg,Al,Si,Fe,Ba,Ca,S,C,O,Mn,Zn,Na), (Ce,La,Ca,Ba) carbonate
B-10	fresh	Calcite ($CaCO_3$), Quartz (SiO_2), Albite ($NaAlSi_3O_8$), amorphous phase (Fe,Mg,Al,Si,O,C,S,Na,Ni,Cu), Pyrite (FeS_2), Fe oxide
T-11	fresh	Gypsum ($CaSO_4\cdot 2H_2O$), Calcite ($CaCO_3$), amorphous phase (Fe,Ca,Mg,S,O,C,Si,Al,Mn), Fe oxide, Quartz (SiO_2),

oxides or carbonates. Quartz, silicates, sulphides and iron oxide particles found in the sludges are detrital in origin.

True particle sizes of several sludge components were measured using mineralogical techniques; these components include calcite (<1 to 20 μm), detrital particles (<1 to 30 μm) and sludge particles (<1 μm). Aging serves to increase particle size through recrystallization and grain growth.

5.4 Thermal Analysis

Thermal analysis provides information about the form and amount of moisture in the sludge sample, as well as about the different compounds present. Gas evolution at different temperatures represents the occurrence of specific compounds such as carbonates or sulphates. This information coupled with weight loss and XRD analyses before and after heating helps to identify the various sludge components.

Although all the samples were non-magnetic before heating, most of the sludges containing high iron became magnetic after heating in air to 1200°C; these samples included Q-2-a, M-3-f, R-4-f/a, J-7-f/a, W-8-a (trace), B-10-f. There was no evidence of melting for these sludges. Table 10 shows the XRD results of the AMD sludges and their residues after heating to 1200°C in air. The constituents are written in order of decreasing concentrations. Most of the sludges showed evidence of a poorly crystalline phase before and after heating. The XRD pattern for the gypsum found in certain sludges showed different intensities. Moreover, XRD patterns for Fe_2O_3, Fe_3O_4 and $MgFe_2O_4$ are very similar and consequently are reported as a general term, iron oxide. The same applies to the long chain phases such as clinochlore, ettringite and pyroaurite. A general term describing the presence of a chlorite mineral, sulphate mineral and carbonate mineral is reported.

Figures H-1 to H-15 (Appendix H) show the TG/DTA/FTIR results for the sludge samples heated in air (Mikhail and Turcotte 1996a). Thermal characterization in helium was also completed (Mikhail and Turcotte 1996b). Although each sludge is different, common thermal characteristics occur. The DTA curve (dashed) shows negative peaks (endotherms) for dehydration, dehydroxylation, decarbonation, polymorphic transformations, and most

Table 10: Main Phases Identified by X-Ray Diffraction for Thermal Analysis

Site Code	Sample	XRD Before Heating[1]	XRD After Heating in Air
D-1	fresh	$CaCO_3$	$Ca_2Fe_2O_5$, MgO, $Ca(OH)_2$, $CaSO_4$, CaO, trace SiO_2, possible $(Fe,Mg)SiO_3$
	aged	$CaCO_3$, trace SiO_2, $CaSO_4 \bullet 2H_2O$	$Ca_2Fe_2O_5$, U^*, MgO, $Ca(OH)_2$, CaO, $CaSO_4$, trace SiO_2
Q-2	aged	$CaCO_3$, SiO_2, FeS_2, P^*, C^*, possible $Mg(OH)_2$	Fe_3O_4, $Ca_2MgSi_2O_7$, $CaSO_4$, $CaMgSiO_4$
M-3	fresh	$CaCO_3$, SiO_2, $CaSO_4 \bullet 2H_2O$	Fe_2O_3, $CaSO_4$, $CaMgSiO_4$
R-4	fresh	$CaCO_3$	Fe_2O_3, possible $Ca_2MgSi_2O_7$
	aged	$CaCO_3$, SiO_2	Fe_3O_4, possible $MnFe_2O_4$, $Ca_2MgSi_2O_7$
F-5	fresh	$CaSO_4.0.5H_2O$, $CaSO_4.\bullet H_2O$, $CaCO_3$	$CaSO_4$, $Ca_4Al_6O_{12}SO_4$, $Mg(Al,Fe)_2O_4$
	aged	$CaSO_4 \bullet 2H_2O$, trace SiO_2, $CaCO_3$	$CaSO_4$
S-6	fresh	$CaCO_3$, SiO_2, $CaSO_4 \bullet 2H_2O$	MgO, $CaSO_4$, SiO_2
	aged	$CaCO_3$, SiO_2, $CaSO_4 \bullet 2H_2O$	$CaSO_4$, Fe_2O_3, MgO, ZnO
J-7	fresh	$CaSO_4 \bullet 2H_2O$	$CaSO_4$
	aged	$CaSO_4 \bullet 2H_2O$	$CaSO_4$
W-8	fresh	Amorphous	Fe_2O_3, $CaSO_4$
	aged	$CaCO_3$, $CaSO_4 \bullet 0.5H_2O$, P^*, E^*, SiO_2	Fe_2O_3, $CaSO_4$, MgO, SiO_2
B-10	aged	$CaCO_3$, SiO_2, $NaAlSi_3O_8$	$(Mg,Fe)(Cr,Al)_2O_4$, $Ca_2MgSi_2O_7$, $CaMgSiO_4$

[1] All the starting materials contained an amorphous phase.

C^*= Clinochlore $(Mg,Fe)_6(Si,Al)_4O_{10}(OH)_8$ (chlorite mineral)

E^*= Ettringite $Ca_6Al_2(SO_4)_3(OH)_{12}.26H_2O$ (sulphate mineral)

P^* = Pyroaurite $Mg_6Fe_2CO_3(OH)_{16}.4H_2O$ (carbonate mineral)

U^*= Unidentified phase

decompositions. Positive peaks (exotherms) occur mainly for combustion, oxidation and recrystallization. The DTA and FTIR help identify the cause(s) of a weight loss shown on the TG curve. The TG data has been summarized in Table 11. Four temperature ranges were chosen for each sludge. The range from 25 to 200°C represents the H_2O release from hydrates and poorly crystallized hydroxides. The DTA patterns normally matches the FTIR gas evolution profile of

Table 11: TG% Weight Losses in Air

Site Code	Sample	% Weight Loss 25-200°C (hydrates)	% Weight Loss 200-500°C (hydroxides, possible trace carbonates)	% Weight Loss 500-800°C (carbonates)	% Weight Loss 800-1185°C (sulphates, silicates..)
D-1	fresh	4.47	7.02	27.57	0.38
	aged	5.99	7.49	23.73	0.18
Q-2	aged	6.36	7.09	9.72	3.26
M-3	fresh	12.92	9.78	5.52	4.29
R-4	fresh	11.45	12.01	3.35	3.62
	aged	12.8	8.36	5.38	2.0
F-5	fresh	11.79	11.08	5.91	1.16
	aged	15.71	10.91	2.74	4.47
S-6	fresh	5.82	5.97	16.14	0.41
	aged	10.5	9.1	8.45	1.47
J-7	fresh	17.11	6.48	1.06	4.28
	aged	16.66	5.37	0.52	4.54
W-8	fresh	13.24	9.11	1.16	5.96
	aged	14.87	9.28	4.03	1.07
B-10	aged	9.46	10.58	7.06	2.95

H_2O. The second temperature range, 200-500°C, normally corresponds to the dehydroxylation of iron, magnesium and calcium hydroxides. Aluminum hydroxides and certain carbonate minerals will also decompose in this temperature range. In certain sludges the FTIR gas evolution showed traces of ammonia but the levels are too low to identify the source. From 500 to 800°C the weight loss is essentially due to the decomposition of calcium carbonate. Any water evolution at this temperature is due to the dehydroxylation of clays and similar minerals. Above 800°C solid phase reactions occur between the various oxides produced from earlier decompositions (MgO, CaO), often including silica and iron oxides. There is no weight change associated with these solid phase reactions.

In certain sludges a small weight loss occurs above 1000°C, coinciding with SO_2 gas evolution which may be due to an early decomposition of poorly crystalline $CaSO_4$ or to the reaction of $CaSO_4$ with another constituent such as silica. Anhydrous $CaSO_4$ is usually stable up to 1200°C in air. Other sulphates, not identified by XRD may also decompose in this temperature range. The trace HF and/or HCl found in the evolved gases of certain sludges, may be due to the partial decomposition of complex silicates or other unknown constituents. The thermal behavior of the sludges are very similar in air and helium up to 800°C. Above this temperature, the calcium sulphate (and other sulphates) show a wide range of decomposition temperatures.

Table 12 shows the estimated composition of the AMD sludges, based on the information obtained from the thermal characterization in air and the XRD results. The thermal characterization in helium, in the final report, may help confirm these compositions.

5.5 Characterization Conclusions

The percent of solids in the sludges studied ranged from 2.4% to 32.8%. As expected, the percent solid measurements were directly related to the treatment process. HDS processes produced much denser sludges in terms of solids content than the conventional and basic treatment processes. Generally, the sludges tend to densify with age though natural dewatering and freeze-thaw. The degree of densification with time is directly dependent on the amount of moisture present in the sludge.

The sludge particles were generally aggregated masses displaying either uni- or bimodal particle size distributions attributed to the degree of homogeneity of the sludge and the amounts of detrital material and calcite (unreacted lime) present. Results indicated that the denser sludges are composed of "particles" of smaller size. Low density sludges report higher median particle size distributions either because of a higher degree of porous particle aggregation and less true particle growth or presence of larger calcite (unreacted lime) particles. HDS treatment sludges display narrower size distribution, indicative of a better crystallization control during hydrolysis/precipitation. In all cases except one, the median particle size increased with aging.

Table 12: Estimated Composition of AMD Sludges Based on Thermal Analysis and XRD

Site Code	Sample	Estimated composition based on thermal analysis and XRD
D-1	fresh	$CaCO_3$, $Ca(OH)_2$, $Mg(OH)_2$, $CaSO_4 \bullet ?H_2O$, iron oxide, SiO_2
	aged	$CaCO_3$, $Ca(OH)_2$, $Mg(OH)_2$, $CaSO_4 \bullet ?H_2O$, iron oxide, SiO_2
Q-2	aged	$CaCO_3$, carbonate mineral, chlorite mineral, $CaSO_4 \bullet ?H_2O$, SiO_2, iron oxide, $Ca(OH)_2$, $Mg(OH)_2$, Zn phase (oxide?), possible FeS_2, trace Cl^-
M-3	fresh	$CaCO_3$, carbonaceous material, $Ca(OH)_2$, $Mg(OH)_2$, $CaSO_4 \bullet 2H_2O$, iron oxide, SiO_2, trace NH_4^+, Cl^-
R-4	fresh	$CaCO_3$, carbonaceous material, $Ca(OH)_2$, $Mg(OH)_2$, $CaSO_4 \bullet ?H_2O$, iron oxide, SiO_2, trace NH_4^+, Cl^-, F^-
	aged	$CaCO_3$, carbonaceous material, $Ca(OH)_2$, $Mg(OH)_2$, $CaSO_4 \bullet ?H_2O$, iron oxide, SiO_2, trace NH_4^+, Cl^-, F^-
F-5	fresh	$CaSO_4 \bullet 0.5H_2O$, $CaSO_4 \bullet 2H_2O$, $Ca(OH)_2$, $Mg(OH)_2$, $CaCO_3$, iron oxide, possible alumina hydrate, trace Cl^-, F^-
	aged	$CaSO_4 \bullet 2H_2O$, possible alumina hydrate, $Mg(OH)_2$, trace SiO_2, $CaCO_3$, trace Cl^-, F^-
S-6	fresh	$CaCO_3$, $CaSO_4 \bullet 2H_2O$, $Ca(OH)_2$, $Mg(OH)_2$, SiO_2, iron phase, trace Cl^-
	aged	$CaCO_3$, $CaSO_4 \bullet 2H_2O$, $Ca(OH)_2$, $Mg(OH)_2$, iron phase, SiO_2, zinc phase (oxide), trace Cl^-
J-7	fresh	$CaSO_4 \bullet 2H_2O$, iron phase, $Mg(OH)_2$, trace $CaCO_3$, SiO_2, Cl^-, F^-
	aged	$CaSO_4 \bullet 2H_2O$, iron phase, $Mg(OH)_2$, trace SiO_2, Cl^-, F^-
W-8	fresh	$CaSO_4 \bullet ?H_2O$, $Ca(OH)_2$, $Mg(OH)_2$, iron phase, SiO_2, zinc phase (oxide?), trace SiO_2, Cl^-
	aged	$CaCO_3$, $CaSO_4 \bullet 0.5H_2O$, $Mg(OH)_2$, carbonate mineral, sulphate mineral, SiO_2, iron oxide, trace Cl^-
B-10	fresh	$CaCO_3$, carbonaceous material, $Ca(OH)_2$, $Mg(OH)_2$, $CaSO_4 \bullet ?H_2O$, iron oxide, SiO_2, trace NH_4^+, Cl^-

Through aging, particle dissolution and recrystallization occur, leading to particle growth and aggregation.

The pH values for the sampled sludges were alkaline and ranged from 8.2 to 11.4. In most cases aged sludges showed a lower pH than their fresh counterparts. E_h values ranged from 58 to 315 mV with the aged sludges commonly recording the lower values. In some samples, the oxidation potential was low (< 100 mV) suggesting that aeration is either needed or not completely effective. The ferric hydrolysis product is more stable than the ferrous form and is also in effective adsorbing several other metal species as it is precipitated.

Metal recovery is possible, in terms of zinc, for at least two of the sludges characterized. The HDS process appears to produce sludges with higher metal contents making metal recovery more attractive.

The available excess alkalinity in the sludges expressed in terms of neutralization potential ranged from 108 to 819 tonnes $CaCO_3$ per 1000 tonnes sludge and gives an indication of plant performance and potential sludge stability. Efficient lime utilization and reaction generally leads to sludges containing gypsum (low NP) rather than calcite/unreacted lime (high NP). Over-dosing through poor slaking and insufficient mixing will also lead to high NP values.

Chemical, mineralogical and thermal analysis has thoroughly characterized the sludge composition. Chemical analysis quantifies the sludge building components in terms of elemental determinations, mineralogy identifies specific mineral species and provides information on sludge morphology, and thermal analysis uses moisture loss and gas emission determinations to document those phases to mineralogy.

Table 13 summarizes the sludge compositions compiled from chemical, mineralogical and thermal data. All the sludges contain an amorphous phase. This hydrated mass serves a collector for several metal species (Fe, Al, Zn, Cu, Ni) as well as carbon and silicon. Calcium commonly occurs in three crystalline forms; as gypsum, as calcite or as bassanite, however $Ca(OH)_2$ is present in many of the sludge samples analyzed. The precipitation of any one of these minerals depends on influent chemistry and process conditions (including lime grade).

The origin of the calcite remains unclear. Several theories have been postulated with respect to the calcite source, including; carbonation as a result of aeration, the presence of unburnt lime in the quick lime or the presence of carbonate from the use of soda ash in the process stream. Morphological analyses of the sludges in this study indicates that calcite is a primary mineral and has been chemical precipitated or recrystallized.

Silicates and to a lesser extent sulphides are a common occurrence in the sludge samples. The origin of these minerals is primarily detrital (dust, runoff, disposal environment). Silicates may also originate from the lime source. In light of the questions regarding lime grade as a source

Table 13: Estimated Composition of AMD Sludges based on Chemical Analyses, Mineralogy and Thermal Analysis

Site Code	Sample	Sludge Composition
D-1	fresh	amorphous phase (Ca,Mg,Fe,Si,C,S,O, trace Mn,Al), $CaCO_3$, $Ca(OH)_2$, $Mg(OH)_2$, $CaSO_4 \cdot ?H_2O$, Fe oxide, SiO_2
	aged	amorphous phase (Ca,Mg,Fe,Si,C,S,O, trace Mn, Al), $CaCO_3$, trace SiO_2, $CaSO_4 \cdot 2H_2O$, Fe oxide, K-Al, Fe-Mg-Ca, Fe-Mg silicates, $Ca(OH)_2$, $Mg(OH)_2$, Fe oxide
Q-2	aged	amorphous phase (Ca,Fe,Mg,CO_3, Si,SO_4,Zn,Na,Cl), $CaCO_3$, SiO_2, $(Mg,Fe)_5Al(Si_3\ Al)O_{10}(OH)_8$, FeS_2, $Mg_3Si_4O_{10}(OH)_2$, $CaFe(CO_3)_2$, $Ca(OH)_2$, $Mg(OH)_2$, Fe oxide, Zn phase (oxide?), trace Cl^-
M-3	fresh	amorphous phase (Fe,Mg,O,C,Si,Na,Ca,Zn,S,Ni,Al,Mn,Cl), $CaCO_3$, SiO_2, $Ca(OH)_2$, $Mg(OH)_2$, $CaSO_4 \cdot ?H_2O$, $(Mg,Fe,Ca)CO_3$, Fe oxide, FeS_2, $ZrSiO_4$, trace Cl^-, NH^+
R-4	fresh	amorphous phase (Mg,Al,Si,Fe,S,Ca,Zn,Mn), $CaCO_3$, Mg-Al-Fe silicates, $Ca(OH)_2$, $Mg(OH)_2$, SiO_2, Fe oxide, carbonaceous material, minor $CaSO_4 \cdot 2H_2O$, trace Cl^-, NH^+, F^-
	aged	amorphous phase (Fe,Mg,Al,Si,Ca,S,O,Zn,Mn), $CaCO_3$, SiO_2, Fe-Ca-Mg-Al silicates, Na-Fe-Mg-Al silicates, Fe oxide, $Ca(OH)_2$, $Mg(OH)_2$, carbonaceous material, minor $CaSO_4 \cdot 2H_2O$, trace Cl^-, NH^+, F^-
F-5	fresh	amorphous phase (Al,O, Fe,Ca,SO_4,Cu,Si,Mg,Mn,Na), $CaSO_4 \cdot 0.5H_2O$, $CaSO_4 \cdot 2H_2O$, $CaCO_3$, $Ca(OH)_2$, $Mg(OH)_2$, possible alumina hydrate, Fe oxide, trace Cl^-, F^-
	aged	amorphous phase (Al,O, Fe,Ca,SO_4,Cu,Si,Mg,Mn,Na), $CaSO_4 \cdot 2H_2O$, $CaCO_3$, SiO_2, possible alumina hydrate, $Mg(OH)_2$, Fe oxide, Mn-Al oxide, K-Al silcates, trace Cl^-, F^-
S-6	fresh	amorphous phase (Mg,C,O,Na,Ca,S,Zn,Si,Mn,Fe), $CaSO_4 \cdot 2H_2O$, $CaCO_3$, $Ca(OH)_2$, $Mg(OH)_2$, SiO_2, silicates, FeS_2, $CuFeS_2$, $(Ca,Zn,Mg)CO_3$, trace Cl^-
	aged	amorphous phase (Mg,C,O,Zn,S,Si,Ca,Na, tr. Mn,Fe), $CaCO_3$, $CaSO_4 \cdot 2H_2O$, $(Mg,Fe)_5Al(Si_3\ Al)O_{10}(OH)_8$, $Ca(OH)_2$, $Mg(OH)_2$, SiO_2, $KAl_2(AlSi_3)O_{1o}(OH)_2$, Fe phase, Zn phase (oxide?), trace Cl^-
J-7	fresh	amorphous phase (Fe,Ca,Al,Mg,SO_4,Si,Mn,Zn), $CaSO_4 \cdot 2H_2O$, $Mg(OH)_2$, trace $CaCO_3$, SiO_2, trace Cl^-, F^-
	aged	amorphous phase (Fe,Ca,S,O,Al,Mg,Si,P,Mn,Zn), $CaSO_4 \cdot 2H_2O$, $Mg(OH)_2$, trace Fe oxide, SiO_2, silicates, trace Cl^-, F^-
W-8	fresh	amorphous phase (Fe,Zn,Mn,Ca,O,Na,Mg,Al,Si), $CaCO_3$, $CaSO_4 \cdot 0.5H_2O$, $Ca_6Al_2(SO_4)_3(OH)_{12} \cdot 26H_2O$, $CaSO_4 \cdot 2H_2O$, Fe oxide, Zn phase (oxide?), $Ca(OH)_2$, $Mg(OH)_2$, trace SiO_2, Cl^-
	aged	amorphous phase (Fe,Ca,S,O,Zn,Al,Mg,Si,Mn,Na), $CaSO_4 \cdot 2H_2O$, $CaSO_4 \cdot \frac{1}{2}H_2O$, $Ca_6Al_2\ (SO_4)_3(OH)_{12} \cdot 26H_2O$, $Mg(OH)_2$, carbonate material, sulphate material, trace $CaCO_3$, FeS_2, ZnS, SiO_2, Fe oxide
N-9	aged	amorphous phase (Mg,Al,Si,Fe,Ba,Ca,S,C,O,Mn,Zn,Na), $BaSO_4$, $CaCO_3$, (Ce,La,Ca,Ba) carbonates
B-10	fresh	amorphous phase (Fe,Mg,Al,Si,O,C,S,Na,Ni,Cu), $CaCO_3$, SiO_2, $NaAlSi_3O_8$, FeS_2, Fe oxide, $Ca(OH)_2$, $Mg(OH)_2$, SiO_2, Fe oxide, carbonaceous material

Site Code	Sample	Sludge Composition
T-11	fresh	amorphous phase (Fe,Ca,Mg,S,O,C,Si,Al,Mn), $CaSO_4 \cdot 2H_2O$, $CaCO_3$, Fe oxide, SiO_2

of impurities, it is recommended that a sample of the lime be collected at the same time in which the fresh sludge sample is collected. An ettringnite compound is present in two sludge samples. The occurrence of this compound is thought to depend on the influent composition rather than any process factors.

The use of chemical, mineralogical and thermal analyses allows for a thorough characterization of AMD treatment sludges. Chemical analysis provides elemental determinations, mineralogy identifies specific mineral species and provides information on sludge morphology, while thermal analysis provides information on the quantity and form of contained water, the speciation of certain major and minor constituents and potential gaseous releases from the sludges. Mineralogical analysis coupled with thermal analysis provides a quantitative evaluation of the sludge samples as well as some insight into the stability of the aged samples. Chemical analysis for major metals and calcium along with NP determination and leaching tests provide a rudimentary indication of the sludge composition and corresponding metal leachability.

6.0 SLUDGE STABILITY

The Canadian mineral industry is faced with questions related to the long term stability of AMD treatment sludges, and their environmentally acceptable disposal. Empirical leaching tests are often used as the best practicable means of predicting the propensity for the metals in the sludges to be re-mobilized. However, the relevance of leaching tests to field conditions is often difficult to identify, and may lead to overly optimistic or pessimistic assessments of long term sludge stability (Mitchell and Atkinson 1995). Nevertheless, leachability data may provide an indication of relative sludge stability under possible disposal scenarios.

6.1 Sludge Leachability

AMD treatment sludge samples were leached using two protocols, the Ontario LEP and the Modified LEP. Use of acetic acid in the Ontario LEP mimics the organic acids expected to be present in a municipal landfill and assumes co-disposal of mineral processing and municipal wastes. On the other hand, the mixture of sulphuric and nitric acids used in the Modified LEP better simulates the inorganic acids that are likely to come in contact, through acidic precipitation, with sludges disposed in ponds, either with or without a water cover. In the Ontario LEP the leachant is acidified with up to 200 mL of 0.5 N acetic acid while the Modified LEP uses up to 200 mL of pH 4.5 synthetic acid rain. Because of the relatively high alkalinity of the sludge samples, the maximum volume of acid was required in all tests. In this situation, the Ontario LEP provides more acid for neutralizing excess alkalinity than does the Modified LEP. It must be noted however, that the Modified LEP was designed to mimic a potential disposal scenario in terms of the source of acid, rather than to match the Ontario LEP in terms of the amount of available acid.

Virtually all lime sludges will become unstable with the addition of enough acid. Nonetheless, the rate of acidification must be considered when choosing a test to assess the leachability of sludges. For example, Orava et al. (1995) calculated the alkalinity available in a 3-metre deep deposit of sludge at 40% solids and 10% free $CaCO_3$. About 40,000 years of marginally acidic rainfall percolating through the sludge would be necessary to give a modest pH depression.

For the sludge samples tested, the final leachant pH ranged from 7.7 to 10.6 with the Modified LEP and from 5.9 to 9.9 with the Ontario LEP. In each case, the final leachant pH for the modified test was greater than or equal to that for the regulatory test. Since metal hydroxide solubilities increase with decreasing pH below about pH 9.5 (Vachon et al. 1987), sludge leachability is strongly dependent on the final leachant pH. This relationship was observed in the GML (1987) study. It has important implications for long term sludge stability since sludges with higher neutralizing potential (NP) are less likely to be leachable.

The concentrations of metals in the leachates are given in Table 14 for the Ontario LEP and Table 15 for the Modified LEP. In general less metal was leached from the sludges when they were subjected to the Modified LEP as opposed to the Ontario LEP.

The level of metal mobility is also a function of the initial metal concentration in the sludge, as noted in the GML (1987) report and confirmed in this study. For example, the highest zinc concentrations occurred in two fresh samples W-8 at 14.2% and S-6 at 14.4 %. The resulting Ontario LEP leachate zinc concentration for W-8 is at least two orders of magnitude greater than the zinc concentration in all the other leachates including S-6. However, sample S-6 has a much higher NP at 523 tonnes $CaCO_3$ equivalent per 1000 tonnes sludge than sample W-8 at 142 tonnes $CaCO_3$ equivalent per 1000 tonnes sludge. The final leachant pH for S-6 was 8.5 while that for W-8 was 6.8. This comparison illustrates the impact of NP on final leachant pH and hence on metal leachability.

Acetic acid will mobilize some metals such as lead because of the high solubility of the metal acetate. Higher concentrations of lead might therefore be expected in the Ontario LEP leachate compared to the modified LEP leachate. This was not observed for these sludges, presumably because the lead concentrations in the sludges are so low (all <0.45%).

For both the Ontario and the modified leach tests the aged sludges seem to have a somewhat lower propensity for metal leaching than the fresh sludges. This correlates with mineralogical data which indicates that sludge stability may improve with age.

Table 14: Sludge Leaching Characteristics, Ontario Leachate Extraction Procedure

Site Code	Sample	Leachate Composition (μg/L)												
		Ag	As	Ba	B[1]	Cd	Cr	Cu	Fe_t[1]	Hg	Ni	Pb	Se	Zn[1]
D-1	fresh	<2	<5	9.4	124.2	<1	<7	<13	<40	<4	4.9	<2	<24	<50
	aged	<2	<5	38.8	397.6	<1	<7	73.3	<40	<4	769.3	<2	<24	<50
Q-2	fresh	<4	<5	109.0	120	35.7	<24	<196	<40	–	<91	<0.7	144.8	0.16
	aged	<6	<6	104.3	149.8	43.0	<34	<235	<40	<27	8.0	<10	153.2	140
M-3	fresh	<6	<6	86.8	293.7	<4	<34	<235	<40	<27	942.6	<10	<97	<60
R-4	fresh	<6	<6	30.0	68.1	<4	<34	<235	<40	<27	32.8	<10	<97	330
	aged	<6	<6	22.4	<62	<4	<34	<235	<40	<27	11.8	<10	<97	160
F-5	fresh	<6	<6	23.1	<77	<3	<8	<28	<40	<40	<6	<5	<35	<60
	aged	<6	<6	65.5	<77	<3	<8	<28	<40	<40	413.0	<5	<35	300
S-6	fresh	<4	<6	60.2	79	47.8	<24	<196	<50	<30	<112	<1	132.4	480
	aged	<2	<5	29.4	150.0	3.7	<7	<13	<40	<4	8.2	<2	186.3	<50
J-7	fresh	<2	<5	219.2	927.1	<1	<7	<13	<40	<4	25.2	<2	<24	<50
	aged	<2	<5	53.3	278.6	<1	<17	<12	<40	<4	60.1	<2	<24	<50
W-8	fresh	25.7	<6	117.7	228	375.5	<24	<196	<40	<40	<91	<5	<35	27,200
	aged	<6	<6	64.9	<77	<3	<8	<28	<40	<40	24.9	<5	<35	80
N-9[2]	aged	<6	<6	49.0	1719.4	3.2	<24	<196	<40	<40	391.7	<5	<35	270
B-10	fresh	<6	<6	40.8	<62	<4	<34	<235	<40	<27	<7	<10	<97	<60
T-11[3]	fresh	<6	<6	16.4	124.5	<3	<8	<28	<40	<40	<6	<5	<35	<60
Most Stringent Cdn. Regulated Limit[4]		5,000	5,000	100,000	500,000	500	5,000	10,000	1x10^6	100	5,000	5,000	1,000	10,000

[1] Since analytical results were originally in mg/L, trailing zeros may not be significant. [2] U = 5,015 μg/L [3] U < 11 μg/L [4] See Table 16 (U limit is 2,000 μg/L)

Table 15: Sludge Leaching Characteristics, Modified Leachate Extraction Procedure

Site Code	Sample	Leachate Composition (μg/L)												
		Ag	As	Ba	B[1]	Cd	Cr	Cu	Fe_t[1]	Hg	Ni	Pb	Se	Zn[1]
D-1	fresh	<2	<5	9.8	131.8	<1	<7	<13	<40	<4	5.2	<2	<24	<50
	aged	<2	<5	16.9	74.2	<1	<7	28.3	<40	<4	26.5	<2	<24	<50
Q-2	fresh	<4	<5	48.4	200	<0.7	<24	<196	<40	–	<91	1.0	141.4	40
	aged	<6	<6	48.8	95.0	4.4	<34	<235	<40	<27	<7	<10	115.7	<60
M-3	fresh	<6	<6	38.3	111.6	<4	<34	<235	<40	<27	187.0	<10	<97	<60
R-4	fresh	<6	<6	<5	<62	<4	<34	<235	<40	<27	<7	<10	<97	<60
	aged	<6	<6	<5	<62	<4	<34	<235	<40	<27	<7	<10	<97	<60
F-5	fresh	<6	<6	23.5	<77	<3	<8	<28	<40	<40	<6	<5	<35	<60
	aged	<6	<6	13.8	<77	<3	<8	<28	<40	<40	<6	<5	<35	<60
S-6	fresh	<3	<3	23.1	<49	9.7	<9	<94	<40	<30	19.9	<9	89.8	5200
	aged	<2	<5	28.2	147.8	3.5	<7	<13	<40	<4	7.2	<2	177.5	<50
J-7	fresh	<2	<5	47.9	317.8	<1	<7	<13	<40	<4	4.6	<2	<24	<50
	aged	<2	<5	7.3	66.1	<1	<7	<13	<40	<4	5.3	<2	<24	<50
W-8	fresh	<6	<6	32.3	<77	<3	<8	<28	<40	<40	<6	<5	<35	<60
	aged	<6	<6	24.4	<77	<3	<8	<28	<40	<40	24.8	<5	<35	<60
N-9[2]	aged	<6	<6	24.9	762.4	<3	<8	<28	<40	<40	<24.2	<5	<35	<60
B-10	fresh	<6	<6	40.7	<62	<4	<34	<235	<40	<27	<7	<10	<97	<60
T-11[3]	fresh	<6	<6	16.3	117.0	<3	<8	<28	<40	<40	<6	<5	<35	<60
Most Stringent Cdn. Regulated Limit[4]		5,000	5,000	100,000	500,000	500	5,000	10,000	1x10^{6}	100	5,000	5,000	1,000	10,000

[1] Since analytical results were originally in mg/L, trailing zeros may not be significant. [2] U = 405 μg/L [3] U = 11 μg/L [4] See Table 16 (U limit is 2,000 μg/L)

In evaluating metal leachability with respect to sludge mineralogy it appears that sludge stability depends on the stability of the amorphous mass rather than on the other sludge components. Readily leached metal species such as zinc are commonly associated with the amorphous phase of the sludge. Several metal species are known to adsorb on the surface of ferric oxyhydroxide during precipitation. Although this amorphous phase appears to be effective in scavenging metal species during precipitation there is no evidence that this phase serves to immobilize the species.

6.2 Regulatory Context of AMD Treatment Sludge

6.2.1 Comparison to regulated limits

AMD treatment sludges are waste products which may be subject to waste management regulations. A leachate extraction test may be used to evaluate if the waste is capable of yielding a leachate which exceeds regulated concentration limits for selected contaminants. When a waste fails the test in relation to the limits specified in a particular jurisdiction, the waste may be classified as hazardous.

All but two of the sludge samples subjected to the Ontario LEP passed the test when the leachate concentrations for metals are compared to the regulated limits governing the classification of hazardous waste material in Canadian jurisdictions (see Table 16). The leachate concentrations were generally at least five times lower than the most stringent of the regulated limits. Of the samples which failed the Ontario LEP, one was a fresh sludge from a base metal operation (W-8), while the second was an aged sludge from a uranium mine (N-9). The base metal mine sludge failed with respect to zinc having a concentration in the leachate of 27.2 mg/L. There are three jurisdictions in Canada which have a regulated limit for zinc, Québec at 10 mg/L and Alberta and British Columbia at 500 mg/L. This sludge would actually fail only in comparison to Québec's current regulation. It must be noted however, that Québec's proposed new regulated limits do not include zinc. The uranium mine sludge failed in terms of uranium with a concentration in the leachate of 5.02 mg/L. The regulated limits for uranium are 2.0 mg/L in most Canadian jurisdictions and 10.0 mg/L in British Columbia. None of the sludge samples

Table 16: Comparison of Toxic Leachate Constituents used in Canadian Jurisdictions

Constituent	Regulated Limits (mg/L)[1]					
	TDGR[2]	Québec[3] (current)	Québec[4] (draft)	Ontario[5] and Manitoba[6]	B.C.[7]	Alberta[8]
Arsenic (As)	5.0	5.0	5.0	5.0	5.0	5.0
Barium (Ba)	100.0		100	100.0	100.0	100.0
Boron (B)	500.0		500	500.0	500.0	500.0
Cadmium (Cd)	0.5	2.0	0.5	0.5	0.5	1.0
Chromium (Cr)	5.0	5.0	5.0	5.0	5.0	5.0
Cobalt (Co)						100.0
Copper (Cu)		10			100.0	100.0
Iron (Fe)						1000.0
Lead (Pb)	5.0	5.0	5.0	5.0	5.0	5.0
Mercury (Hg)	0.1	0.2	0.1	0.1	0.1	0.2
Nickel (Ni)		10				5.0
Selenium (Se)	1.0	1.0	1.0	1.0	1.0	1.0
Silver (Ag)			5.0	5.0	5.0	5.0
Uranium (U)	2.0		2.0	2.0	10.0	2.0
Zinc (Zn)		10			500.0	500.0

[1] Numerical values are recorded as given in the source.

[2] Transportation of Dangerous Goods Regulations, Part III, ∮ 3.27(3) (Transport Canada 1997).

[3] Hazardous Waste Regulation [Q-2, r.3.01] Schedule III (Government of Québec 1992).

[4] Draft Hazardous Materials Regulation (Government of Québec 1995); comments from the public in response to this draft have included a recommendation that silver be removed from the list of contaminants in order to harmonize with the TDGR (St-Laurent 1996).

[5] General - Waste Management Regulation (Ont. Reg. 347) Schedule 4 (Government of Ontario 1994b).

[6] Classification Criteria for Products, Substances and Organisms Regulation (Man. Reg. 282/87) Schedule B, Table 1 (Government of Manitoba 1987).

[7] Special Waste Regulation (B.C. Reg. 63/88) Schedule 4, Table 1 (Government of British Columbia 1992). Draft amendments to this regulation, which will be released for public consultation in 1997, will likely

include recommendations for changes to the Leachate Quality Standards (Bindra 1996).

[8] Schedule to the Alberta User Guide for Waste Managers, Table 2 (Government of Alberta 1995).

failed when tested with the Modified LEP. Therefore most of the sludges sampled are stable with respect to the applied leach protocols when compared to current Canadian regulated limits.

6.2.2 Leach test protocols

Numerous leach protocols have been developed to test solid wastes. None of these leaching tests have been specifically designed for evaluating AMD treatment sludge leachability. However, some of the research and test methods simulate an actual sludge environment more closely than the regulatory leach protocols do (e.g., through the use of simulated acid rain rather than acetic acid as a leachant). Appendix I provides background information and a comparative summary of a number of regulatory and research leach protocols from Canada and the United States. Further protocol details (including the intended purpose, test variables and noteworthy features) are given in Appendix J.

The regulatory leach test adopted by the United States Environmental Protection Agency (U.S. EPA) is the Toxicity Characteristic Leaching Procedure (TCLP) (United States Environmental Protection Agency 1990). Since the development of this test was based on a municipal/industrial co-disposal mismanagement scenario, the TCLP uses an acetic acid leaching medium. The applicability of the TCLP to mineral processing wastes is currently being debated in the United States. The National Mining Association contends that the Synthetic Precipitation Leaching Procedure (SPLP) is a more appropriate test (United States Environmental Protection Agency 1995). The SPLP is a variation of the TCLP which uses a synthetic acid rain as the leaching medium and is considered to more closely simulate in situ conditions for mining waste.

Most of the regulatory leach test protocols currently used in Canada are based on the predecessor of the TCLP, the U.S. EPA Extraction Procedure Toxicity Test (EP Tox) (United States Environmental Protection Agency 1986). Compared to the EP Tox, the TCLP incorporates a number of procedural and equipment modifications. The metal concentrations in TCLP leachates generally tend to be slightly higher than those in EP Tox leachates (United States Environmental Protection Agency 1989). Modifications to waste management regulations which may replace existing EP Tox-based leach tests with the TCLP have been initiated in Québec (St-Laurent 1996) and British Columbia (Bindra 1996). As well, Environment Canada has initiated a review of the applicability and suitability of the leach test used in the Transportation of Dangerous Goods Regulations (TDGR). The adoption of a TCLP-type test is under consideration (Wittwer 1996). Many provinces and territories utilize the provisions of the TDGR for waste management control (e.g., New Brunswick, Newfoundland, Northwest Territories, Nova Scotia, Saskatchewan and the Yukon).

While regulatory leach tests for classification of hazardous wastes in Canada are likely to continue to involve the use of acetic acid in the protocol, more appropriate tests, such as the Modified LEP, need to be considered for assessing the leachability of AMD treatment sludges for on-site disposal in a pond environment. Ultimately, the context within which sludge leachability (stability) is measured must be kept in mind when a leach test is applied. It should be noted that the real environment of sludge disposal with tailings has not been addressed in this study. The long term effects of such action are uncertain and need to be evaluated.

6.2.3 Regulations and guidelines

As previously stated, a tested waste is designated as hazardous if the concentration of a contaminant in the leachate exceeds the regulated limit for that contaminant. The regulated limits for most jurisdictions are based on drinking water guidelines as described below.

The U.S. EPA defined the toxicity of a waste by attempting to measure the potential for the toxic constituents present in the waste to leach out, thereby contaminating groundwater and surface water, and by extension drinking water sources, at levels of health or environmental concern. The National Interim Primary Drinking Water Standards (NIPDWS) were used as the basis for the regulated limits because at that time, the NIPDWS were the only health-based regulatory standards available. To account for the likely dilution and attenuation of the toxic constituents that would occur as they travelled from the point of generation to a drinking water source, the EPA multiplied the NIPDWS by a ‘dilution and attenuation factor’ (DAF) of 100. The DAF of 100 was not derived from any model or empirical data, but rather was an estimated factor that the EPA believed would indicate substantial hazard (United States Environmental Protection Agency 1995).

The U.S. EPA regulated limits (see Table I-3 in Appendix I) reflect the eight metals from the NIPDWS of the day. However, many of these limits do not correlate by a factor of 100 with the current drinking water standards (see Table I-4 in Appendix I) since drinking water standards have been amended since the promulgation of the first U.S. EPA leach test protocol. It is notable that the original suite of eight metals used by the U.S. EPA did not include copper, nickel and zinc and hence, many jurisdictions do not include limits for these metals (see Table 16 for Canadian jurisdictions and Table I-3 in Appendix I for some American jurisdictions). However, Alberta does include all three metals and British Columbia includes copper and zinc in their limits. In the move towards national harmonization in Canada, there is a strong possibility that these metals will be added to the regulated limits in most provinces/territories.

Superimposed upon the issues of: i) which leach test is applicable to mineral processing wastes; and ii) which regulated limits are compared to the leachate concentrations, are the waste management regulations themselves, along with other pertinent pieces of legislation which regulate mining operations in each jurisdiction. The waste management regulations may specify whether certain wastes need to be tested or not. In most provinces and territories, the testing of AMD treatment sludge and its storage/disposal is controlled by site-specific licences or permits based on appropriate legislation. For example, in Ontario AMD treatment sludges are commonly stored/disposed in mill tailings areas or sludge ponds, which in turn are regulated through site-specific Certificates of Approval under the Ontario Water Resources Act. See Appendix I for a review of Canadian and American hazardous waste regulations and other pertinent regulations and guidelines as they apply to the leachability testing of AMD treatment sludges.

6.3 Sludge Stability Conclusions

The sludge samples collected were tested for metal leachability using both an acetic acid test (Ontario LEP) and a synthetic acid rain test (Modified LEP). The aged sludges seem to have a somewhat lower propensity for metal leachability than the fresh sludges. All but two of the sludge samples subjected to the Ontario LEP passed the test when the leachate concentrations for metals are compared to the regulated limits governing the classification of hazardous waste material in Canadian jurisdictions. A fresh sludge from a base metal operation failed on zinc and sludge samples subjected to the Ontario LEP passed the test when the leachate concentrations for metals are compared to the regulated limits governing the classification of hazardous waste material in Canadian jurisdictions. A fresh sludge from a base metal operation failed on zinc and an aged sludge from a uranium mine failed on uranium. There are only three jurisdictions in Canada which have a regulated limit for zinc and this sludge would actually fail only in comparison to Québec's current regulation. It must be noted however, that Québec's proposed new regulated limits do not include zinc. None of the sludge samples failed when tested with the Modified LEP. The leachate concentrations from both tests were generally at least five times lower than the most stringent of the regulated limits.

Therefore, fresh AMD treatment sludges would not generally be classified as hazardous wastes based on current leaching protocols and regulated contaminant limits. Aged sludges are even less likely to be classified as hazardous wastes. Based on the samples tested, this work has underlined that while sludge stability is an issue, greater emphasis should be placed on sludge disposal and volume reduction.

Leach test protocols must be correlated directly with the disposal environment, be it pond disposal, co-disposal with tailings or another environment. The Modified LEP used to evaluate metal mobility in this study estimates sludge stability in a pond environment. The co-disposal (with tailings) environment is more complex and is not adequately represented by the Modified LEP.

7.0 CONCLUSIONS

This report provides a data bank of lime treatment sludge characteristics which has been applied here both in the discussion of sludge stability and to draw the following conclusions:

- ☐ Physical and chemical characteristics of the sludges sampled showed marked differences between sites and also between fresh and aged sludges from the same site.
- ☐ Sludge density increases with sludge aging; however the method of disposal (water cover, sand filtration beds, etc.) does not appear to profoundly influence this process.
- ☐ Sludge volume/density remains an issue at many sites where percent solids measurements were less than 10% even after aging over several freeze-thaw cycles.
- ☐ Metal recovery may be possible for some of the characterized sludges, particularly those with zinc contents in excess of 10%.
- ☐ NP/NNP and metal content may be rough indicators of sludge stability. Sludges with low metal contents and higher free alkalinity tend to indicate a lower propensity for metal leachability.
- ☐ In general, the aged sludges show an increase in stability relative to the fresh samples as indicated by the leaching results and supported by the mineralogical data and particle size analyses.
- ☐ Fresh AMD treatment sludges would not generally be classified as hazardous wastes based on current leaching protocols and regulated contaminant limits. Previously disposed sludges are even less likely to be classified as hazardous wastes.
- ☐ Mineralogical data suggest long term aging (thousands of years) would transform the sludge into a stable carbonate rock with a minor iron oxide component.

This data bank may be used to assist operators, researchers and regulators in the development of improved treatment processes, effective disposal methods and appropriate regulatory tests for lime treatment sludges. This information may also be used to compare treatment operations and to forecast sludge related issues arising at treatment plants.

8.0 RECOMMENDATIONS

☐ It must be underlined that the leach protocol used should be applicable to the disposal scenario proposed. The Modified LEP conducted in this study estimated sludge stability for sludge pond type disposal environments. The mineral industry uses co-disposal of sludge with tailings in many sites. The stability of lime sludge in this type of disposal environment is complex and needs to be investigated further.

☐ Where AMD treatment sludges are to be disposed of on site, and will not come in contact with municipal waste in the long term, the leach test should involve the use of an inorganic versus organic leachant, such as in the Modified LEP.

☐ Based on the samples tested, this phase of work has underlined that while sludge stability is an issue, greater emphasis should be placed on the other major concern related to sludge disposal, volume reduction. In extreme situations, the cumulative volume of sludge produced over the long term may exceed the volume of stored tailings. Results indicate that even industry's most sophisticated lime treatment processes produce sludges containing only 20-35% solids. Further work is necessary to yield denser sludges than are currently observed. The effective containment and disposal of these sludges coupled with improvements in sludge densification are recommended for further study.

9.0 REFERENCES

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APPENDIX A: SITE QUESTIONNAIRE

in English

en français

AMD TREATMENT AND SLUDGE PRODUCTION QUESTIONNAIRE

BACKGROUND

1. Company Name: ____

2. Contact Persons:

Name	Title	Phone/Fax

3. Site Name: ____

4. Location: ____

5. Type of Operation: *(base metal, gold uranium, etc.)* ______________________

SITE DESCRIPTION

6. Facilities present: *(brief description)* ______________________________

____

7. Geographical/Geological features: *(brief description)* ______________________

____

8. Site History: ____

____

____

9. The mine is Producing **G**

Closed **G** What year? _____

ACID MINE DRAINAGE

10. Source of AMD: *(brief description)*

11. Average chemical composition of the AMD stream (s)

Parameter	Concentration (mg/L)	Parameter	
Al		Temperature (°C)	
As		Acidity (mg/L as $CaCO_3$)	
Ca		pH	
Cd		TSS (mg/L)	
Cr		Turbidity (NTU)	
Co		E_h (mv)	
Cu		Other parameters	
Fe_{tot}			
Hg			
Mn		Comments	
Ni			
Pb			
U			
Zn			
SO_4^{2-}			

TREATMENT PROCESS

12. Are other effluents, process waters treated with the AMD? YES**G** NO**G**

12a. Type of Effluent: ____________________

____

____

12b. Composition of effluent: ____________________

____

____

13. Type of treatment process *(lime precipitation, HDS/lime, lime/air, etc.)*: ____________________

____

14. Description of treatment process/facility (provide flowsheet if available): ______

____

____

____

15. Flowrate / capacity (m^3/min): ____________________

16. Reagents used *(lime, limestone, caustic, ferric sulphate, etc.)*: ____________________

____

____

17. Flocculant / polymer: ____________________

SLUDGE CHARACTERISTICS

18. Chemical composition of the sludge

Parameter	Concentration (wt. %)	Parameter	
Al		Temperature (°C)	
As		pH	
Ca		E_h	
Cd		Settling rate (m/h)	
Cr		Percent solids (%)	
Co		Other parameters	
C_{tot}			
Cu			
Fe_{tot}		Comments	
Hg			
Mn			
Ni			
Pb			
SiO_2			
SO_4^{2-}			
U			
Zn			

19. Sludge production:

Annual (t/a): ______________________________

Total (t): ______________________________

20. Disposal Scenario: ______________________________

____

____

____

21. Pond History: ______________________________

____

____

____

<u>GENERAL QUESTIONS ON SLUDGE PRODUCTION</u>

22. Describe any AMD treatment success that you feel are unique to your operation:

____

____

____

23. Describe any problems encountered in your operation: ______________________

24. What are the most significant problems in your operation that you would like to see resolved: ____

25. What are your immediate long term operating priorities?: ______________________

QUESTIONNAIRE SUR LE TRAITEMENT DU DMA
ET LA PRODUCTION DE BOUES

DONNÉES DE BASE

1. Nom de la société : ____

2. Personnes-ressources :

Nom	Titre	Téléphone/Télécopieur

3. Nom du site : ____

4. Lieu : ____

5. Type d'exploitation *(métaux communs, or, uranium, etc.)* : ____________________

DESCRIPTION DU SITE

6. Installations présentes : *(brève description)* ____________________

____

7. Éléments géographiques/géologiques : *(brève description)* ____________________

____

8. Historique du site : ____

____

____

9. La mine produit actuellement? G
est fermée G année de fermeture? _____

DRAINAGE MINIER ACIDE

10. Source du DMA *(brève description)* :

11. Composition chimique moyenne du DMA

Paramètre	Concentration (mg/L)	Paramètre	
Al		Température (°C)	
As		Acidité (mg/L $CaCO_3$)	
Ca		pH	
Cd		TSS (mg/L)	
Cr		Turbidité (N.T.U.)	
Co		E_h (mV)	
Cu		Autres paramètres	
Fe_{tot}			
Hg			
Mn		Remarques	
Ni			
Pb			
U			
Zn			
SO_4^{2-}			

PROCÉDÉ TRAITEMENT

12. Y a-t-il d'autres effluents ou de l'eau de traitement traités avec le DMA?

OUI **G** NON **G**

12a. Type d'effluent : ______________________________

____

____

12b. Composition de l'effluent : ______________________________

____

____

13. Type de procédé de traitement *(précipitation à la chaux, HDS/chaux, chaux/air, etc.)* :

____

____

14. Description du procédé ou de l'installation de traitement *(produire un schéma de traitement si possible)* : ______________________________

____

____

____

15. Débit/capacité (m^3/min) : ______________________________

16. Réactifs utilisés *(chaux, calcaire, soude caustique, sulphate ferrique, etc.)* : ______________

____

____

17. Floculant / polymère : ______________________________

CARACTÉRISTIQUES DES BOUES

18. Composition chimique des boues

Paramètre	Concentration (% poids)
Al	
As	
Ca	
Cd	
Cr	
Co	
C_{tot}	
Cu	
Fe_{tot}	
Hg	
Mn	
Ni	
Pb	
SiO_2	
SO_4^{2-}	
U	
Zn	

Paramètre	
Température (ºC)	
pH	
E_h	
Vitesse de sédimentation (m/h)	
% des solides	
Autres paramètres	
Remarques	

19. Production de boues :

Annuelle (t/a) : ______

Totale (t) : ______

20. Scénario d'évacuation : ______

______

______

______

21. Historique du bassin : ______

______

______

______

QUESTIONS GÉNÉRALES SUR LA PRODUCTION DE BOUES

22. Décrivez tout traitement réussi du DMA dans votre exploitation que vous considérez exceptionnel :

______

______

______

23. Décrivez les problèmes auxquels vous avez eu à faire face dans votre exploitation :

____

____

____

____

24. Quels sont les problèmes les plus significatifs que vous aimeriez voir résoudre dans votre exploitation? ______________________________

____

____

____

25. Quelles sont vos priorités à long terme en matière d'exploitation? ______________

____

____

____

APPENDIX B: SITE INFORMATION

(information compiled as given from site surveys)

Site: D-1

Background Information

Location:	Ontario
Operation:	Ni/Cu smelter, mining ceased in 1991, milling ceased in 1988, started operating in 1930, tailings impoundment from previous operations remains.

Acid Mine Drainage

AMD stream: tailings pond water (Dam #8)

Cu	0.1 mg/L
Fe_{tot}	1.6 mg/L
Ni	1.3 mg/L
SO_4	500 mg/L
pH	6.9
Flowrate	546 L/min

- AMD from Ni/Cu sulphide tailings
- problems with AMD include impact on groundwater and iron concentration in the final effluent
- most of the seepage is collected and treated by the effluent system.

Effluent Treatment Process

Effluent treated: smelter process water + collected AMD

Cu	5 mg/L
Fe_{tot}	1-10 mg/L
Ni	1-10 mg/L
SO_4	500 mg/L
pH	2.5
Flowrate	27,276 L/min (max)

Reagents: lime ($Ca(OH)_2$) precipitation and aeration. Lime slurry is added to mixing basin

and discharged to the settling area.
Flocculant: none

Sludge Production and Disposal

Annual production: ~ 4,050 m^3

Total production (17 years, summer 1995): 68,861 m^3

Sludge characteristics:

Fe	4.24 %
Ni	0.84 %
Cu	0.31 %
pH	7.7
Suspended: Solids	17.6
Dissolved: Solids	822

Mineralogy: Calcite, minor Quartz

Percent solids: ~ 7 %

Density: 0.96 g/cm^3

Long term options:
(1) recycle sludge to smelter
(2) ship the sludge to a sister site
(3) place the sludge in the polishing pond
(4) place the sludge in the tailings pond

- Smelting proved unsuitable because of the low grade of the sludge as well as its high water content. The economic benefits of the metal recovery is insignificant compared to the cost of preparation and smelting.

- It was recommended that the sludge be added to the polishing pond, thus reducing the lime requirement and in the long term producing a denser sludge that can be recycled to the smelter.

Site: Q-2

Background

Location: Manitoba

Operation: Base Metal (Cu, Zn)
- operations started 1929, open pit mining 1938, underground to present
- facilities present are a concentrator, zinc refinery and copper smelter

Acid Mine Drainage

AMD stream:
- tailing disposal area run-off and seepage
- process discharges
- residue pond decant

Composition of AMD stream

Cd	< 0.01 mg/L
Cu	0.001 - 0.005 mg/L
Fe_{tot}	0.05 - 0.5 mg/L
Mn	0.01 - 0.7 mg/L
Ni	< 0.01 mg/L
Pb	<0.04 mg/L
Zn	0.50 - 3.00 mg/L
TSS	2 - 15 mg/L
Acidity	20 - 50 mg/L (as $CaCO_3$)
pH	7.0 - 10.80

Treatment Process

Effluent treated:
- tailings and process waters from the Zn plant
- mill concentrate
- powerhouse cooling water and smelter and site runoff

- all effluents are treated with lime prior to discharge
- the treatment facility is a settling system with a finishing pond (clarification pond) as final treatment
- lime is added at the influent to clarification pond during open water months

Reagent: lime

Flocculant: used with system upsets

Sludge Production and Disposal

Total sludge production: approximately 7600 t

- free settling disposal

Chemical composition of the sludge (by weight)

Al	1.30%
Cu	0.05%
Fe_{tot}	3.00%
Ni	0.009%
SiO_2	5.8%
SO_4^{2-}	2.60%
Zn	7.60%
S^-	1.15%
Cl	2.2
F	0.011 to 0.044%

- final discharge pH has been maintained at 10.00 (+/-0.5) since 1976

- the problems encountered with this operation are seasonal influences and process upsets.
- the immediate long term operating priorities are to comply with MMLER.

Site: M-3

Background Information:

Location: Ontario

Operation: Base Metal (Cu-Ni)

- treatment plant consisting of two 130' diameter reactors
- plant treats excess runoff from tailings area, smelter area & townsite
- built in 1975
- lime precipitation in reactor clarifier

Acid Mine Drainage

- source of AMD tailings

Treatment Process

- AMD from tailings, smelter water and watershed, sewage treatment water
- lime precipitation in reactor clarifier to pH 10.5

Flowrate: 40 million m^3/year, 272,000 m^3/day

Reagents: slaked lime, Percol 338

Average chemical composition of the total effluent (1994) .

Parameter	Concentration (mg/L)	Parameter	
Al	3.46	Temperature (ºC)	
As	0.053	Acidity (mg/L as $CaCO_3$)	86.2
Ca	364	pH	3.10
Cd	0.0031	TSS (mg/L)	10
Cr	0.0124	Turbidity (NTU)	
Co	0.263	Other parameters TDS NH_3 COD Phenol Oil CN B Cr In K Li Mg Na P Sb Si Zr	 2453 mg/L 7.71 3.8 <0.02 <0.4 0.012 0.105 0.0124 0.0338 35.4 0.548 92.7 122 0.0829 0.0125 6.27 0.0721
Cu	1.02		
Fe_{tot}	33.6		
Hg	<0.0008		
Mg	92.7		
Ni	11.3		
Pb	<0.0150		
Zn	0.188		
SO_4^{2-}	1675	Comments	

Sludge Production and Disposal

Annual production: 1000 Usgpm @ 1% solids
Total production: 100,000 to 150,000 tonnes

Sludge Characteristics:

0.5% solids, Ca (8-15%), Fe (10-14%), Ni (2.5-6%), Cu (0.1-0.2%), Mg (4-6%), S (1.8-2%), C (2-4.5%)

Disposal:

- codisposal with tailings

Difficulties encountered:

- very large watershed, 10,000 acres

Successes

- manage water input-rate as much as possible using control structures, values, etc
- ensure sufficient sludge pumping capacity
- flexible flocculant addition rate - add up to 1 ppm during periods of "light floc formation"

Site: R-4

Background Information:

Location: Québec

Operation: copper (massive sulphide*)*
- the mine has been closed since 1962

Acid Mine Drainage

AMD stream: 5,000,000 tonnes of sulfide tailings

Chemical composition of AMD

Cd	0.016 mg/L
Cu	1.0 mg/L
Fe_{tot}	75 mg/L
Mn	4.0 mg/L
Pb	< 0.05 mg/L
Zn	5.3 mg/L
SO_4^{2-}	950 mg/L
Acidity	210 - 450 mg/L (in $CaCO_3$)
TSS	10 mg/L
pH	2.88

Treatment Process

Effluent treated:	- effluents coming partly from sulphide tailings as well as water basin environments
Treatment process:	- HDS / lime - flocculant / air
Flowrate:	6 m^3/min
Reagents used:	hydrated lime
Flocculant:	Percol 90L

Sludge Production and Disposal

Annual sludge production: 2,500 m^3

Chemical composition of the sludge (by weight)

Ca	18%
Cu	1.8%
Fe_{tot}	11.7%
SO_4^{2-}	7.4%
Zn	1.4%
Solids	10-15%

- the problems encountered with this operation are difficulties with principle drainage ditch, specifically holding acid water. The immediate long term goals of this operation are to maintain environmental norms with the lowest costs.

Site: F-5

Background

Location:	New Brunswick
Operation:	Coal (Cut and fill surface mine)

Site Description

- lime neutralization facility, sludge storage ponds
- site is on plateau
- lithology is grey/green
- fine to course grained sandstone with minor conglomerate and shale
- area mined between 1982 and 1986 when potential of acid generating mine was recognized
- mined depth of 8 to 20 m
- the mine is now closed

Acid Mine Drainage

AMD stream: sandstone overburden with pyrite nodules with no source of alkalinity in the waste rock overburden

Chemical composition of AMD

Al	126 mg/L (Sept 92 - Oct 95)
Fe_{tot}	40 mg/L (Sept 92 - Oct 95)
SO_4^{2-}	1850 mg/L (Sept 92 - Oct 95)
Acidity	800 mg $CaCO_3$/L (average)
TSS	LT5 - 50 (25 average)
pH	2.8 - 3.5 (1991-1995)
Turbidity	25 NTU

- started placing sludge on waste rock in 1982
- acidity has dropped from 1500 mg/L to approximately 800 mg/L
- the pH has risen from 2.8 to 3.2-3.7 range
- a significant change in the metals concentration has not been identified
- sulphate has decreased from 2100 mg/L to an average of 1600 mg/L in 1995

Treatment Process

Effluent treated: AMD
Treatment process: hydrated lime slurried and injected into suction of pumps
Flowrate: 11 m^3/min
Reagents: hydrated lime (approx. 2000 tonnes/a)

Sludge Production and Disposal

Annual production: 30,000 m^3/a
Total production: 300000 m^3

Disposal Scenario: -currently 10 active storage ponds
-each year, 3 to 6 ponds are dredged on the acid generating waste rock

Pond history: ponds 90-1, 90-2, 89-3, 89-1 and 88-1 were all dredged in 1995

- the sludge deposition concept of placing it on the waste rock is unique to this operation. Studies have indicated however, that nearly 90% of the sludge goes below the surface of the waste rock. The only impact of mine water quality to date has been increased mine water pH, decreased SO_4 and lime consumption.

- the problems encountered in this operation are the cost of dredging, and the inability to retain larger % of sludge on surface of waste rock. The problem that must be resolved is the automation of the neutralization facility and a place to put the sludge safely to provide a walk away reclamation.

- the immediate long term operating priorities are:
    1. Determine if sludge will dissolve in acidic conditions of the waste rock.
    2. Determine if sludge or what is the cause of increased pH, decreased acidity in mine water.
    3. Design sludge deposition plan (if #1 proves negative).
    4. Automate facility.

Site: S-6

Background Information:

Location: Ontario

Operation: Base metal, mining and milling since 1965 and 1967 respectively, copper smelter/refinery, zinc roaster, purification, leach plant and cell house.

Acid Mine Drainage

AMD stream: 3000 acre (1200 Ha) tailings area

Composition of AMD stream (mg/L)

Al	N/A
As	0.09-<0.050
Ca	200-400
Cd	N/A
Cr	N/A
Co	<0.020
Cu	2-<0.010
Fe_t	1-20
Hg	<0.001
Mn	<0.010
Ni	0.05-<dl
Pb	<0.020
U	N/A
Zn	0.05-120
SO_4^{2-}	1200-2000
pH	3.5-7.5
TSS	15-<1
Na	60-100
Cl	30-60

- effluent is seasonably variable, Zn values vary with mill discharge mainly.

Flowrate: 6.94 m^3/min

Effluent Treatment Process

- process water and storm water from ditch #10 are also treated with the AMD

Composition of process/storm water (mg/L)

Cu	0.4-3
Fe_t	1-10
Pb	0.1-1.2
Zn	1-40
SO_4^{2-}	5000-1200
pH	6-8

Reagent : Lime, stations #1 and #2 located in northwest and southwest corners of the ponds.
- lime is slaked at the concentrator and trucked to the lime stations
- the slaked lime is gravity feed to the stream

Flocculant: none

Sludge Production and Disposal

Annual production: 3.5-4.0 x 10^6 m^3

Total production: N/A

Sludge Characteristics

% solids 1-2 %

- little information available
  - Zn 7-10%
  - mostly comprised of hydrated silica

Disposal:
- impounded in tailings discharge pond where volume is reduced through freeze-thaw. See attached diagram.
- sludge is impounded in the sludge pond then dredged out and placed with the tailings
- pond was constructed in early 70s to accommodate enlargement of tailings area.
- in 1992, the pond was divided into two cells to improve efficiency
- volume of sludge is dramatically reduced through freeze-thaw
- estimated sludge volume in the pond was 1,081,170 m^3 at time of sampling however this is a low end estimated at the sludge volume in the pond could be 2-3 times greater

- desludging program is scheduled for late spring 1996

Difficulties encountered:

- filling of ponds by low density sludge results in expensive desludging programs and if the sludge volume produced is excessive, then poor effluent quality may result.

Operating priorities:

- treatment of effluent at earlier stage (thickener), increases in density of sludge (at thickener)

Site: J-7

Location:	Québec
Operation:	gold

Site Information

- mineral treatment factory
- operations started in 1980, open air mine up to 1989
- underground mine since 1989

Acid Mine Drainage

AMD stream: north and south waste rock dumps, sericitic schist

Chemical Composition of AMD

Fe_{tot}	774 mg/L
SO_4^{2-}	4516 mg/L
Acidity	3349 mg/L (in $CaCO_3$)
Conductivity	4997 µhos

Treatment Procedure

Effluent treated: water draining from the mine

Composition of effluent

conductivity	2552 µhos
Fe_{tot}	144 mg/L
Acidity	102 mg/L (of CaCO3)
SO_4^{2-}	1431 mg/L

Treatment process - HDS / lime

Flowrate: max 19 m^3/min
min 5.8 m^3/min

Flocculant: Percol 338

Sludge Production and Disposal

Chemical composition of sludge

Al	23900 µg/g
As	<50 µg/g
Ca	171000 µg/g
Cr	<50 µg/g
Co	90 µg/g
Cu	320 µg/g
Fe_{tot}	93100 µg/g
Mn	1530 µg/g
Ni	50 µg/g
Pb	<50 µg/g
Zn	130 µg/g
Mg	13700 µg/g

Pond history: In operation since 1991

- the most significant need of the mine is a study on the stability of the aged sludge and methods of restoration of the basins.

Site: W-8

Background Information

Location: New Brunswick

Operation: Base Metal; open in 1965 with continuous production

Acid Mine Drainage

AMD stream: underground mine drainage, tailings, waste rock, contaminated roadways

Composition of AMD stream

Cu	5 mg/L
Fe_{tot}	150 mg/L
Pb	3 mg/L
Zn	100 mg/L
SO_4^{2-}	3500 mg/L
Thiosalts	200mg/L
TSS	10 mg/L

pH 4.5

- 10,500 t/day underground mine mill complex
- there are no other effluents or process waters treated with the AMD
- massive sulphide ore body

Treatment Process

Effluent treated: AMD

Treatment Process HDS/lime

Reagents: Lime and air

Flocculant: Percol 727
Flowrate: 60 m^3/min

Sludge Production

Annual Production: 12,000 t
Disposal: Codeposit with mill tailings
- there is periodic turbidity; out of compliance with Zn_{tot}

Site: N-9

Background Information:

Location: Ontario

Operation: Uranium mining

Site Description

- flooded tailings basin, containment dams, effluent treatment plant (lime, $BaCl_2$), 2 limed settling ponds
- site sits on the Canadian Shield, tailings in natural bedrock basins, thin surficial deposits
- mine operated 1958-1961, tailings placed in two lakes; mine reactivated in 1979 to close in August 1990.
- current treatment system in place from 1979 to date.

Acid Mine Drainage

AMD Stream: source of AMD is bleached uranium tailings containing 5-7% pyrite, very minor amounts waste rock.

Treatment Process

- AMD stream is combined with treated mill effluent (pH 8-9, TDS = 4,500 mg/L, SO_4 = 2,300 mg/L Ca = 590 mg/L, $Ra_{226(diss)}$ = 30Bq/L) prior to treatment

- simple lime, $BaCl_2$ precipitation
- effluent enters first of two agitated tanks in series (gravity flow). Lime is added to the first tank, pH measure at outlet of second tank. Treated water flows through two hypalon lined settling ponds.

Flowrate: Max=11 m^3/min; 3 year Avg. = 7.8 m^3/min
Flocculant: none
Reagents: lime slurry, $BaCl_2$, no flocculant

Average chemical composition of the AMD stream (s) (1987-1989)

Parameter	Concentration (mg/L)	Parameter	
Al		Temperature (ºC)	0.5 to 22
As		Acidity (mg/L as $CaCO_3$)	50
Ca	460	pH	4.5
Cd		TSS (mg/L)	3
Cr		Turbidity (NTU)	
Co	0.075	E_h (mv)	
Cu	1.60	Other parameters	
Fe_{tot}	6.0	TDS	2650 mg/L
Hg		Ra_{226} total	6.5 Bq/L
Mn	1.6	Comments	Average data over 3 year period, 1987, 1988, and 1989 when sludge produced
Ni	0.07		
Pb	0.08		
U	0.30		
Zn	0.5		
SO_4^{2-}	1580		

Present chemical composition of the AMD stream (s) (Feb. 1996)

Parameter	Concentration (mg/L)	Parameter	
Al		Temperature (°C)	0.5
As		Acidity (mg/L as $CaCO_3$)	7
Ca	200	pH	5.7
Cd		TSS (mg/L)	
Cr		Turbidity (NTU)	
Co		E_h (mv)	
Cu		Other parameters	
Fe_{tot}		TDS	823 mg/L
Hg		Ra_{226} total	1.13 Bq/L
Mn		Comments	Parameters analyzed in 1995 & 1996 reduced due to near neutral conditions of untreated water
Ni			
Pb			
U			
Zn			
SO_4^{2-}	500		

- no other effluent is treated with this AMD stream

Sludge Production and Disposal

Annual production: N/A

Total production: N/A

Sludge Characteristics: No information reported

Disposal:

- all the sludge produced to date has been deposited in the sludge pond. Final disposal will be into the tailings basins then flooded with water.

- settling pond #1 is full: presently depositing sludge in settling pond #2

- since 1990, treatment plant and system shut down often due to low flow to treat.

- sludge in pond #1 allowed to dry out and/or freeze resulting in sludge consolidation (dewatering)

- tailings basin completely covered with water late 1994. No acid generation of significance since that time

Difficulties encountered:

- settling pond had baffles made of hypalon. Sludge accumulation on up-stream side of baffles pulled the baffles down allowing for short circuiting over top of baffles.

Long Term priorities:

- minimization of the rate of acid generation by flooding with water as quickly as possible after deposition

Site: B-10

Background Information:

Location: Ontario

Operation: Base Metal (Cu-Ni)

Acid Mine Drainage

- source of AMD tailings

Treatment Process

- AMD from tailing, smelter water and watershed, sewage treatment water
- lime precipitation in reactor clarifier to pH 10.5

Flowrate: 4 million m^3/year

Reagents: slaked lime, Percol 338

Sludge Production and Disposal

Annual production: N/A
Total production: N/A

Disposal:

- sludge is deposited in a pond with sulphide thickener overflows. Pond dredged every 2-3 years and sludge is stock piled.

Difficulties encountered:

- very large watershed, 10,000 acres

Site: T-11

Background Information:

Location: Ontario

Operation: Uranium mining

Site Description

- elevated tailings, effluent treatment plant (lime, $BaCl_2$), sludge settling area in lake
- site sits on the Canadian Shield, exposed bedrock, tailings sitting on glacial/alluvial sand/gravel deposit, thin surficial deposits
- mine operated 1956-1968, 1970/71 run-off regrading/rerouting, effluent treatment plant installed, sludge settling in lake commenced.

Acid Mine Drainage

- source of AMD is elevated uranium tailings containing 5-7% pyrite.

Treatment Process

- only AMD stream is treated at this site

- simple lime, $BaCl_2$ precipitation

- lime slurry is metered into seepage/run-off collection stream that drains through mixing flume into lake for settling.

- bubbler system installed in lake to prevent short circuiting

- lime slurry holding tank and pumps (no pH control)

Flowrate: No limit, treat run-off water as it comes, 1995 Avg. = 4 m^3/min

Reagents: lime slurry, $BaCl_2$, no flocculant

Average chemical composition of the AMD stream (s)

Parameter	Concentration (mg/L)	Parameter	
Al		Temperature (ºC)	
As		Acidity (mg/L as $CaCO_3$)	1015
Ca		pH	3.8
Cd		TSS (mg/L)	
Cr		Turbidity (NTU)	
Co	0.21	E_h (mv)	
Cu	0.05	Other parameters TDS Ra_{226} total	 2550 mg/L 0.2 Bq/L
Fe_{tot}	487 (Fe^{2+})		
Hg			
Mn	2.8	Comments	
Ni	0.07		
Pb	< 0.10		
U	0.052		
Zn	0.57		
SO_4^{2-}			

Sludge Production and Disposal

Annual production: N/A

Total production: N/A

Sludge Characteristics: No information reported

Disposal:

- located in permanent location inn the bottom of lake
- sludge deposited in lake in 70/71

Difficulties encountered:

- short circuiting in the winter period from treated water/sludge inlet to outlet. (This problem has not occurred in late winter 1994/1995 and 1995/1996.
- sludge consolidation

Long Term priorities:

- sludge volume and relocation within settling areas

APPENDIX C: SLUDGE SAMPLING TECHNIQUES

CONTENTS

LIST OF TABLES

LIST OF FIGURES

Note: See Section 9.0 “References” in the main body of the report for details of the references used in this appendix.

Information contained in this guide has been compiled, for the most part, from sampling manuals developed for other materials, such as sediments, tailings and waste rock.

C.1 The Sampling Plan

It is important to base the sampling plan on the project objectives (Senes 1994b,c). Extensive and thorough planning is a necessary and important step for the successful completion of a sludge sampling program (Senes 1994b,c). Characterization data is meaningless if samples are collected at inappropriate locations or do not represent the study area. Furthermore, the proper selection and use of sampling equipment, sample handling, storage and transport are all equally as important as the selection of the sampling locations. According to Mudroch and Azcue (1995), sixty percent of the time allocated to sampling should be spent on detailed planning to ensure that project objectives are met. The plan should include the selection of the number and location of sampling stations. The methods used for sludge sampling, handling, preservation, storage and transport of collected samples should be described. Logistics such as shipping equipment to the site, travel of personnel, weather and road conditions must also be considered.

C.1.1 Selecting sampling stations

The selection of sampling stations depends on the nature, dimensions and shape of the sludge pond. In a lake/pond, preliminary sampling stations should be located on a transect down the long axis of the lake (Figure C-1). The minimum number of samples will depend on the bottom morphology and variance between sludge composition within the pond (from previous sampling events). Regularly shaped ponds will require fewer transects than irregularly shaped ponds. Near a point source, sampling stations can be located on a ray transect (Figure C-1). The spacing of sampling stations along transects depends on available funds and time. After the preliminary samples are taken, additional samples can be taken to provide more information if necessary or desired. A description of the sampling procedure, field observations of the sample and water depth information should be taken at each site.

C.1.2 Required number of sampling stations

The first step in determining the number of sampling stations should be an estimation of variability and the mean concentration of the contaminants and metals of interest in the sludge. A statistical method is then used to calculate the number of samples needed to achieve the acceptable confidence levels and to meet the objectives of the sampling program. Knowledge of the historical background of the sampling site, and experience are also very useful in selecting the number and locations of the sampling stations. Mudrock and Azcue (1995) derived the following equation to calculate the number of sampling stations required:

$$n = \frac{t^2 s^2}{D^2}$$

n is the number of sampling stations required.

t is a number chosen from a “t” table for a chosen level of precision.

s^2 is the variance, known from previous studies, $s^2 = (R/4)^2$, where R is the estimated range of concentration likely to be encountered from sampling.

D is the acceptable variability, expressed as mean concentration of contaminant.

Figure C-1: Design of a preliminary sampling plans for different disposal bodies (Mudroch and Azcue 1995).

C.1.3 Positioning sampling stations

To select the sampling stations, background information on the sampling site must first be collected. If possible, navigational charts or maps of the site/pond should be acquired as well as any chemical or characterization data available for the sludge to be sampled. To position the stations, first start by marking the position of the planned sampling stations on the chart or map. Accurate positioning of sampling stations is especially important in programs where the sampling is to be repeated. A positioning technique must be selected before sampling commences. A distance line or taut wire can be used for direct positioning of the sampling stations within a small sampling area or close to the shore. The number of samples to be collected depends on the sampling area and the volume of sludge in the pond (Table C-1). Most of the site ponds sampled for this project were of small to average size, while a few ponds were large.

Table C-1: Number of Samples to be Collected as a Function of Sludge Volume.

Sludge Volume in Pond (m^3)	Number of Samples to be Collected
Very small (<10,000)	3-6[1]
Small (10,001 - 50,000)	7-12
Average (50,001 - 100,000)	13-18
Large (100,001 to 1,000,000)	19-40
Very Large (>1,000,001)	41 + (volume -1,000,000)/75,000[2]

[1] It is assumed that six samples are sufficient to obtain an acceptable level of confidence for a project of 10,000 m^3 (Ocean Chem Sciences Ltd. 1984).

[2] Atkinson, 1985

C.1.4 Example of a sampling protocol

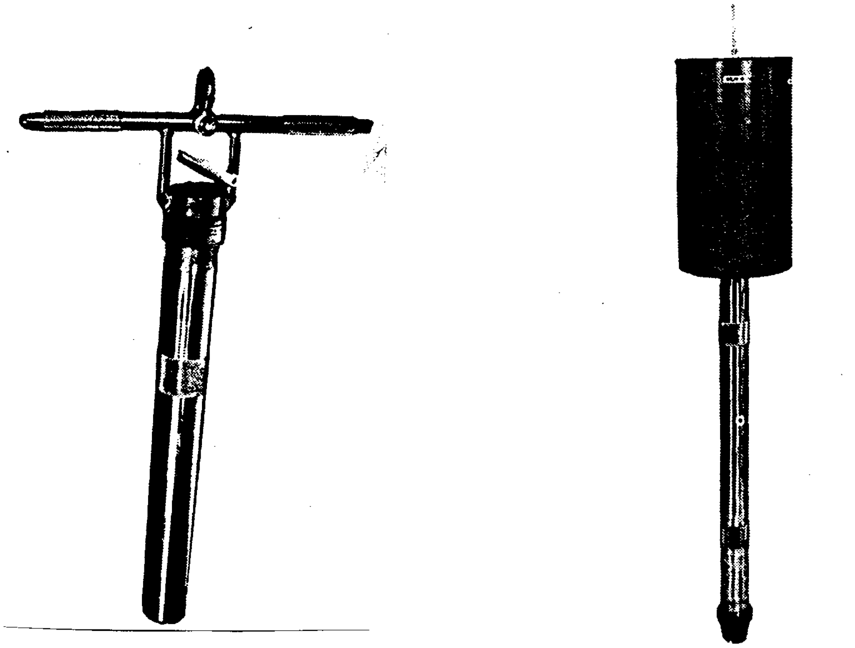
The sampling protocal should be prepared prior to selection of the sampling stations. An example of a protocol is given below (modified from Mudroch and Azcue, 1995).

1. Define the character of the samples to be collected to meet the objectives of the sampling program and the study objectives.
2. Confirm available funds and number and availability of trained and nontrained personnel needed for the sampling program.
3. List all physical, mineralogical, and chemical analyses that will be carried out on sludge samples in the laboratory as well as observations and tests that will be carried out in the field.
4. Estimate and compile the quantity (i.e., volume, weight) of wet and dry samples necessary to carry out all listed analyses and assays.
5. Collect information on various parameters in the study area relevant to the sampling program, such as water depth, morphometry (shape) of the pond, hydrological conditions, size distribution, flow patterns within the pond, depositional history of the pond, sludge age, climatic conditions, etc.
6. Plot the sampling stations on a chart containing the study area. Number the sampling stations in the most logical sequence relevant to the sampling program and objectives.
7. Select the time frame of the sampling program. Consider the optimal use of the time spent on the sludge sampling in the study area.
8. Consider the safety of the personnel carrying out the sampling program, such as weather conditions expected during the sampling period (wind speed and direction, air and water temperature, ice depth) and severity of contamination of the sludge and water to which the sampling personnel will be exposed.
9. Select and list all sampling equipment and other materials that will be used in support of the sampling program in the study area, such as tools and spare parts for emergency repairs and maintenance of the sampling equipment in the field; maps; charts; note books, logging sheets; equipment for measuring sludge properties in the field, such as pH and Eh meters, etc.; equipment for homogenization; sample containers; extruders for cores; storage boxes, ice auger, boat, personnel safety materials and other equipment specific to the sampling program.
10. List the last date the sampling equipment was tested together with any problems encountered during the testing and repairs of the equipment. List all necessary spare parts and tools that must accompany the sampling equipment for emergency repairs during the sampling program.
11. Select and compile appropriate sludge sampling and subsampling procedures, sample handling, sample preservation, field storage, transport from the site to the laboratory, and storage after samples delivery including required temperature, freezing of the samples. etc.

C.1.5 Sampling equipment

Several different sampling devices are commercially available for grab and core sampling. Due to the fluid nature of the sludges, sampling equipment must be chosen to eliminate the possibility of sludge loss during sampling. An inert container (as discussed below) is a suitable device for both the collection and storage of a grab sample. Commercially available corers can be used to collect core samples of sludge deposited in a pond/lake environment. The hand corer shown in Figure C-2 is appropriate for collecting cores in shallow water environments or when an ice cover is present over the pond. With an extension handle the corer can reach depths in excess of 5 m. The second type of corer for sludge sampling is the Ballchek corer (Figure C-2). This type of corer is designed for deep water environments where a hand corer is not practical.

The Ballchek corer has stabilizing fins which offer high strength and low drag characteristics. A practical feature of the Ballchek sampler is a simple positive sealing mechanism which acts as a check valve to retain the sludge in the core tube during raising operations.



Hand Corer

Ballchek Corer

Figure C-2: Core samplers used in sludge sampling; a) hand corer b) Ballchek corer.

C.2 Handling, Preservation Techniques, and Storage of Sludge Samples

C.2.1 Containers for sludge samples

Careful planning and selection of containers and utensils involved in sludge handling prior to analysis is extremely important because they can be a significant source of contamination. Containers should neither contaminate the sample, nor promote loss of parameters of interest through adsorption onto walls, etc. Table C-2 summarizes the recommended sample containers for collecting sediments samples prior to determining different parameters. These recommendations can be applied to sludge samples as they face similar problems for storage and handling. Generally, containers used for drying should be made of material resistant to corrosion and not subject to change in weight or to disintegration on repeated heating and cooling. The selection of the material depends on subsequent analyses of sludge samples. Prior to sampling, all containers should be properly labelled with a waterproof marker with the following information: site and sample identification; date and time of collection; sludge use; preservative used (if any); name of collector.

Table C-2: Sampling Containers and Preservation Methods for Different Parameters Measured in Sediments (Mudroch and Azcue 1995)

Parameter	Containers	Preservation	Maximum Storage	Comments
Particle size	P, G, or M	Wet, 4°C, tightly sealed	14 days	Drying, freezing and thawing cause aggregation of particles
pH, E_h	Bucket or core	Wet, undisturbed and untreated	Determined in the field	Very difficult and problematic temperature corrections
Metals	P or T	Dry (60°C), freeze (-20°C) or freeze dry	6 months	If samples are not analyzed within 48 hours, freeze dried -20°C up to 6 months
Mercury	G or T	Freeze, -20°C	1 month	Mercury analysis is performed with wet samples

P: Polyethylene; G: Glass; M: Metal; T: Teflon

C.2.2 Handling samples for testing and analysis of wet samples

Particle Size Distribution

Particle size distribution analyses should be carried out on wet sludges. Samples for the analyses should be stored at 4°C and never frozen. Sludges with a high iron content should be stored in airtight containers, under inert gas, to avoid oxidation of ferrous iron to ferric iron and should be analyzed as soon as possible after collection. Drying, freezing and thawing of the sludges can cause irreversible aggregation of particles and should be avoided.

Freezing

Freezing is primarily used when organic analyses are required. Deep freezing (-80°C) can best maintain sample integrity thus enabling the investigation for contaminant concentrations.

C.2.3 Handling samples for testing and analysis of dry samples

Drying

Three types of drying are commonly used to prepare solid samples prior to analysis: air-drying, oven-drying, and freeze-drying. Freeze-drying was the drying technique selected for this study as it best maintained the integrity of the samples for further characterization.

i) air-drying

Although time consuming (3-5 days), air-drying is commonly used in soil science (McKeague 1978) and in sedimentology (Folk 1974). Air-drying may generate undesirable changes in sludge properties and increases the risks of contamination. Air-drying has been used in some studies to avoid the loss of components, such as mercury, that are volatile at temperatures greater than 50-60°C (Forstner and Salomons 1980).

It is difficult to achieve thorough drying to constant weight as specified by the ASTM method D421-58 (ASTM 1969). Depending on the quantity of material, air-drying is carried out in a fume hood (small samples), an air-drying cabinet with air circulation, or in sheltered ventilated rooms (large samples). However, because of the possibility of air contamination of samples by dust, air-drying is not recommended for the accurate determination of inorganic and organic constituents. For chemical analyses, where preservation is required in addition to the drying, this method is not suitable because microbial degradation, oxidation, and other processes that can alter the sample are not halted (Mudroch and Azcue 1995). In this study, chemical analysis of wet samples was found to be unreliable in terms of reproducibility.

ii) oven-drying

Oven-drying of sludges is usually carried out on samples collected for determining inorganic components, such as major and trace elements. However, oven-drying is not suitable for particle size determination, since wet fine-grained particles become hard-to-break aggregates (Folk, 1974) and mineral transformations are more likely to occur, particularly with iron compounds. Oven-drying is not acceptable for sludges containing any volatile or oxidizable components, whether they are organic or inorganic (Luepke 1979a), and may contribute to the alteration of even nonvolatile organics.

Lower heating temperatures (less than 60ºC) are essential when preparing the sludge for the determination of volatile trace elements, such as mercury (Luepke 1979b). The effects of various drying temperatures on the determination of mercury in sludges were reviewed by de Groot and Zschuppe (1981). They concluded that mercury should be determined in sludges that have preferably been either air-dried or oven-dried at 40ºC.

iii) freeze-drying

In the freeze-drying process, water in the frozen or solid state is sublimated and removed from the material as a vapour. Freeze-drying (also called lyophilization) can be used for drying sediments and sludges collected for the determination of most organic components as well as for analyses of inorganic components, such as major and trace elements. The principal advantages of freeze-drying are:

- the low temperatures avoid chemical changes in labile components;
- the loss of volatile constituents is minimized (Bourbonniere et al. 1986);
- most particles of dried sludges remain dispersed;
- the aggregation of the particles is minimized;
- sterility is maintained;
- oxidation of various inorganic or organic compounds is minimized or eliminated.

C.2.4 Visual analysis of sludge cores

A visual description of each core sampled should contain the following information:

- Length of retained core.
- Equipment used for core collection.
- Name of the operator who collected, handled, and split (or extruded) the core.
- Description of splitting (or extruding) of the core.
- Thickness of the sludge units in the core
- Consistency, for example, described as soupy, soft, firm, stiff, loose, packed, etc.
- Structure (if any).
- Presence of foreign matter.

C.2.5 Mixing and Homogenization

The degree of success attained in sample homogenization and splitting is largely responsible for the variability in analytical results, aside from the the original sample collection. Homogenization of sludge cores can be carried out by the following simple operations:

- Quartering;
- Turning the sample over and over with a spatula;
- Using mechanical rotating mixers.

The second and third operations were used to homogenize the samples in this study.

C.2.6 Preservation of samples

Preservation techniques are usually intended to retard microbial degradation, oxidation, and/or loss of volatile components.

Temperature is an important factor that can affect the collected samples from the time of sample recovery through handling and processing to the final analyses. Sludge samples intended for analyses or experiments after drying can be stored in containers, cans, plastic bags, etc., at ambient or room temperature. However, sludges collected for determining organic contaminants and mercury should be stored in a refrigerator (about $4^{o}C$). The other important parameter is storage time. Determination of certain physico-chemical parameters, such as pH, E_h, temperature, and cation exchange capacity, should be made in the field immediately after sample collection, when possible.

C.2.7 Storage of sludge samples

The storage conditions of sludge samples depend on practical aspects and limitations as well as prior knowledge of the stability of known or expected contaminants in the samples. Preservation and storage are two aspects of sample handling that go hand-in-hand. Recommended storage times according to parameters to be measured are given in Table C-2.

C.2.8 Documentation of sample collection

Documentation of collection and analysis of environmental samples requires all the information necessary to: trace a sample from the field to the final result of analysis; describe the sampling and analytical methodology; and describe the QA/QC program (Keith et al. 1982).

Correct and complete field notes are necessary in any sampling program. Poor or incomplete documentation of sample collection can make analytical results impossible to interpret. The following items should be recorded at the time of sludge sampling:

1. Project name and number.
2. Name of sampling site and sample number.
3. Time and date of sample collection.
4. Weather conditions, particularly wind strength and direction, air and water temperature, snow or ice cover, thickness of ice when sampling from the ice.
5. Positioning information (positioning tools, any problems encountered during positioning of a station, drawings of sampling site's positions on a chart).
6. Type of vessel used (generally for summer samples).
7. Type of sampler used (grab, corer) and modifications made to the sampler during sampling.
8. Names of sampling personnel.
9. Notes of unusual events that occurred during sampling
10. Sludge description including texture, consistency, colour, odour, core appearance.

11. Notes on further processing of samples in the field, particularly subsampling methods, type of containers and temperature used for sample storage, and record of any measurements made in the field, such as pH, E_h.

APPENDIX D: CORE DESCRIPTIONS

Site D-1
Collection Date: December 5, 1995
No. of cores: 9

Core No.	Description (colour, layering, etc.)
D1-1-012-95	hydrogen sulfide from slag, rusty brown, some black slag, unreacted lime
D1-1-010-95	watery, rusty brown, little or no slag, granular (undissolved lime?)
D1-1-016-95	watery, rust brown, some slag and unreacted lime particles
D1-1-017-95	rusty brown, lighter brown colour, some larger particles present
D1-1-011-95	dark brown material, thicker, lighter sludge, mostly rusty brown
D1-1-015-95	rusty brown, unreacted lime
D1-1-014-95	a lot of slag, most of core contaminated with slag...therefore discarded
D1-1-018-95	sample dried out, higher percent solids
D1-1-013-95	sample highly contaminated with slag, discarded

Site Q-2
Collection Date: March 26, 1996 No. of cores 37

Core No.	Description (colour, layering, etc.)
Q2-1-001-96	greenish brown colour, some gray
Q2-1-002-96	greenish brown colour, some unreacted lime
Q2-1-003-96	greenish brown evidence of large grainy particles
Q2-1-004-96	greenish brown
Q2-1-005-96	brown sludge, some unreacted lime, tailings
Q2-1-006-96	brown sludge, rusty brown
Q2-1-007-96	greyish brown colour mixed with some tailings
Q2-1-008-96	brown layers, some grey layers, some tailings at the bottom
Q2-1-009-96	reddish brown sludge with some tailings at the bottom
Q2-1-010-96	dark brown sludge mixed with some tailings
Q2-1-011-96	brown sludge mixed with tailings
Q2-1-012-96	mostly tailings, too difficult to separate
Q2-1-013-96	brown sludge with some tailings at bottom
Q2-1-014-96	dark brown sludge with some tailings at bottom
Q2-1-015-96	appears to be all tailings
Q2-1-016-96	brown sludge
Q2-1-017-96	reddish brown sludge, tailings at bottom, patches of red
Q2-1-018-96	mainly tailings...discarded
Q2-1-019-96	mainly tailings, some areas of reddish brown/red
Q2-1-020-96	mainly tailings, some areas of lime/red
Q2-1-021-96	reddish brown colour, some tailing along side
Q2-1-022-96	mainly reddish brown
Q2-1-023-96	mainly reddish-brown
Q2-1-024-96	reddish brown with some tailings
Q2-1-025-96	mainly tailings, discarded
Q2-1-027-96	reddish brown sludge with tailings along side
Q2-1-028-96	mainly tailings, some patches of reddish brown sludge
Q2-1-029-96	mainly tailings, discarded
Q2-1-030-96	red-brownish sludge, tailings on side
Q2-1-031-96	mainly tailings, some patches of reddish brown sludge
Q2-1-032-96	approximately 10 mL of light brown sludge
Q2-1-033-96	approximately 100 mL of dark brown sludge
Q2-1-034-96	reddish brown sludge
Q2-1-035-96	reddish brown sludge
Q2-1-036-96	reddish brown sludge
Q2-1-037-96	reddish brown sludge
Q2-1-038-96	reddish brown sludge

Site R-4
Collection Date: March 19, 1996
No. of cores: 13

Core No.	Description (colour, layering, etc.)
R4-1-001-96	little sample, mainly tailings
R4-1-002-96	chunks of red-brown sludge
R4-1-003-96	red brown sludge, little water visible
R4-1-004-96	red brown sludge, little water visible
R4-1-005-96	red brown sludge, lumpy
R4-1-006-96	red brown sludge, lumpy
R4-1-007-96	red brown sludge, lumpy
R4-1-008-96	red brown sludge
R4-1-009-96	red brown sludge
R4-1-010-96	red brown sludge
R4-1-011-96	red brown sludge
R4-1-012-96	red brown sludge
R4-1-013-96	red brown sludge

Site F-5
Collection Date: February 21, 1996
No. of cores 16

Core No.	Description (colour, layering, etc.)
F5-1-001-96	brown, dark grown, greasy, grainy
F5-1-002-96	mostly dark brown, very thick
F5-1-003-96	mostly dark brown, some light brown material
F5-1-004-96	light brown to dark brown from bottom to top
F5-1-005-96	dark brown to brown
F5-1-006-96	light brown, dark brown closer to eggshell
F5-1-007-96	mainly brown
F5-1-008-96	light to dark brown from top to bottom
F5-1-009-96	dark brown mainly
F5-1-010-96	dark brown, some lighter brown
F5-1-011-96	dark brown with large grains, more water than usual
F5-1-012-96	dark brown, similar to #11, again with more water than usual
F5-1-013-96	dark brown, evidence of undissolved lime
F5-1--15-96	dark brown large grains, more water than usual
F5-1-016-96	dark brown, large grains, more water than usual
F5-1-017-96	dark brown to brown, top to bottom, more water than usual

Site S-6
Collection Date: February 2, 1996
No. of Cores: 8

Core No.	Description (colour, layering, etc.)
S6-1-006-96	brown, a lot of water
S6-1-007-96	small sample, no layering, a lot of water
S6-1-008-96	granular particles, some undissolved lime; water
S6-1-013-96	denser sludge, darker brown, some water on top
S6-1-015-96	light brown, some larger particles
S6-1-016-96	brown, large grains, water
S6-1-009-96	brown, no layers -water layer on top
S6-1-027-96	brown, no layers, lots of water

Site J-7
Collection Date: March 20, 1996
No. of cores 29

Core No.	Description (colour, layering, etc.)
J7-2-001-96	Reddish brown sludge, uniform colour
J7-2-002-96	Reddish brown sludge, uniform colour
J7-2-003-96	Reddish brown sludge, uniform colour
J7-2-004-96	Reddish brown sludge, uniform colour
J7-2-005-96	uniform reddish brown sludge
J7-2-006-96	uniform reddish brown sludge
J7-2-007-96	uniform reddish brown sludge
J7-2-008-96	uniform reddish brown sludge
J7-2-009-96	reddish brown, darker brown layer in centre
J7-2-010-96	reddish brown
J7-2-011-96	some water sitting on top of brown sludge
J7-2-012-96	reddish brown sludge
J7-2-013-96	reddish brown sludge with a bit more water
J7-2-014-96	reddish brown
J7-1-015-96	reddish brown, more water than usual
J7-1-016-96	reddish brown
J7-1-017-96	similar to 016, contains water
J7-1-018-96	similar to 016 with some small black patches
J7-1-019-96	similar to 016
J7-1-024-96	reddish brown with patches of black material
J7-1-028-96	reddish brown with patches of black material
J7-1-029-96	reddish brown with black layer in middle

Site W-8
Collection Date: February 22, 1996
No. of cores: 11

Core No.	Description (colour, layering, etc.)
NB-2-001-96	very dark brown, very little layering
NB-2-002-96	similar to 001, denser
NB-2-003-96	eggshell logged half way, preserved leaves in core, less dense
NB-2-004-96	dense, some layering and lime, granular
NB-2-005-96	very distinct layering, dark brown and lime layers
NB-2-006-96	only half core, no layering, granular....DIRT?
NB-2-007-96	dark brown, some lime layering at bottom
NB-2-008-96	dark brown, some layering, very dense
NB-2-009-96	lime layering, more lime at bottom of core
NB-2-010-96	increasingly more lime, half of core in layers
NB-2-011-96	75% of sample appears to be lime

Site N-9
Collection Date: February 14, 1996
No. of cores: 11

Core No.	Description (colour, layering, etc.)
N9-3-001-96	dark brown, mostly green colour at bottom
N9-3-002-96	reddish brown, some large particles, unreacted lime
N9-3-003-96	reddish brown, grayish layer at bottom
N9-3-004-96	reddish brown, grayish layer at bottom, some large particles
N9-3-005-96	dark brown with black layers at bottom, odour
N9-3-006-96	reddish brown, some layer gray colour, large particles
N9-3-007-96	brown sand, like appearance, black layer at bottom
N9-3-008-96	reddish brown, lots of water on top, large particles
N9-3-009-96	reddish brown, grayish layer at bottom, lots of water on top
N9-3-010-96	brown colour, large grain, some gray layers
N9-3-011-96	reddish brown layers, some gray layers

APPENDIX E: PARTICLE SIZE TERMINOLOGY

1. Title Section:

This area contains the following information:

a. Type of display
b. Type of bench
c. Software version

2. Header Section:

This area contains a user entered custom header accessed through the **Primary Keypad - Sample Desc key**

3. ASVR Section:

This area contains the current Automatic Small Volume Recirculator parameters when an ASVR is present.

4. Summary Data:

dv - Uncalibrated Sample Volume is used by an operator to achieve proper sample concentration prior to proceeding with a measurement: laser attenuation is used for Microtrac II models.

10%, 50%, 90% - Percentile Points (microns) show the given percent of the volume (or weight, if the specific gravity for all the particles is the same; generally a good assumption) that is smaller than the indicated size. The 50% is also known as the median diameter which is one of several measures of "average particle size".

mv - Mean Diameter (microns) of the Volume distribution represents the center of gravity of the distribution. The MV value is weighted by the presence of coarse particles. It is another type "average particle size."

$$mv = \frac{\Sigma V_i d_i}{\Sigma V_i}$$

Where:

Σ = "sum of"
V = volume percent in a channel size
d = channel diameter in microns

mn - Mean Number Diameter (microns) is the mean particle diameter calculated from the number versus diameter distribution (number frequency distribution).

$$mn = \frac{\Sigma \frac{V_i}{d_i^2}}{\Sigma \frac{V_i}{d_i^3}}$$

Where:

$\sum$ = "sum of"
V = volume percent in a channel size
d = channel diameter in microns

cs - Calculated Specific Surface Area (M^2/cc) provides an indication of specific surface area. Since the CS computation assumes solid, spherical particles, it should not be interchanged with BET or other adsorption methods of surface area determination since CS does not reflect porosity or unique topographic characteristics of particles.

ma - Mean Diameter (microns) of the Area distribution is calculated according to the equation below. Area means are another type of "average" which are less weighted by the presence of coarse particles than MV and therefore show smaller particle size.

$$ma = \frac{\Sigma V_i}{\Sigma\left(\frac{V_i}{d_i}\right)}$$

Where:

$\sum$ = "sum of"
V = volume percent in a channel size
d = channel diameter in microns

sd - Standard Deviation (microns) describes the width of the measured particle size distribution. It does not provide an indication of the statistical error about the mean of multiple measurements.

$$sd = \frac{(84\% - 16\%)}{2}$$

5. Run Information:

This section is located on both sides of the Summary Data and contains a description of all the parameters associated with the current sample analysis.

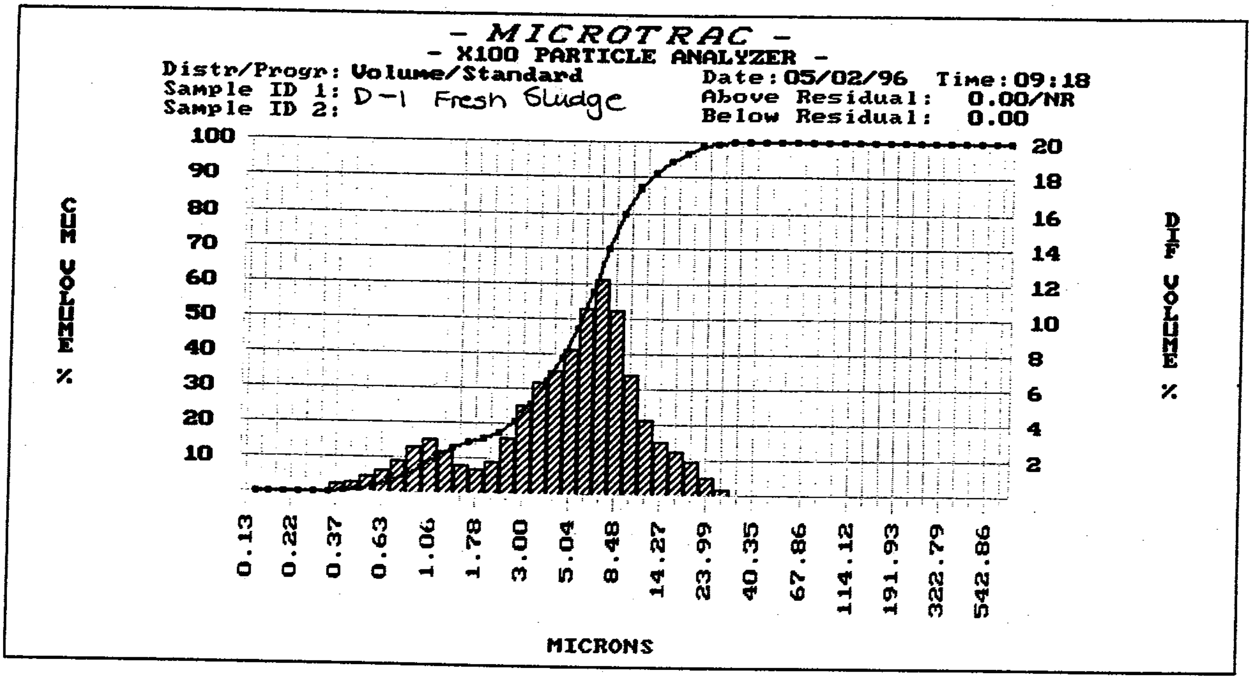
APPENDIX F: PARTICLE SIZE ANALYSES

```
                 - MICROTRAC X100 PARTICLE ANALYZER -
                         Percent Passing Data
                             Version 4.13

   D-1 fresh-treatment plant                        ASVR Parameters
 Fresh sludge
                                                Flow Rate: n/a ml/sec
                                                Ultrasonic Power: n/a watts
                                                Ultrasonic Time: n/a seconds

 Id #1:  D-1                      Summary Data    Id #2:
 Distrib. Format: Volume          dv  = 0.0323    Date: 05/02/96   Time:  9:18
 Filter: On                       10% =   1.13    Chan. Progression: Standard
 Run Time:  60 seconds            50% =   5.79    Upper Channel Edge: 704.00
 Run Number:   1 of  1 runs       90% =  12.34    Lower Channel Edge:   0.12
 Transmission: 0.95               mv  =   6.51    Number of Channels:  50
 Laser Int: 1.020/1.005/1.006     mn  =  0.711    Fluid Refractive Index: n/a
 Residuals: Disabled              ma  =   3.06    Transparent Particles: No
 Above Residual:    0.00          cs  =  1.962    Spherical Particles: n/a
 Below Residual:    0.00          sd  =   4.04    Part. Refractive Index: n/a

ch top  %pass %-chn  ch top  %pass %-chn  ch top  %pass %-chn  ch top  %pass %-chn
704.00 100.00  0.00    9.25  80.22 10.41
591.99 100.00  0.00    7.78  69.81 12.18
497.80 100.00  0.00    6.54  57.63 10.55
418.60 100.00  0.00    5.50  47.08  8.18
352.00 100.00  0.00    4.62  38.90  6.95
296.00 100.00  0.00    3.89  31.95  6.36
248.90 100.00  0.00    3.27  25.59  5.04
209.30 100.00  0.00    2.75  20.55  3.08
176.00 100.00  0.00    2.31  17.47  1.75
148.00 100.00  0.00    1.94  15.72  1.33
124.45 100.00  0.00    1.64  14.39  1.59
104.65 100.00  0.00    1.38  12.80  2.36
 88.00 100.00  0.00    1.16  10.44  2.96
 74.00 100.00  0.00    0.97   7.48  2.61
 62.23 100.00  0.00    0.82   4.87  1.81
 52.33 100.00  0.00    0.69   3.06  1.21
 44.00 100.00  0.00    0.58   1.85  0.85
 37.00 100.00  0.00    0.49   1.00  0.59
 31.11 100.00  0.36    0.41   0.41  0.41
 26.16  99.64  1.02    0.34   0.00  0.00
 22.00  98.62  1.84    0.29   0.00  0.00
 18.50  96.78  2.46    0.24   0.00  0.00
 15.56  94.32  3.05    0.20   0.00  0.00
 13.08  91.27  4.25    0.17   0.00  0.00
 11.00  87.02  6.80    0.14   0.00  0.00
```



- MICROTRAC -
- X100 PARTICLE ANALYZER -

Distr/Progr: Volume/Standard
Sample ID 1: D-1 Fresh Sludge
Sample ID 2:

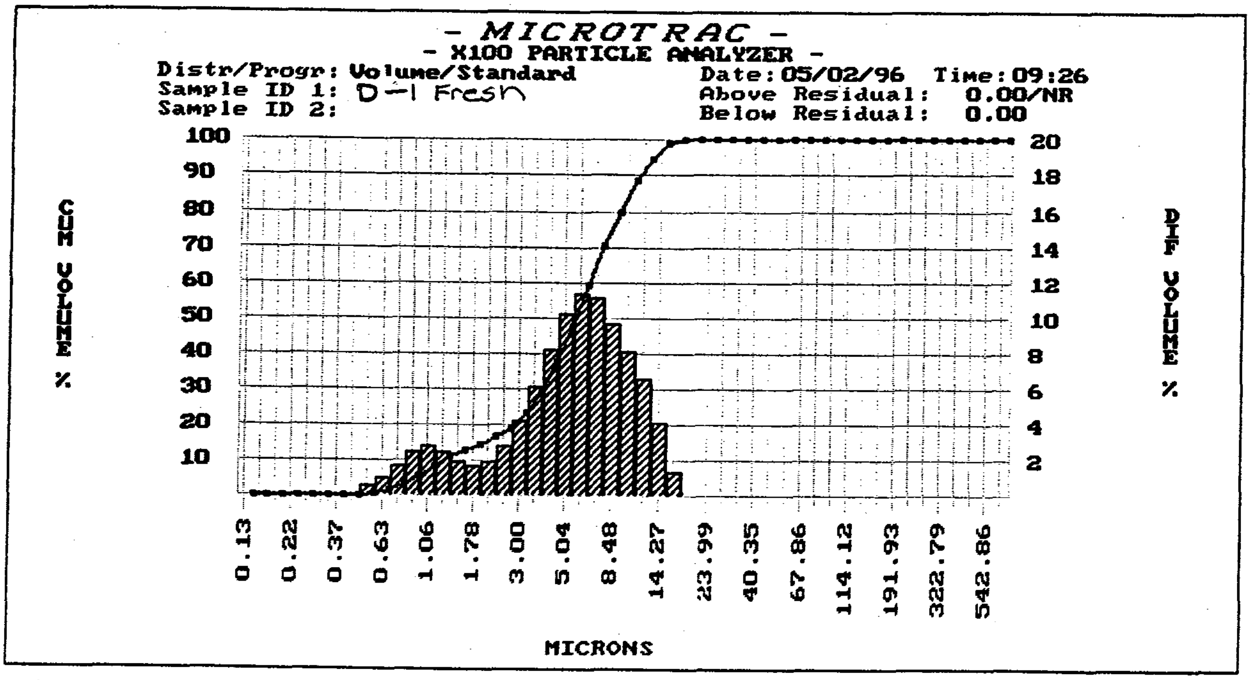
Date: 05/02/96 Time: 09:18
Above Residual: 0.00/NR
Below Residual: 0.00

```
                    - MICROTRAC X100 PARTICLE ANALYZER -
                           Percent Passing Data
                               Version 4.13

   D-1      treatment plant                        ASVR Parameters
 Fresh sludge
                                              Flow Rate: n/a ml/sec
                                              Ultrasonic Power: n/a watts
                                              Ultrasonic Time: n/a seconds

 Id #1:  D-1                  Summary Data    Id #2:
 Distrib. Format: Volume      dv  = 0.0344    Date: 05/02/96    Time:  9:26
 Filter: On                   10% =   1.28    Chan. Progression: Standard
 Run Time:  60 seconds        50% =   5.68    Upper Channel Edge: 704.00
 Run Number:   1 of   1 runs  90% =  11.50    Lower Channel Edge:   0.12
 Transmission: 0.94           mv  =   6.16    Number of Channels:  50
 Laser Int: 1.020/1.005/1.006 mn  =  0.949    Fluid Refractive Index: n/a
 Residuals: Disabled          ma  =   3.36    Transparent Particles: No
 Above Residual:    0.00      cs  =  1.785    Spherical Particles: n/a
 Below Residual:    0.00      sd  =   3.90    Part. Refractive Index: n/a

ch top  %pass %-chn ch top  %pass %-chn ch top  %pass %-chn ch top  %pass %-chn
704.00 100.00  0.00   9.25  80.01  9.68
591.99 100.00  0.00   7.78  70.33 11.06
497.80 100.00  0.00   6.54  59.27 11.34
418.60 100.00  0.00   5.50  47.93 10.18
352.00 100.00  0.00   4.62  37.75  8.24
296.00 100.00  0.00   3.89  29.51  6.16
248.90 100.00  0.00   3.27  23.35  4.22
209.30 100.00  0.00   2.75  19.13  2.73
176.00 100.00  0.00   2.31  16.40  1.89
148.00 100.00  0.00   1.94  14.51  1.66
124.45 100.00  0.00   1.64  12.85  1.90
104.65 100.00  0.00   1.38  10.95  2.45
 88.00 100.00  0.00   1.16   8.50  2.78
 74.00 100.00  0.00   0.97   5.72  2.45
 62.23 100.00  0.00   0.82   3.27  1.71
 52.33 100.00  0.00   0.69   1.56  1.02
 44.00 100.00  0.00   0.58   0.54  0.54
 37.00 100.00  0.00   0.49   0.00  0.00
 31.11 100.00  0.00   0.41   0.00  0.00
 26.16 100.00  0.00   0.34   0.00  0.00
 22.00 100.00  0.00   0.29   0.00  0.00
 18.50 100.00  1.28   0.24   0.00  0.00
 15.56  98.72  4.06   0.20   0.00  0.00
 13.08  94.66  6.52   0.17   0.00  0.00
 11.00  88.14  8.13   0.14   0.00  0.00
```



- MICROTRAC -

- X100 PARTICLE ANALYZER -

Distr/Progr: Volume/Standard
Sample ID 1: D-1 Fresh
Sample ID 2:

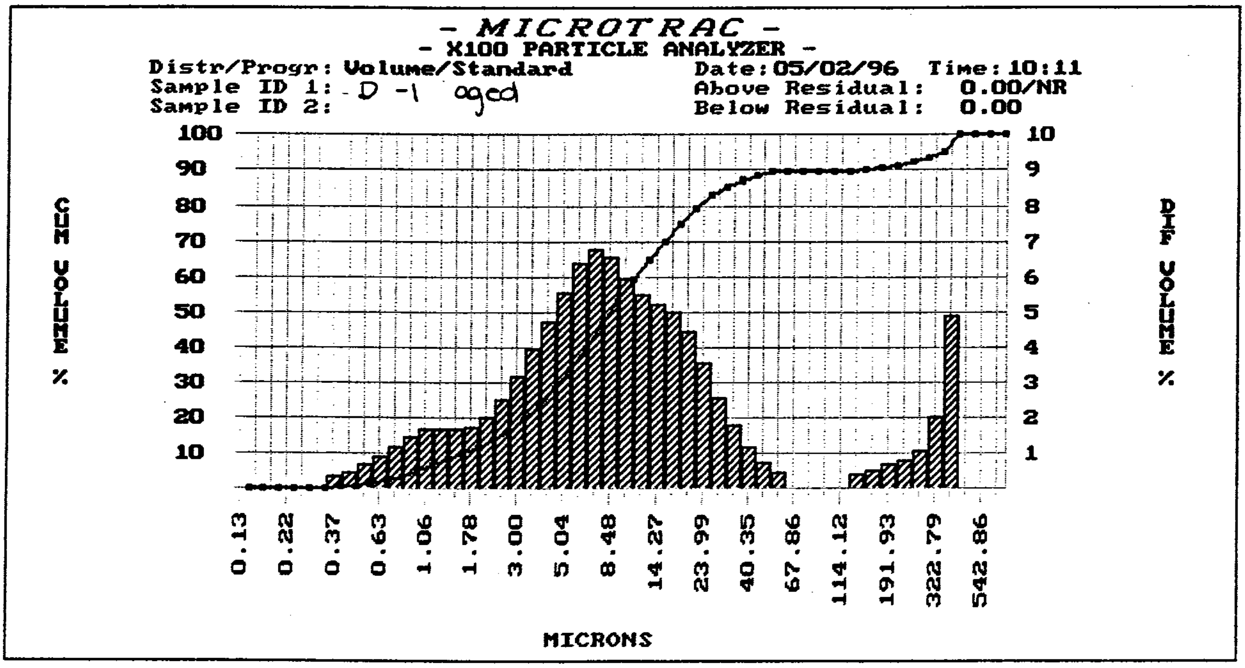
Date: 05/02/96 Time: 09:26
Above Residual: 0.00/NR
Below Residual: 0.00

```
                 - MICROTRAC X100 PARTICLE ANALYZER -
                        Percent Passing Data
                            Version 4.13

 D-1    sludge pond                               ASVR Parameters
 Composite aged sludge
                                           Flow Rate: n/a ml/sec
                                           Ultrasonic Power: n/a watts
                                           Ultrasonic Time: n/a seconds

 Id #1:  D-1.                  Summary Data   Id #2:
 Distrib. Format: Volume       dv  = 0.0554   Date: 05/02/96   Time: 10:11
 Filter: On                    10% =   1.63   Chan. Progression: Standard
 Run Time:  60 seconds         50% =   8.44   Upper Channel Edge: 704.00
 Run Number:   1 of  2 runs    90% = 142.85   Lower Channel Edge:   0.12
 Transmission: 0.94            mv  =  41.98   Number of Channels:  50
 Laser Int: 1.020/1.005/1.005  mn  =  0.692   Fluid Refractive Index: n/a
 Residuals: Disabled           ma  =   4.12   Transparent Particles: No
 Above Residual:   0.00        cs  =  1.455   Spherical Particles: n/a
 Below Residual:   0.00        sd  =  12.50   Part. Refractive Index: n/a

ch top  %pass %-chn ch top  %pass %-chn ch top  %pass %-chn ch top  %pass %-chn
704.00 100.00  0.00   9.25  53.40  6.54
591.99 100.00  0.00   7.78  46.86  6.80
497.80 100.00  0.00   6.54  40.06  6.39
418.60 100.00  4.89   5.50  33.67  5.57
352.00  95.11  2.02   4.62  28.10  4.70
296.00  93.09  1.07   3.89  23.40  3.92
248.90  92.02  0.76   3.27  19.48  3.18
209.30  91.26  0.64   2.75  16.30  2.51
176.00  90.62  0.52   2.31  13.79  2.01
148.00  90.10  0.39   1.94  11.78  1.74
124.45  89.71  0.00   1.64  10.04  1.67
104.65  89.71  0.00   1.38   8.37  1.69
 88.00  89.71  0.00   1.16   6.68  1.66
 74.00  89.71  0.00   0.97   5.02  1.47
 62.23  89.71  0.43   0.82   3.55  1.18
 52.33  89.28  0.71   0.69   2.37  0.89
 44.00  88.57  1.15   0.58   1.48  0.65
 37.00  87.42  1.78   0.49   0.83  0.47
 31.11  85.64  2.58   0.41   0.36  0.36
 26.16  83.06  3.54   0.34   0.00  0.00
 22.00  79.52  4.42   0.29   0.00  0.00
 18.50  75.10  5.00   0.24   0.00  0.00
 15.56  70.10  5.25   0.20   0.00  0.00
 13.08  64.85  5.49   0.17   0.00  0.00
 11.00  59.36  5.96   0.14   0.00  0.00
```



- MICROTRAC -

- X100 PARTICLE ANALYZER -

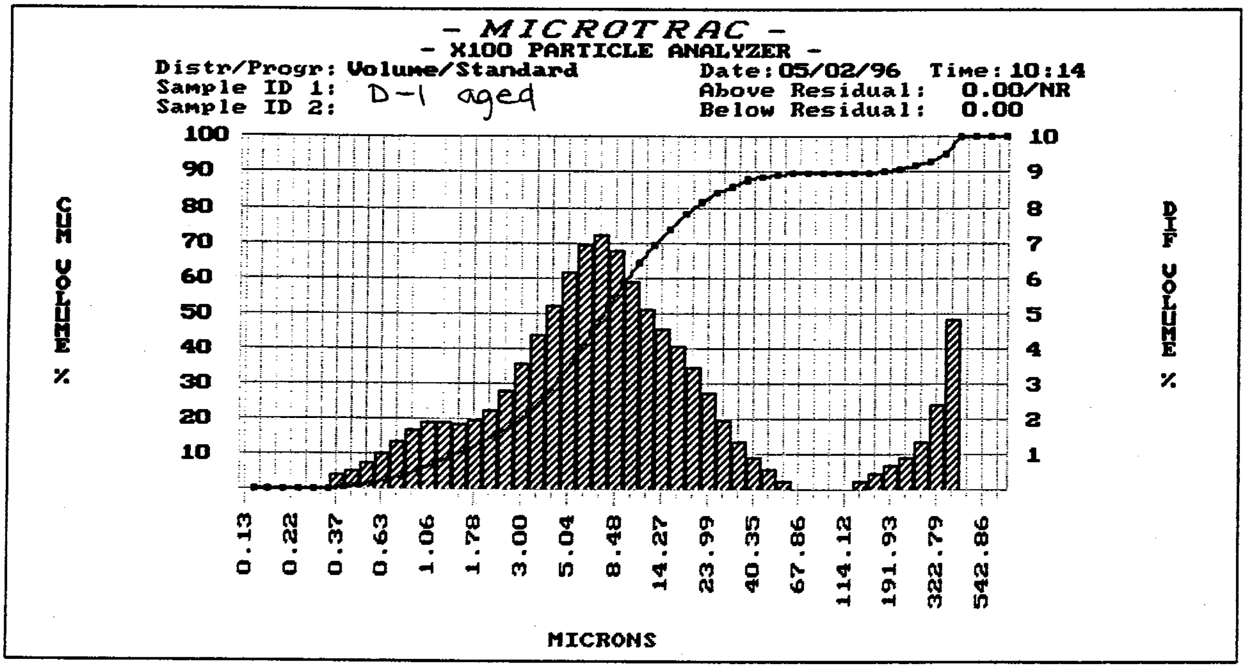
Distr/Progr: Volume/Standard Date: 05/02/96 Time: 10:11
Sample ID 1: D -1 aged Above Residual: 0.00/NR
Sample ID 2: Below Residual: 0.00

```
                 - MICROTRAC X100 PARTICLE ANALYZER -
                        Percent Passing Data
                            Version 4.13

     D-1    sludge pond                         ASVR Parameters
 Composite aged sludge
                                         Flow Rate: n/a ml/sec
                                         Ultrasonic Power: n/a watts
                                         Ultrasonic Time: n/a seconds

 Id #1: D-1                  Summary Data     Id #2:
 Distrib. Format: Volume     dv  = 0.0521     Date: 05/02/96   Time: 10:14
 Filter: On                  10% =   1.46     Chan. Progression: Standard
 Run Time:  60 seconds       50% =   7.47     Upper Channel Edge: 704.00
 Run Number: Avg of  2 runs  90% = 179.74     Lower Channel Edge:   0.12
 Transmission: 0.94          mv  =  42.42     Number of Channels:  50
 Laser Int: 1.020/1.005/1.005 mn =  0.693     Fluid Refractive Index: n/a
 Residuals: Disabled         ma  =   3.79     Transparent Particles: No
 Above Residual:    0.00     cs  =  1.581     Spherical Particles: n/a
 Below Residual:    0.00     sd  =  11.58     Part. Refractive Index: n/a

ch top  %pass  %-chn ch top   %pass  %-chn ch top   %pass %-chn ch top   %pass %-chn
704.00 100.00   0.00   9.25   58.50   6.79
591.99 100.00   0.00   7.78   51.71   7.23
497.80 100.00   0.00   6.54   44.48   6.97
418.60 100.00   4.83   5.50   37.51   6.15
352.00  95.17   2.38   4.62   31.36   5.24
296.00  92.79   1.33   3.89   26.12   4.39
248.90  91.46   0.88   3.27   21.73   3.57
209.30  90.58   0.65   2.75   18.16   2.80
176.00  89.93   0.47   2.31   15.36   2.22
148.00  89.46   0.20   1.94   13.14   1.92
124.45  89.26   0.00   1.64   11.22   1.85
104.65  89.26   0.00   1.38    9.37   1.90
 88.00  89.26   0.00   1.16    7.47   1.87
 74.00  89.26   0.00   0.97    5.60   1.66
 62.23  89.26   0.21   0.82    3.94   1.32
 52.33  89.05   0.53   0.69    2.62   0.99
 44.00  88.52   0.88   0.58    1.63   0.72
 37.00  87.64   1.35   0.49    0.91   0.52
 31.11  86.29   1.97   0.41    0.39   0.39
 26.16  84.32   2.71   0.34    0.00   0.00
 22.00  81.61   3.46   0.29    0.00   0.00
 18.50  78.15   4.08   0.24    0.00   0.00
 15.56  74.07   4.56   0.20    0.00   0.00
 13.08  69.51   5.11   0.17    0.00   0.00
 11.00  64.40   5.90   0.14    0.00   0.00
```



- MICROTRAC -

- X100 PARTICLE ANALYZER -

Distr/Progr: Volume/Standard Date: 05/02/96 Time: 10:14
Sample ID 1: D-1 aged Above Residual: 0.00/NR
Sample ID 2: Below Residual: 0.00

```
               - MICROTRAC X100 PARTICLE ANALYZER -
                       Percent Passing Data
                           Version 4.13

    Q-2 Fresh                                     ASVR Parameters

                                         Flow Rate: n/a ml/sec
                                         Ultrasonic Power: n/a watts
                                         Ultrasonic Time: n/a seconds

 Id #1: Q-2 Fresh            Summary Data   Id #2:
 Distrib. Format: Volume     dv  = 0.0251   Date: 12/13/96   Time: 12:22
 Filter: On                  10% =   1.73   Chan. Progression: Standard
 Run Time:  60 seconds       50% =   6.78   Upper Channel Edge: 704.00
 Run Number: Avg of  2 runs  90% =  17.08   Lower Channel Edge:   0.12
 Transmission: 0.96          mv  =   8.18   Number of Channels:  50
 Laser Int: 1.021/1.003/1.008 mn =  0.930   Fluid Refractive Index: n/a
 Residuals: Disabled         ma  =   3.95   Transparent Particles: No
 Above Residual:   0.00      cs  =  1.519   Spherical Particles: n/a
 Below Residual:   0.00      sd  =   6.02   Part. Refractive Index: n/a

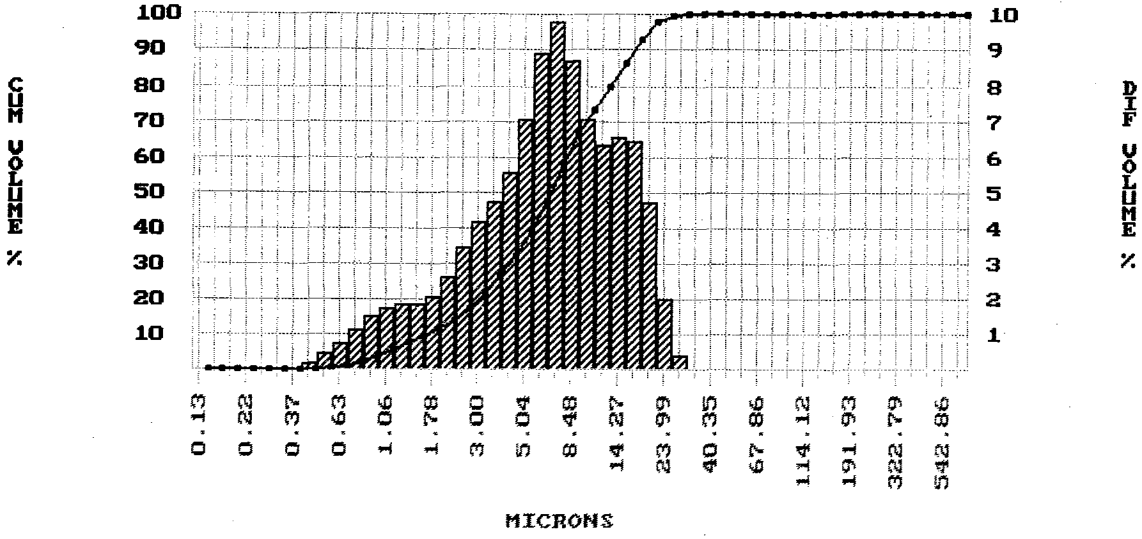
ch top  %pass %-chn ch top  %pass %-chn ch top   %pass %-chn ch top   %pass %-chn
704.00 100.00  0.00   9.25  66.46  8.72
591.99 100.00  0.00   7.78  57.74  9.77
497.80 100.00  0.00   6.54  47.97  8.91
418.60 100.00  0.00   5.50  39.06  7.08
352.00 100.00  0.00   4.62  31.98  5.58
296.00 100.00  0.00   3.89  26.40  4.75
248.90 100.00  0.00   3.27  21.65  4.15
209.30 100.00  0.00   2.75  17.50  3.42
176.00 100.00  0.00   2.31  14.08  2.63
148.00 100.00  0.00   1.94  11.45  2.08
124.45 100.00  0.00   1.64   9.37  1.86
104.65 100.00  0.00   1.38   7.51  1.82
 88.00 100.00  0.00   1.16   5.69  1.74
 74.00 100.00  0.00   0.97   3.95  1.48
 62.23 100.00  0.00   0.82   2.47  1.10
 52.33 100.00  0.00   0.69   1.37  0.74
 44.00 100.00  0.00   0.58   0.63  0.46
 37.00 100.00  0.00   0.49   0.17  0.17
 31.11 100.00  0.38   0.41   0.00  0.00
 26.16  99.62  1.99   0.34   0.00  0.00
 22.00  97.63  4.75   0.29   0.00  0.00
 18.50  92.88  6.46   0.24   0.00  0.00
 15.56  86.42  6.54   0.20   0.00  0.00
 13.08  79.88  6.34   0.17   0.00  0.00
 11.00  73.54  7.08   0.14   0.00  0.00
```

- *MICROTRAC* -

- X100 PARTICLE ANALYZER -

Distr/Progr: **Volume/Standard**
Sample ID 1: Q-2 **-F**
Sample ID 2:

Date: **12/13/96** Time: **12:22**
Above Residual: **0.00/NR**
Below Residual: **0.00**



```
                 - MICROTRAC X100 PARTICLE ANALYZER -
                         Percent Passing Data
                             Version 4.13

   Q-2 Fresh                                     ASVR Parameters

                                             Flow Rate: n/a ml/sec
                                             Ultrasonic Power: n/a watts
                                             Ultrasonic Time: n/a seconds

 Id #1:  Q-2   -F              Summary Data   Id #2:
 Distrib. Format: Volume       dv  = 0.0222   Date: 12/13/96   Time: 12:21
 Filter: On                    10% =   1.51   Chan. Progression: Standard
 Run Time:  60 seconds         50% =   5.91   Upper Channel Edge: 704.00
 Run Number:   2 of  2 runs    90% =  14.38   Lower Channel Edge:   0.12
 Transmission: 0.96            mv  =   6.96   Number of Channels:  50
 Laser Int: 1.021/1.003/1.008  mn  =  0.879   Fluid Refractive Index: n/a
 Residuals: Disabled           ma  =   3.51   Transparent Particles: No
 Above Residual:   0.00        cs  =  1.710   Spherical Particles: n/a
 Below Residual:   0.00        sd  =   5.00   Part. Refractive Index: n/a

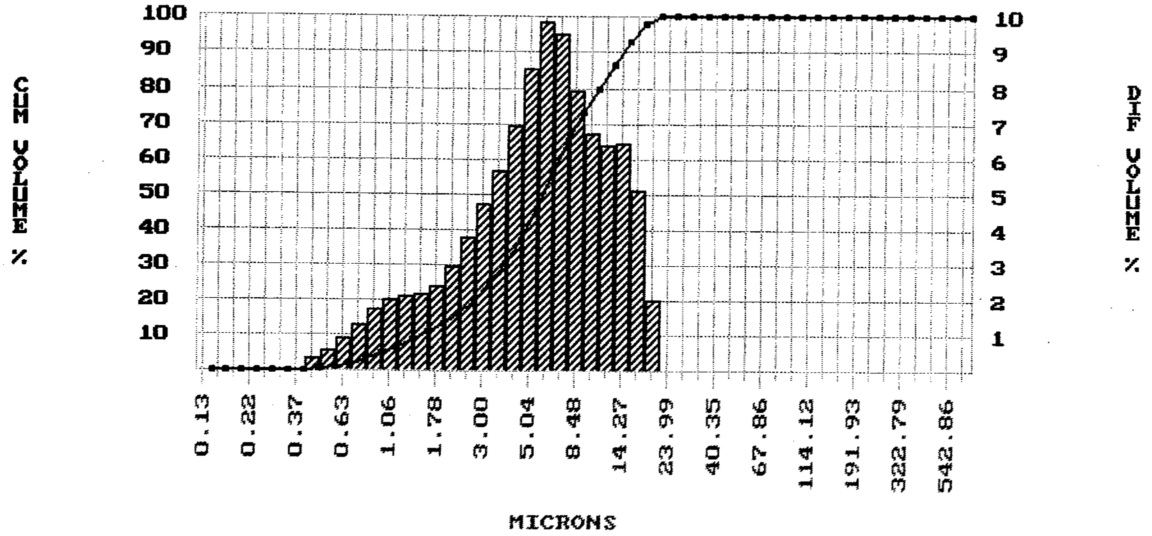
ch top  %pass %-chn ch top  %pass %-chn ch top  %pass %-chn ch top  %pass %-chn
704.00 100.00  0.00   9.25  73.34  7.97
591.99 100.00  0.00   7.78  65.37  9.52
497.80 100.00  0.00   6.54  55.85  9.82
418.60 100.00  0.00   5.50  46.03  8.58
352.00 100.00  0.00   4.62  37.45  6.92
296.00 100.00  0.00   3.89  30.53  5.67
248.90 100.00  0.00   3.27  24.86  4.71
209.30 100.00  0.00   2.75  20.15  3.79
176.00 100.00  0.00   2.31  16.36  2.95
148.00 100.00  0.00   1.94  13.41  2.39
124.45 100.00  0.00   1.64  11.02  2.15
104.65 100.00  0.00   1.38   8.87  2.09
 88.00 100.00  0.00   1.16   6.78  1.99
 74.00 100.00  0.00   0.97   4.79  1.70
 62.23 100.00  0.00   0.82   3.09  1.29
 52.33 100.00  0.00   0.69   1.80  0.88
 44.00 100.00  0.00   0.58   0.92  0.57
 37.00 100.00  0.00   0.49   0.35  0.35
 31.11 100.00  0.00   0.41   0.00  0.00
 26.16 100.00  0.00   0.34   0.00  0.00
 22.00 100.00  2.01   0.29   0.00  0.00
 18.50  97.99  5.10   0.24   0.00  0.00
 15.56  92.89  6.46   0.20   0.00  0.00
 13.08  86.43  6.39   0.17   0.00  0.00
 11.00  80.04  6.70   0.14   0.00  0.00
```

- MICROTRAC -

- X100 PARTICLE ANALYZER -

Distr/Progr: **Volume/Standard**
Sample ID 1: Q-2 **-F**
Sample ID 2:

Date: **12/13/96** Time: **12:21**
Above Residual: **0.00/NR**
Below Residual: **0.00**



- MICROTRAC X100 PARTICLE ANALYZER -
Percent Passing Data
Version 4.13

Q-2 Fresh Sludge

ASVR Parameters

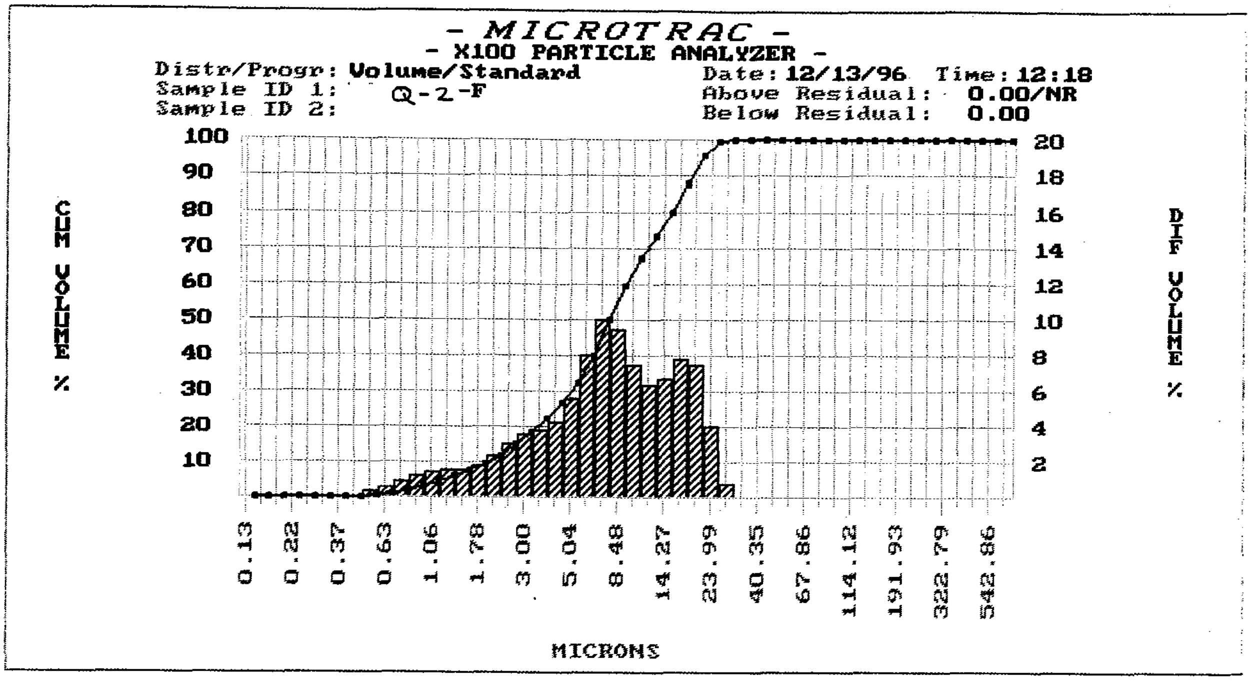
Flow Rate: n/a ml/sec
Ultrasonic Power: n/a watts
Ultrasonic Time: n/a seconds

Id #1: Q-2 -F
Distrib. Format: Volume
Filter: On
Run Time: 60 seconds
Run Number: 1 of 2 runs
Transmission: 0.96
Laser Int: 1.022/1.003/1.008
Residuals: Disabled
Above Residual: 0.00
Below Residual: 0.00

Summary Data
dv = 0.0280
10% = 2.02
50% = 7.76
90% = 19.39
mv = 9.39
mn = 1.01
ma = 4.50
cs = 1.332
sd = 7.06

Id #2:
Date: 12/13/96 Time: 12:18
Chan. Progression: Standard
Upper Channel Edge: 704.00
Lower Channel Edge: 0.12
Number of Channels: 50
Fluid Refractive Index: n/a
Transparent Particles: No
Spherical Particles: n/a
Part. Refractive Index: n/a

ch top	%pass	%-chn
704.00	100.00	0.00
591.99	100.00	0.00
497.80	100.00	0.00
418.60	100.00	0.00
352.00	100.00	0.00
296.00	100.00	0.00
248.90	100.00	0.00
209.30	100.00	0.00
176.00	100.00	0.00
148.00	100.00	0.00
124.45	100.00	0.00
104.65	100.00	0.00
88.00	100.00	0.00
74.00	100.00	0.00
62.23	100.00	0.00
52.33	100.00	0.00
44.00	100.00	0.00
37.00	100.00	0.00
31.11	100.00	0.75
26.16	99.25	3.97
22.00	95.28	7.48
18.50	87.80	7.81
15.56	79.99	6.62
13.08	73.37	6.29
11.00	67.08	7.47
9.25	59.61	9.48
7.78	50.13	10.02
6.54	40.11	8.00
5.50	32.11	5.58
4.62	26.53	4.25
3.89	22.28	3.82
3.27	18.46	3.59
2.75	14.87	3.04
2.31	11.83	2.30
1.94	9.53	1.78
1.64	7.75	1.57
1.38	6.18	1.55
1.16	4.63	1.49
0.97	3.14	1.26
0.82	1.88	0.92
0.69	0.96	0.60
0.58	0.36	0.36
0.49	0.00	0.00
0.41	0.00	0.00
0.34	0.00	0.00
0.29	0.00	0.00
0.24	0.00	0.00
0.20	0.00	0.00
0.17	0.00	0.00
0.14	0.00	0.00



- *MICROTRAC* -

- X100 PARTICLE ANALYZER -

Distr/Progr: **Volume/Standard**
Sample ID 1: Q-2-F
Sample ID 2:

Date: **12/13/96** Time: **12:18**
Above Residual: **0.00/NR**
Below Residual: **0.00**

```
              - MICROTRAC X100 PARTICLE ANALYZER -
                      Percent Passing Data
                          Version 4.13

   Q-2 Fresh                                       ASVR Parameters

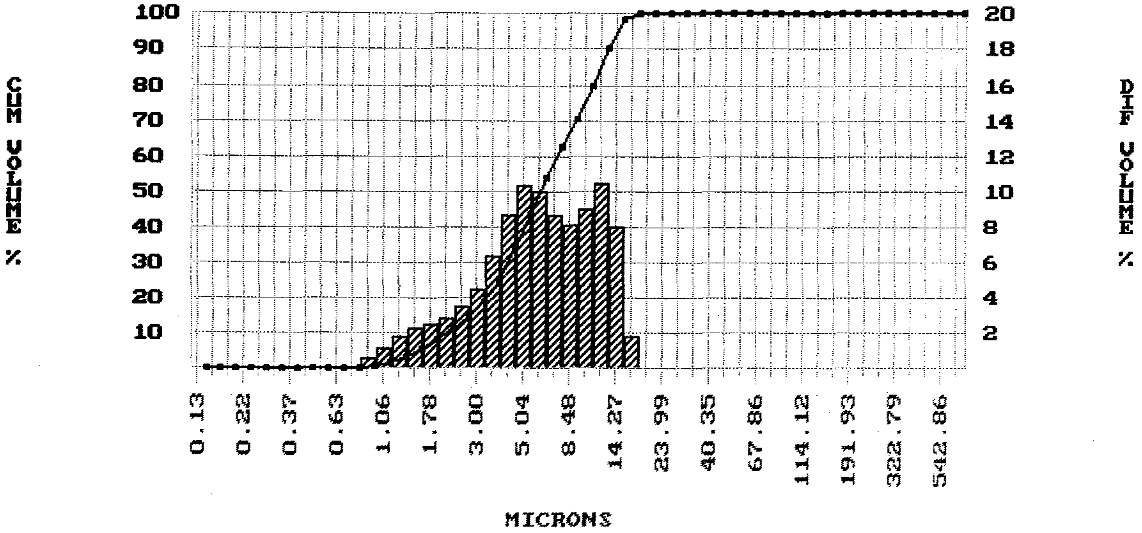
                                             Flow Rate: n/a ml/sec
                                             Ultrasonic Power: n/a watts
                                             Ultrasonic Time: n/a seconds

 Id #1:   Q-2-F                 Summary Data    Id #2:
 Distrib. Format: Volume        dv  = 0.0260    Date: 12/13/96   Time: 12:34
 Filter: On                     10% =   2.18    Chan. Progression: Standard
 Run Time:  60 seconds          50% =   6.09    Upper Channel Edge: 704.00
 Run Number:   2 of  2 runs     90% =  13.03    Lower Channel Edge:   0.12
 Transmission: 0.97             mv  =   7.01    Number of Channels:  50
 Laser Int: 1.021/1.003/1.007   mn  =   1.75    Fluid Refractive Index: n/a
 Residuals: Disabled            ma  =   4.50    Transparent Particles: No
 Above Residual:   0.00         cs  =  1.333    Spherical Particles: n/a
 Below Residual:   0.00         sd  =   4.43    Part. Refractive Index: n/a

ch top  %pass %-chn ch top  %pass %-chn ch top  %pass %-chn ch top  %pass %-chn
704.00 100.00  0.00   9.25  70.76  8.06
591.99 100.00  0.00   7.78  62.70  8.65
497.80 100.00  0.00   6.54  54.05  9.98
418.60 100.00  0.00   5.50  44.07 10.28
352.00 100.00  0.00   4.62  33.79  8.65
296.00 100.00  0.00   3.89  25.14  6.28
248.90 100.00  0.00   3.27  18.86  4.46
209.30 100.00  0.00   2.75  14.40  3.39
176.00 100.00  0.00   2.31  11.01  2.81
148.00 100.00  0.00   1.94   8.20  2.50
124.45 100.00  0.00   1.64   5.70  2.24
104.65 100.00  0.00   1.38   3.46  1.80
 88.00 100.00  0.00   1.16   1.66  1.13
 74.00 100.00  0.00   0.97   0.53  0.53
 62.23 100.00  0.00   0.82   0.00  0.00
 52.33 100.00  0.00   0.69   0.00  0.00
 44.00 100.00  0.00   0.58   0.00  0.00
 37.00 100.00  0.00   0.49   0.00  0.00
 31.11 100.00  0.00   0.41   0.00  0.00
 26.16 100.00  0.00   0.34   0.00  0.00
 22.00 100.00  0.00   0.29   0.00  0.00
 18.50 100.00  1.78   0.24   0.00  0.00
 15.56  98.22  8.01   0.20   0.00  0.00
 13.08  90.21 10.47   0.17   0.00  0.00
 11.00  79.74  8.98   0.14   0.00  0.00
```

- MICROTRAC -
- X100 PARTICLE ANALYZER -

Distr/Progr: **Volume/Standard** Date: **12/13/96** Time: **12:34**
Sample ID 1: . Q-2 -F Above Residual: **0.00/NR**
Sample ID 2: Below Residual: **0.00**



- MICROTRAC X100 PARTICLE ANALYZER -
Percent Passing Data
Version 4.13

Q-z
composite aged sludge

ASVR Parameters

Flow Rate: n/a ml/sec
Ultrasonic Power: n/a watts
Ultrasonic Time: n/a seconds

Id #1:
Distrib. Format: Volume
Filter: On
Run Time: 60 seconds
Run Number: 1 of 1 runs
Transmission: 0.96
Laser Int: 1.021/1.006/1.007
Residuals: Disabled
Above Residual: 0.00
Below Residual: 0.00

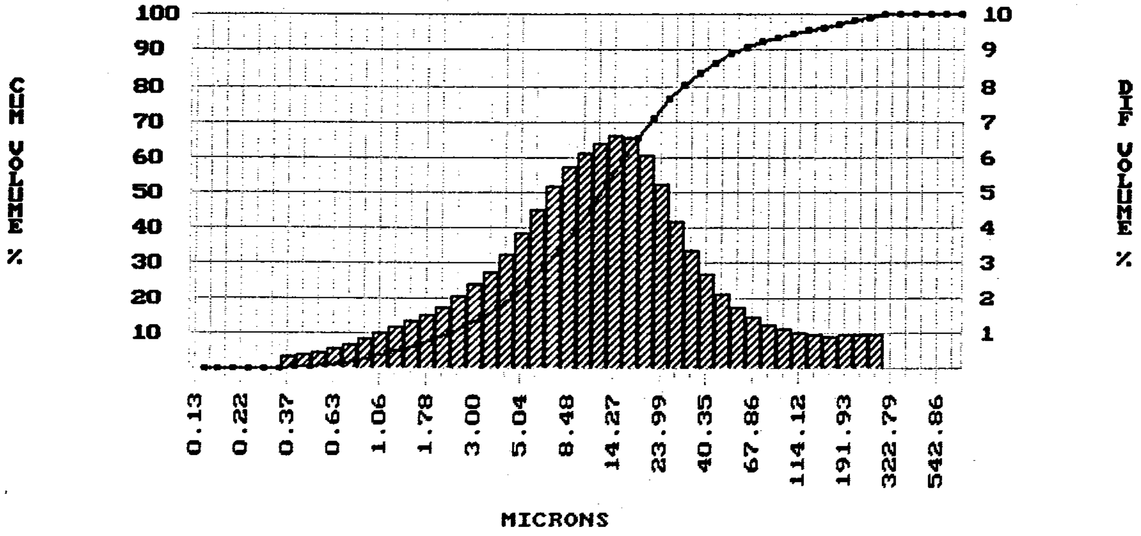
Summary Data
dv = 0.0434
10% = 2.31
50% = 12.37
90% = 58.49
mv = 26.47
mn = 0.671
ma = 5.33
cs = 1.125
sd = 16.60

Id #2:
Date: 05/01/96 Time: 13:33
Chan. Progression: Standard
Upper Channel Edge: 704.00
Lower Channel Edge: 0.12
Number of Channels: 50
Fluid Refractive Index: n/a
Transparent Particles: No
Spherical Particles: n/a
Part. Refractive Index: n/a

ch top	%pass	%-chn	ch top	%pass	%-chn	ch top	%pass	%-chn	ch top	%pass	%-chn
704.00	100.00	0.00	9.25	39.60	5.72						
591.99	100.00	0.00	7.78	33.88	5.19						
497.80	100.00	0.00	6.54	28.69	4.51						
418.60	100.00	0.00	5.50	24.18	3.81						
352.00	100.00	0.00	4.62	20.37	3.22						
296.00	100.00	0.95	3.89	17.15	2.75						
248.90	99.05	0.94	3.27	14.40	2.37						
209.30	98.11	0.92	2.75	12.03	2.03						
176.00	97.19	0.90	2.31	10.00	1.74						
148.00	96.29	0.93	1.94	8.26	1.50						
124.45	95.36	0.98	1.64	6.76	1.32						
104.65	94.38	1.09	1.38	5.44	1.17						
88.00	93.29	1.24	1.16	4.27	1.02						
74.00	92.05	1.46	0.97	3.25	0.85						
62.23	90.59	1.74	0.82	2.40	0.69						
52.33	88.85	2.13	0.69	1.71	0.55						
44.00	86.72	2.64	0.58	1.16	0.45						
37.00	84.08	3.33	0.49	0.71	0.38						
31.11	80.75	4.19	0.41	0.33	0.33						
26.16	76.56	5.20	0.34	0.00	0.00						
22.00	71.36	6.07	0.29	0.00	0.00						
18.50	65.29	6.58	0.24	0.00	0.00						
15.56	58.71	6.61	0.20	0.00	0.00						
13.08	52.10	6.40	0.17	0.00	0.00						
11.00	45.70	6.10	0.14	0.00	0.00						

- MICROTRAC -
- X100 PARTICLE ANALYZER -

Distr/Progr: Volume/Standard
Date: 05/01/96 Time: 13:33
Sample ID 1: Q-2 aged
Above Residual: 0.00/NR
Sample ID 2:
Below Residual: 0.00

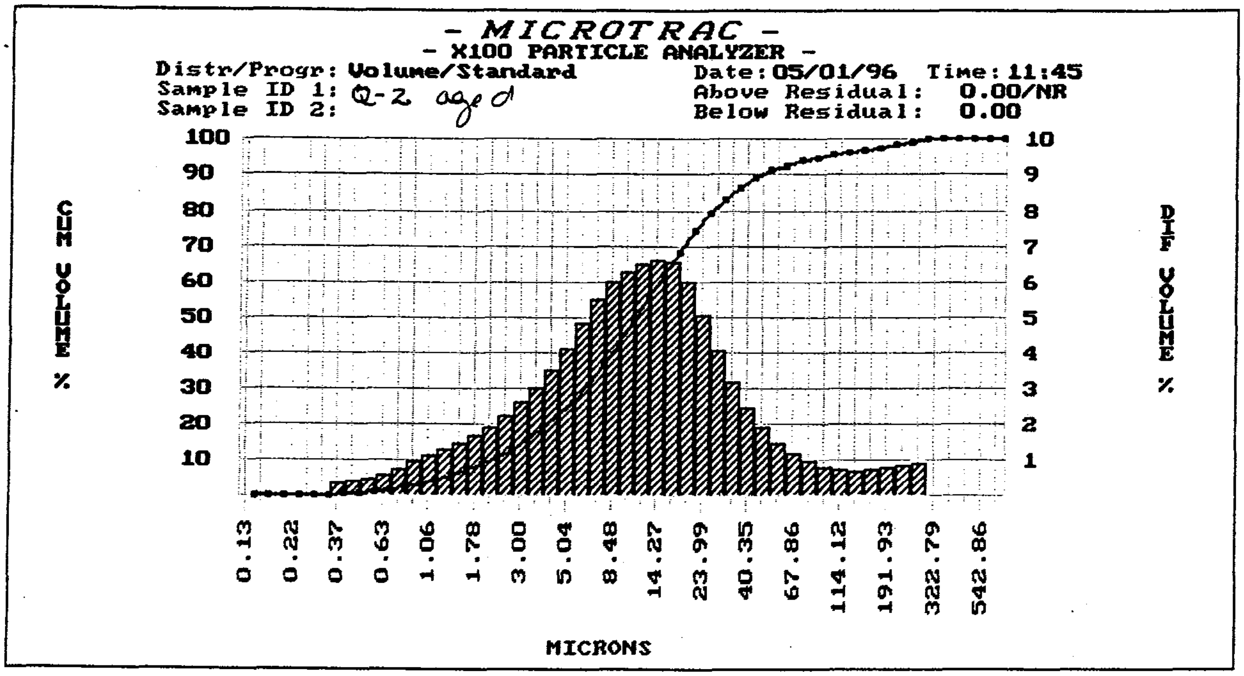


```
                 - MICROTRAC X100 PARTICLE ANALYZER -
                         Percent Passing Data
                             Version 4.13

 Q-2 sludge pond                               ASVR Parameters
 composite aged sludge
                                         Flow Rate: n/a ml/sec
                                         Ultrasonic Power: n/a watts
                                         Ultrasonic Time: n/a seconds

 Id #1:                       Summary Data   Id #2:
 Distrib. Format: Volume      dv  = 0.0670   Date: 05/01/96   Time: 11:45
 Filter: On                   10% =   2.16   Chan. Progression: Standard
 Run Time:  60 seconds        50% =  11.34   Upper Channel Edge: 704.00
 Run Number:   1 of  1 runs   90% =  47.31   Lower Channel Edge:    0.12
 Transmission: 0.94           mv  =  23.77   Number of Channels:  50
 Laser Int: 1.021/1.006/1.006 mn  =  0.680   Fluid Refractive Index: n/a
 Residuals: Disabled          ma  =   5.04   Transparent Particles: No
 Above Residual:    0.00      cs  =  1.190   Spherical Particles: n/a
 Below Residual:    0.00      sd  =  14.20   Part. Refractive Index: n/a

ch top  %pass %-chn|ch top  %pass %-chn|ch top  %pass %-chn|ch top  %pass %-chn
704.00 100.00  0.00|  9.25  42.57  6.02|                   |
591.99 100.00  0.00|  7.78  36.55  5.52|                   |
497.80 100.00  0.00|  6.54  31.03  4.83|                   |
418.60 100.00  0.00|  5.50  26.20  4.10|                   |
352.00 100.00  0.00|  4.62  22.10  3.48|                   |
296.00 100.00  0.90|  3.89  18.62  3.00|                   |
248.90  99.10  0.84|  3.27  15.62  2.60|                   |
209.30  98.26  0.77|  2.75  13.02  2.23|                   |
176.00  97.49  0.71|  2.31  10.79  1.90|                   |
148.00  96.78  0.69|  1.94   8.89  1.64|                   |
124.45  96.09  0.72|  1.64   7.25  1.45|                   |
104.65  95.37  0.80|  1.38   5.80  1.29|                   |
 88.00  94.57  0.93|  1.16   4.51  1.11|                   |
 74.00  93.64  1.15|  0.97   3.40  0.92|                   |
 62.23  92.49  1.45|  0.82   2.48  0.73|                   |
 52.33  91.04  1.88|  0.69   1.75  0.57|                   |
 44.00  89.16  2.42|  0.58   1.18  0.46|                   |
 37.00  86.74  3.14|  0.49   0.72  0.38|                   |
 31.11  83.60  4.03|  0.41   0.34  0.34|                   |
 26.16  79.57  5.07|  0.34   0.00  0.00|                   |
 22.00  74.50  5.98|  0.29   0.00  0.00|                   |
 18.50  68.52  6.54|  0.24   0.00  0.00|                   |
 15.56  61.98  6.61|  0.20   0.00  0.00|                   |
 13.08  55.37  6.50|  0.17   0.00  0.00|                   |
 11.00  48.87  6.30|  0.14   0.00  0.00|                   |
```



- MICROTRAC -

- X100 PARTICLE ANALYZER -

Distr/Progr: Volume/Standard

Sample ID 1: Q-2 aged

Sample ID 2:

Date: 05/01/96 Time: 11:45

Above Residual: 0.00/NR

Below Residual: 0.00

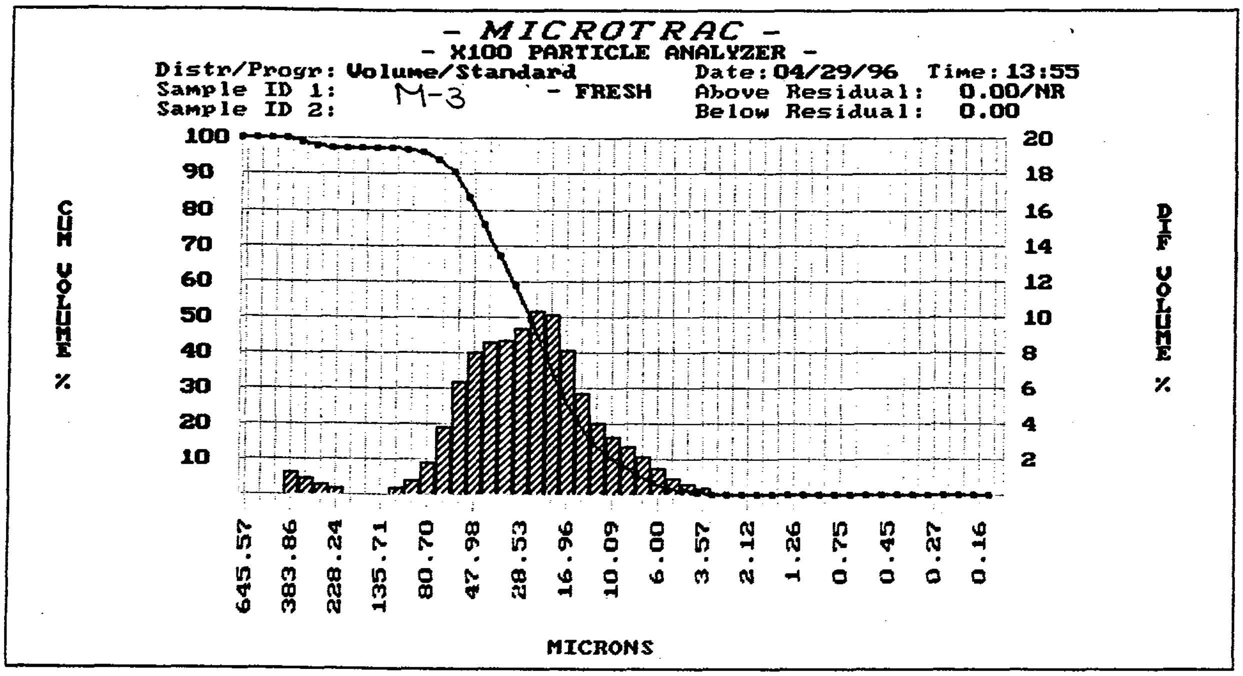
```
                  - MICROTRAC X100 PARTICLE ANALYZER -
                          Percent Passing Data
                              Version 4.13

      M-3     Fresh Sludge                         ASVR Parameters

 Sample 1                                 Flow Rate: n/a ml/sec
                                          Ultrasonic Power: n/a watts
                                          Ultrasonic Time: n/a seconds

 Id #1:     M-3         FRESH     Summary Data   Id #2:
 Distrib. Format: Volume          dv  = 0.2284   Date: 04/29/96   Time: 13:55
 Filter: On                       10% =  10.32   Chan. Progression: Standard
 Run Time:  60 seconds            50% =  26.43   Upper Channel Edge: 704.00
 Run Number:   1 of  1 runs       90% =  61.64   Lower Channel Edge:   0.12
 Transmission: 0.93               mv  =  39.41   Number of Channels:  50
 Laser Int: 1.020/1.005/1.005     mn  =   7.91   Fluid Refractive Index: n/a
 Residuals: Disabled              ma  =  20.49   Transparent Particles: No
 Above Residual:   0.00           cs  =  0.293   Spherical Particles: n/a
 Below Residual:   0.00           sd  =  19.43   Part. Refractive Index: n/a

ch top  %pass %-chn ch top  %pass  %-chn ch top  %pass %-chn ch top  %pass %-chn
704.00 100.00  0.00   9.25   8.07   2.71
591.99 100.00  0.00   7.78   5.36   2.14
497.80 100.00  0.00   6.54   3.22   1.44
418.60 100.00  1.20   5.50   1.78   0.87
352.00  98.80  0.88   4.62   0.91   0.54
296.00  97.92  0.54   3.89   0.37   0.37
248.90  97.38  0.33   3.27   0.00   0.00
209.30  97.05  0.00   2.75   0.00   0.00
176.00  97.05  0.00   2.31   0.00   0.00
148.00  97.05  0.00   1.94   0.00   0.00
124.45  97.05  0.38   1.64   0.00   0.00
104.65  96.67  0.79   1.38   0.00   0.00
 88.00  95.88  1.80   1.16   0.00   0.00
 74.00  94.08  3.78   0.97   0.00   0.00
 62.23  90.30  6.29   0.82   0.00   0.00
 52.33  84.01  8.05   0.69   0.00   0.00
 44.00  75.96  8.51   0.58   0.00   0.00
 37.00  67.45  8.68   0.49   0.00   0.00
 31.11  58.77  9.34   0.41   0.00   0.00
 26.16  49.43 10.31   0.34   0.00   0.00
 22.00  39.12 10.14   0.29   0.00   0.00
 18.50  28.98  8.14   0.24   0.00   0.00
 15.56  20.84  5.63   0.20   0.00   0.00
 13.08  15.21  3.97   0.17   0.00   0.00
 11.00  11.24  3.17   0.14   0.00   0.00
```



- MICROTRAC -

- X100 PARTICLE ANALYZER -

Distr/Progr: Volume/Standard Date: 04/29/96 Time: 13:55
Sample ID 1: M-3 - FRESH Above Residual: 0.00/NR
Sample ID 2: Below Residual: 0.00

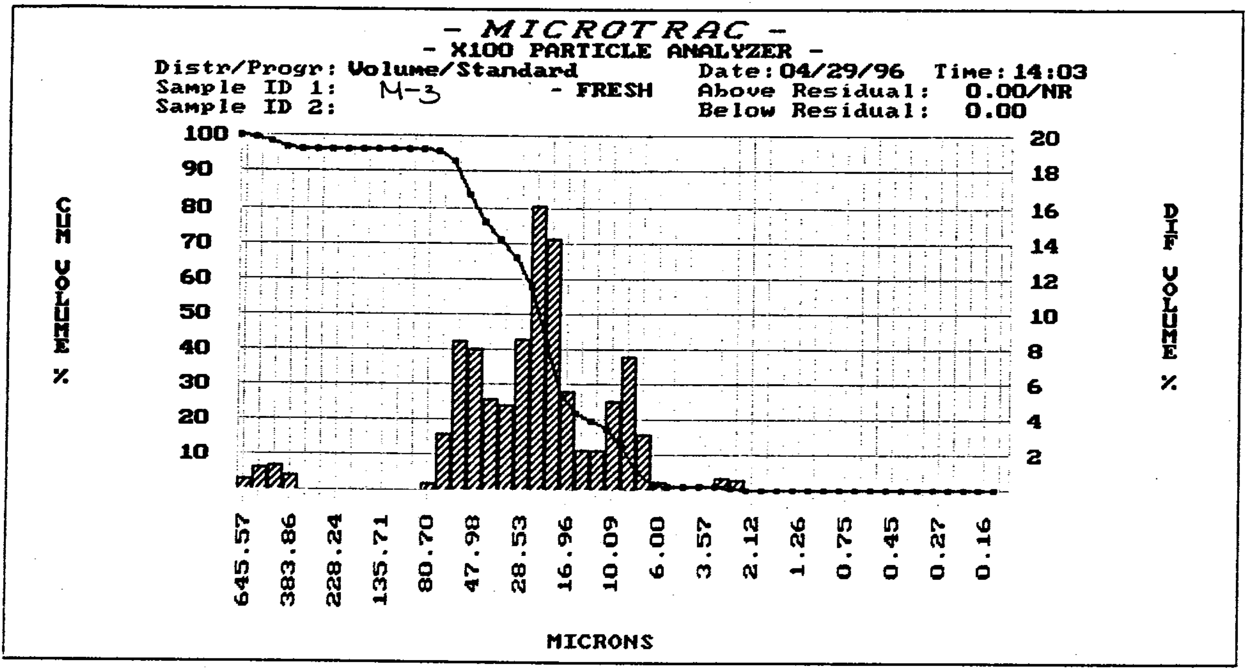
```
                 - MICROTRAC X100 PARTICLE ANALYZER -
                         Percent Passing Data
                             Version 4.13

    M-3      Fresh Sludge                          ASVR Parameters

 Sample 1                                   Flow Rate: n/a ml/sec
                                            Ultrasonic Power: n/a watts
                                            Ultrasonic Time: n/a seconds

 Id #1:  M-3          - FRESH     Summary Data     Id #2:
 Distrib. Format: Volume          dv  = 0.2893     Date: 04/29/96   Time: 14:03
 Filter: On                       10% =   8.78     Chan. Progression: Standard
 Run Time:  60 seconds            50% =  23.97     Upper Channel Edge: 704.00
 Run Number:   1 of  1 runs       90% =  58.60     Lower Channel Edge:   0.12
 Transmission: 0.91               mv  =  46.38     Number of Channels:  50
 Laser Int: 1.020/1.005/1.005     mn  =   5.53     Fluid Refractive Index: n/a
 Residuals: Disabled              ma  =  18.77     Transparent Particles: No
 Above Residual:   0.00           cs  =  0.320     Spherical Particles: n/a
 Below Residual:   0.00           sd  =  20.90     Part. Refractive Index: n/a

ch top  %pass %-chn|ch top  %pass %-chn|ch top   %pass %-chn|ch top   %pass %-chn
704.00 100.00  0.60|  9.25  12.34  7.52|                    |
591.99  99.40  1.21|  7.78   4.82  3.10|                    |
497.80  98.19  1.33|  6.54   1.72  0.50|                    |
418.60  96.86  0.79|  5.50   1.22  0.00|                    |
352.00  96.07  0.00|  4.62   1.22  0.00|                    |
296.00  96.07  0.00|  3.89   1.22  0.00|                    |
248.90  96.07  0.00|  3.27   1.22  0.64|                    |
209.30  96.07  0.00|  2.75   0.58  0.58|                    |
176.00  96.07  0.00|  2.31   0.00  0.00|                    |
148.00  96.07  0.00|  1.94   0.00  0.00|                    |
124.45  96.07  0.00|  1.64   0.00  0.00|                    |
104.65  96.07  0.00|  1.38   0.00  0.00|                    |
 88.00  96.07  0.38|  1.16   0.00  0.00|                    |
 74.00  95.69  3.12|  0.97   0.00  0.00|                    |
 62.23  92.57  8.42|  0.82   0.00  0.00|                    |
 52.33  84.15  7.96|  0.69   0.00  0.00|                    |
 44.00  76.19  5.06|  0.58   0.00  0.00|                    |
 37.00  71.13  4.83|  0.49   0.00  0.00|                    |
 31.11  66.30  8.61|  0.41   0.00  0.00|                    |
 26.16  57.69 16.13|  0.34   0.00  0.00|                    |
 22.00  41.56 14.22|  0.29   0.00  0.00|                    |
 18.50  27.34  5.53|  0.24   0.00  0.00|                    |
 15.56  21.81  2.21|  0.20   0.00  0.00|                    |
 13.08  19.60  2.26|  0.17   0.00  0.00|                    |
 11.00  17.34  5.00|  0.14   0.00  0.00|                    |
```



- MICROTRAC -

- X100 PARTICLE ANALYZER -

Distr/Progr: Volume/Standard   Date: 04/29/96   Time: 14:03
Sample ID 1: M-3   - FRESH   Above Residual: 0.00/NR
Sample ID 2:   Below Residual: 0.00

- MICROTRAC X100 PARTICLE ANALYZER -
Percent Passing Data
Version 4.13

R-4 - Fresh Sludge

Sample 1

ASVR Parameters

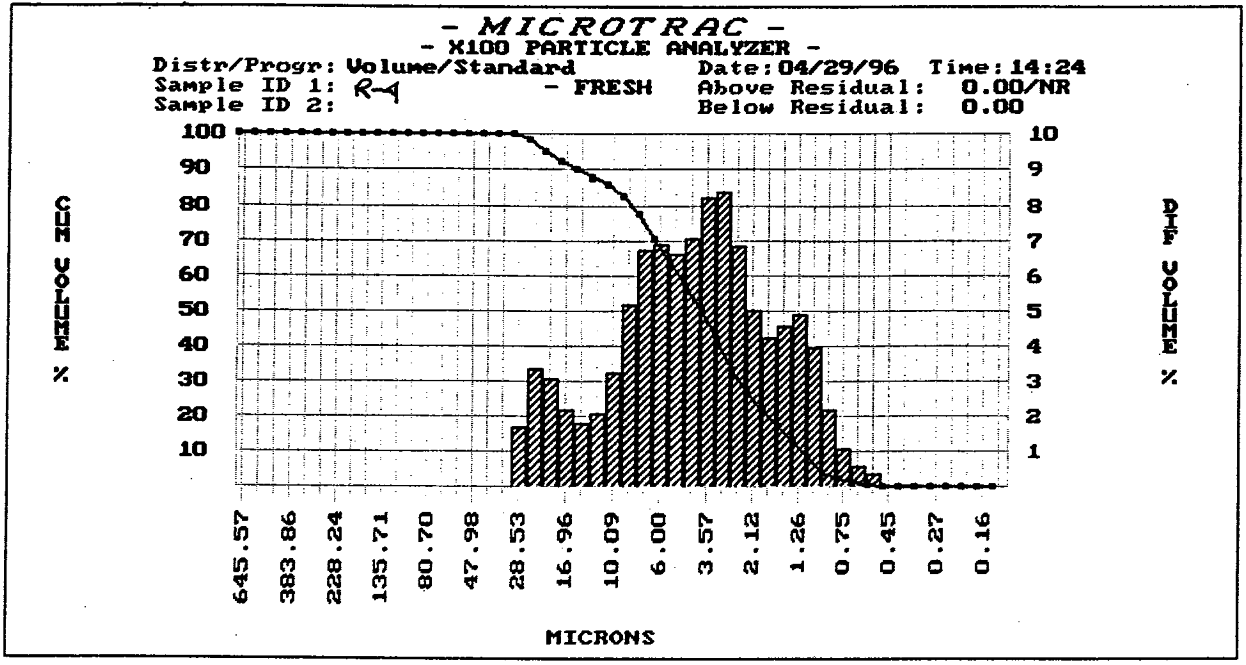
Flow Rate: n/a ml/sec
Ultrasonic Power: n/a watts
Ultrasonic Time: n/a seconds

Id #1: - FRESH
Distrib. Format: Volume
Filter: On
Run Time: 60 seconds
Run Number: 1 of 1 runs
Transmission: 0.81
Laser Int: 1.020/1.005/1.005
Residuals: Disabled
Above Residual: 0.00
Below Residual: 0.00

Summary Data
dv = 0.1291
10% = 1.24
50% = 3.88
90% = 15.87
mv = 6.15
mn = 1.13
ma = 2.83
cs = 2.117
sd = 4.14

Id #2:
Date: 04/29/96 Time: 14:24
Chan. Progression: Standard
Upper Channel Edge: 704.00
Lower Channel Edge: 0.12
Number of Channels: 50
Fluid Refractive Index: n/a
Transparent Particles: No
Spherical Particles: n/a
Part. Refractive Index: n/a

ch top	%pass	%-chn	ch top	%pass	%-chn	ch top	%pass	%-chn	ch top	%pass	%-chn
704.00	100.00	0.00	9.25	82.71	5.18						
591.99	100.00	0.00	7.78	77.53	6.75						
497.80	100.00	0.00	6.54	70.78	6.91						
418.60	100.00	0.00	5.50	63.87	6.63						
352.00	100.00	0.00	4.62	57.24	7.08						
296.00	100.00	0.00	3.89	50.16	8.20						
248.90	100.00	0.00	3.27	41.96	8.41						
209.30	100.00	0.00	2.75	33.55	6.85						
176.00	100.00	0.00	2.31	26.70	4.99						
148.00	100.00	0.00	1.94	21.71	4.23						
124.45	100.00	0.00	1.64	17.48	4.53						
104.65	100.00	0.00	1.38	12.95	4.88						
88.00	100.00	0.00	1.16	8.07	3.92						
74.00	100.00	0.00	0.97	4.15	2.19						
62.23	100.00	0.00	0.82	1.96	1.05						
52.33	100.00	0.00	0.69	0.91	0.56						
44.00	100.00	0.00	0.58	0.35	0.35						
37.00	100.00	0.00	0.49	0.00	0.00						
31.11	100.00	1.64	0.41	0.00	0.00						
26.16	98.36	3.32	0.34	0.00	0.00						
22.00	95.04	3.07	0.29	0.00	0.00						
18.50	91.97	2.19	0.24	0.00	0.00						
15.56	89.78	1.78	0.20	0.00	0.00						
13.08	88.00	2.07	0.17	0.00	0.00						
11.00	85.93	3.22	0.14	0.00	0.00						



- MICROTRAC -
- X100 PARTICLE ANALYZER -
Distr/Progr: Volume/Standard Date: 04/29/96 Time: 14:24
Sample ID 1: R-4 - FRESH Above Residual: 0.00/NR
Sample ID 2: Below Residual: 0.00

- MICROTRAC X100 PARTICLE ANALYZER -
Percent Passing Data
Version 4.13

R-4 - Fresh Sludge

Sample 1

ASVR Parameters

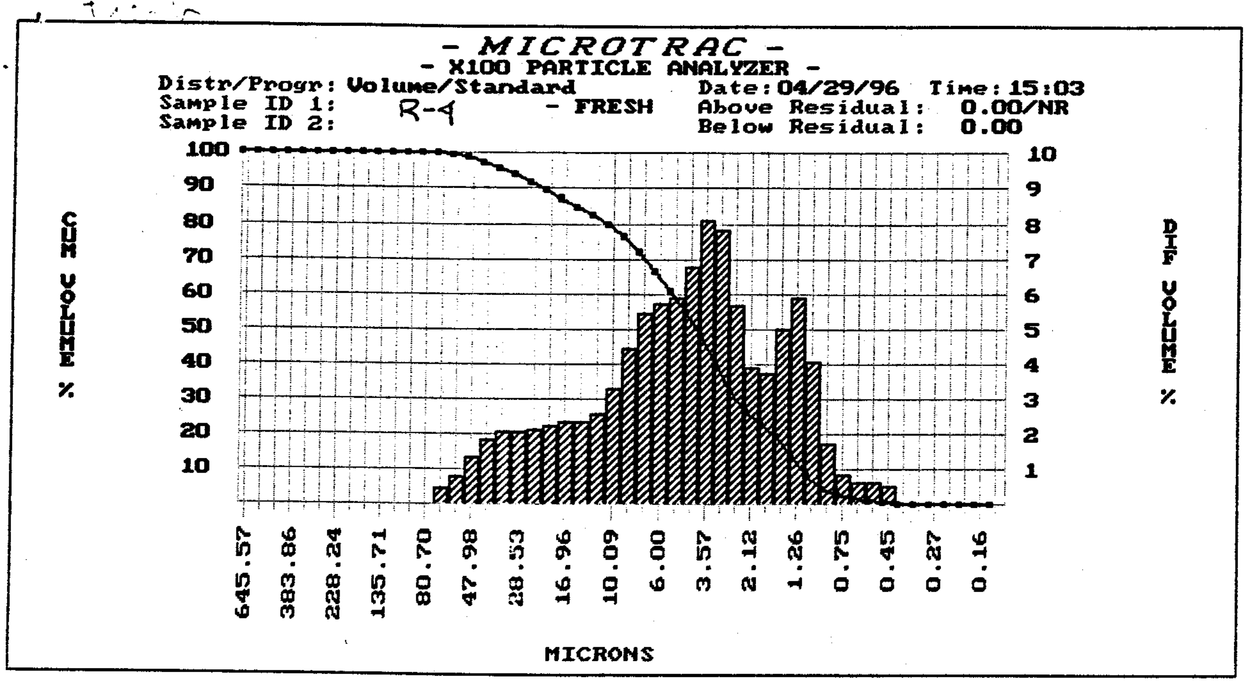
Flow Rate: n/a ml/sec
Ultrasonic Power: n/a watts
Ultrasonic Time: n/a seconds

Id #1: - FRESH
Distrib. Format: Volume
Filter: On
Run Time: 60 seconds
Run Number: 1 of 1 runs
Transmission: 0.69
Laser Int: 1.020/1.005/1.005
Residuals: Disabled
Above Residual: 0.00
Below Residual: 0.00

Summary Data
dv = 0.2796
10% = 1.22
50% = 4.04
90% = 22.96
mv = 8.43
mn = 0.945
ma = 2.83
cs = 2.122
sd = 6.55

Id #2:
Date: 04/29/96 Time: 15:03
Chan. Progression: Standard
Upper Channel Edge: 704.00
Lower Channel Edge: 0.12
Number of Channels: 50
Fluid Refractive Index: n/a
Transparent Particles: No
Spherical Particles: n/a
Part. Refractive Index: n/a

ch top	%pass	%-chn
704.00	100.00	0.00
591.99	100.00	0.00
497.80	100.00	0.00
418.60	100.00	0.00
352.00	100.00	0.00
296.00	100.00	0.00
248.90	100.00	0.00
209.30	100.00	0.00
176.00	100.00	0.00
148.00	100.00	0.00
124.45	100.00	0.00
104.65	100.00	0.00
88.00	100.00	0.00
74.00	100.00	0.43
62.23	99.57	0.78
52.33	98.79	1.32
44.00	97.47	1.82
37.00	95.65	2.03
31.11	93.62	2.04
26.16	91.58	2.11
22.00	89.47	2.24
18.50	87.23	2.33
15.56	84.90	2.35
13.08	82.55	2.57
11.00	79.98	3.26
9.25	76.72	4.44
7.78	72.28	5.46
6.54	66.82	5.73
5.50	61.09	5.88
4.62	55.21	6.76
3.89	48.45	8.10
3.27	40.35	7.86
2.75	32.49	5.64
2.31	26.85	3.90
1.94	22.95	3.74
1.64	19.21	5.00
1.38	14.21	5.91
1.16	8.30	4.03
0.97	4.27	1.75
0.82	2.52	0.82
0.69	1.70	0.60
0.58	1.10	0.59
0.49	0.51	0.51
0.41	0.00	0.00
0.34	0.00	0.00
0.29	0.00	0.00
0.24	0.00	0.00
0.20	0.00	0.00
0.17	0.00	0.00
0.14	0.00	0.00



- *MICROTRAC* -

- X100 PARTICLE ANALYZER -

Distr/Progr: Volume/Standard   Date: 04/29/96   Time: 15:03
Sample ID 1: R-4   - FRESH   Above Residual: 0.00/NR
Sample ID 2:   Below Residual: 0.00

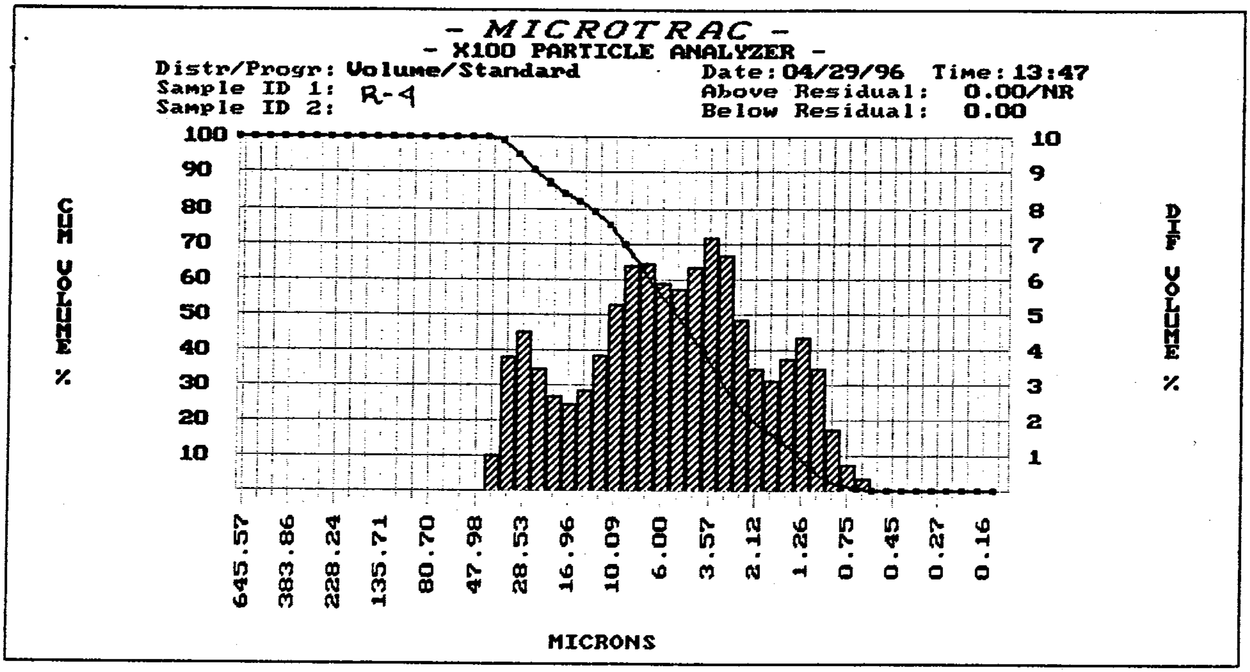
```
                  - MICROTRAC X100 PARTICLE ANALYZER -
                          Percent Passing Data
                              Version 4.13

     R-4    - Aged Sludge                        ASVR Parameters

  Sample 1                                 Flow Rate: n/a ml/sec
                                           Ultrasonic Power: n/a watts
                                           Ultrasonic Time: n/a seconds

 Id #1:                       Summary Data  Id #2:
 Distrib. Format: Volume      dv  = 0.1548  Date: 04/29/96   Time: 13:47
 Filter: On                   10% =   1.35  Chan. Progression: Standard
 Run Time:  60 seconds        50% =   5.25  Upper Channel Edge: 704.00
 Run Number:    1 of   1 runs 90% =  25.30  Lower Channel Edge:    0.12
 Transmission: 0.81           mv  =   8.92  Number of Channels:  50
 Laser Int: 1.020/1.005/1.005 mn  =   1.25  Fluid Refractive Index: n/a
 Residuals: Disabled          ma  =   3.45  Transparent Particles: No
 Above Residual:    0.00      cs  =  1.741  Spherical Particles: n/a
 Below Residual:    0.00      sd  =   7.94  Part. Refractive Index: n/a

ch top  %pass  %-chn ch top  %pass  %-chn ch top  %pass %-chn ch top  %pass %-chn
704.00 100.00  0.00    9.25  70.24  6.39
591.99 100.00  0.00    7.78  63.85  6.45
497.80 100.00  0.00    6.54  57.40  5.88
418.60 100.00  0.00    5.50  51.52  5.71
352.00 100.00  0.00    4.62  45.81  6.36
296.00 100.00  0.00    3.89  39.45  7.17
248.90 100.00  0.00    3.27  32.28  6.68
209.30 100.00  0.00    2.75  25.60  4.85
176.00 100.00  0.00    2.31  20.75  3.42
148.00 100.00  0.00    1.94  17.33  3.10
124.45 100.00  0.00    1.64  14.23  3.73
104.65 100.00  0.00    1.38  10.50  4.35
 88.00 100.00  0.00    1.16   6.15  3.42
 74.00 100.00  0.00    0.97   2.73  1.70
 62.23 100.00  0.00    0.82   1.03  0.70
 52.33 100.00  0.00    0.69   0.33  0.33
 44.00 100.00  0.99    0.58   0.00  0.00
 37.00  99.01  3.78    0.49   0.00  0.00
 31.11  95.23  4.48    0.41   0.00  0.00
 26.16  90.75  3.45    0.34   0.00  0.00
 22.00  87.30  2.65    0.29   0.00  0.00
 18.50  84.65  2.47    0.24   0.00  0.00
 15.56  82.18  2.85    0.20   0.00  0.00
 13.08  79.33  3.83    0.17   0.00  0.00
 11.00  75.50  5.26    0.14   0.00  0.00
```



- MICROTRAC -

- X100 PARTICLE ANALYZER -

Distr/Progr: Volume/Standard   Date: 04/29/96   Time: 13:47
Sample ID 1: R-4   Above Residual: 0.00/NR
Sample ID 2:   Below Residual: 0.00

- MICROTRAC X100 PARTICLE ANALYZER -
Percent Passing Data
Version 4.13

R-4 - Aged Sludge

Sample 1

ASVR Parameters

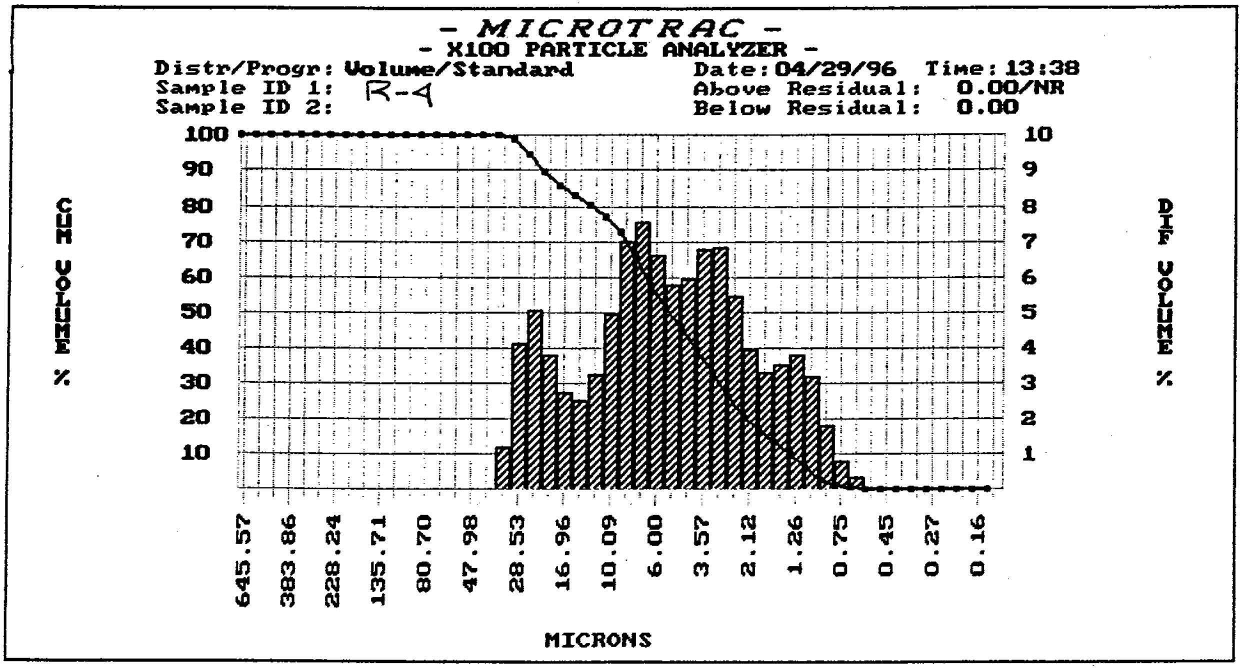
Flow Rate: n/a ml/sec
Ultrasonic Power: n/a watts
Ultrasonic Time: n/a seconds

Id #1:
Distrib. Format: Volume
Filter: On
Run Time: 60 seconds
Run Number: 1 of 1 runs
Transmission: 0.87
Laser Int: 1.020/1.005/1.005
Residuals: Disabled
Above Residual: 0.00
Below Residual: 0.00

Summary Data
dv = 0.0969
10% = 1.39
50% = 5.29
90% = 22.27
mv = 8.19
mn = 1.24
ma = 3.43
cs = 1.749
sd = 7.30

Id #2:
Date: 04/29/96 Time: 13:38
Chan. Progression: Standard
Upper Channel Edge: 704.00
Lower Channel Edge: 0.12
Number of Channels: 50
Fluid Refractive Index: n/a
Transparent Particles: No
Spherical Particles: n/a
Part. Refractive Index: n/a

ch top	%pass	%-chn
704.00	100.00	0.00
591.99	100.00	0.00
497.80	100.00	0.00
418.60	100.00	0.00
352.00	100.00	0.00
296.00	100.00	0.00
248.90	100.00	0.00
209.30	100.00	0.00
176.00	100.00	0.00
148.00	100.00	0.00
124.45	100.00	0.00
104.65	100.00	0.00
88.00	100.00	0.00
74.00	100.00	0.00
62.23	100.00	0.00
52.33	100.00	0.00
44.00	100.00	0.00
37.00	100.00	1.17
31.11	98.83	4.12
26.16	94.71	5.05
22.00	89.66	3.78
18.50	85.88	2.71
15.56	83.17	2.49
13.08	80.68	3.20
11.00	77.48	4.95
9.25	72.53	7.01
7.78	65.52	7.56
6.54	57.96	6.59
5.50	51.37	5.77
4.62	45.60	5.96
3.89	39.64	6.78
3.27	32.86	6.82
2.75	26.04	5.44
2.31	20.60	3.92
1.94	16.68	3.30
1.64	13.38	3.49
1.38	9.89	3.80
1.16	6.09	3.15
0.97	2.94	1.78
0.82	1.16	0.80
0.69	0.36	0.36
0.58	0.00	0.00
0.49	0.00	0.00
0.41	0.00	0.00
0.34	0.00	0.00
0.29	0.00	0.00
0.24	0.00	0.00
0.20	0.00	0.00
0.17	0.00	0.00
0.14	0.00	0.00



- MICROTRAC -

- X100 PARTICLE ANALYZER -

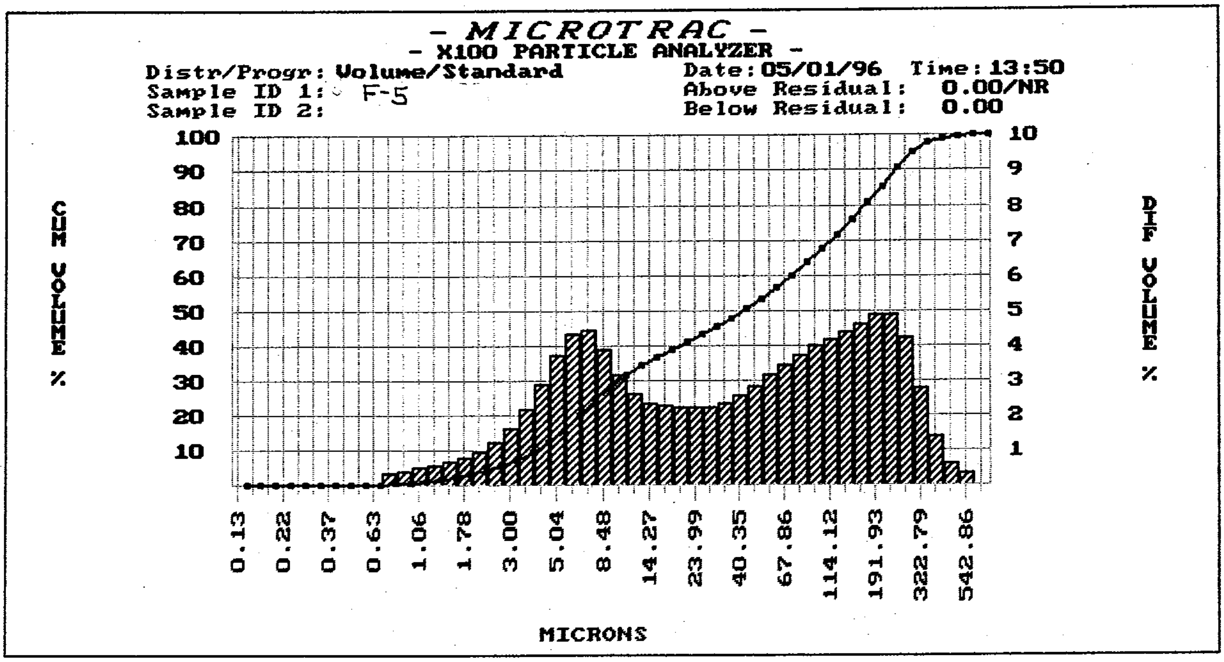
Distr/Progr: Volume/Standard
Date: 04/29/96 Time: 13:38
Sample ID 1: R-4
Above Residual: 0.00/NR
Sample ID 2:
Below Residual: 0.00

```
                 - MICROTRAC X100 PARTICLE ANALYZER -
                         Percent Passing Data
                             Version 4.13

  F-5    Treatment plant                            ASVR Parameters
 Fresh sludge
                                                Flow Rate: n/a ml/sec
                                                Ultrasonic Power: n/a watts
                                                Ultrasonic Time: n/a seconds

 Id #1:                           Summary Data    Id #2:
 Distrib. Format: Volume          dv  = 0.0820    Date: 05/01/96   Time: 13:50
 Filter: On                       10% =   4.10    Chan. Progression: Standard
 Run Time:  60 seconds            50% =  42.53    Upper Channel Edge: 704.00
 Run Number:   1 of  1 runs       90% = 242.80    Lower Channel Edge:   0.12
 Transmission: 0.95               mv  =  87.61    Number of Channels:  50
 Laser Int: 1.021/1.006/1.006     mn  =   1.39    Fluid Refractive Index: n/a
 Residuals: Disabled              ma  =  10.34    Transparent Particles: No
 Above Residual:    0.00          cs  =  0.580    Spherical Particles: n/a
 Below Residual:    0.00          sd  =  95.56    Part. Refractive Index: n/a

ch top  %pass %-chn|ch top   %pass %-chn|ch top   %pass %-chn|ch top   %pass %-chn
704.00 100.00  0.00|  9.25   28.51  3.90|                    |
591.99 100.00  0.34|  7.78   24.61  4.43|                    |
497.80  99.66  0.63|  6.54   20.18  4.36|                    |
418.60  99.03  1.38|  5.50   15.82  3.72|                    |
352.00  97.65  2.75|  4.62   12.10  2.89|                    |
296.00  94.90  4.21|  3.89    9.21  2.17|                    |
248.90  90.69  4.91|  3.27    7.04  1.62|                    |
209.30  85.78  4.88|  2.75    5.42  1.22|                    |
176.00  80.90  4.63|  2.31    4.20  0.95|                    |
148.00  76.27  4.41|  1.94    3.25  0.77|                    |
124.45  71.86  4.19|  1.64    2.48  0.66|                    |
104.65  67.67  3.98|  1.38    1.82  0.58|                    |
 88.00  63.69  3.72|  1.16    1.24  0.51|                    |
 74.00  59.97  3.46|  0.97    0.73  0.41|                    |
 62.23  56.51  3.15|  0.82    0.32  0.32|                    |
 52.33  53.36  2.84|  0.69    0.00  0.00|                    |
 44.00  50.52  2.54|  0.58    0.00  0.00|                    |
 37.00  47.98  2.33|  0.49    0.00  0.00|                    |
 31.11  45.65  2.23|  0.41    0.00  0.00|                    |
 26.16  43.42  2.23|  0.34    0.00  0.00|                    |
 22.00  41.19  2.24|  0.29    0.00  0.00|                    |
 18.50  38.95  2.28|  0.24    0.00  0.00|                    |
 15.56  36.67  2.36|  0.20    0.00  0.00|                    |
 13.08  34.31  2.63|  0.17    0.00  0.00|                    |
 11.00  31.68  3.17|  0.14    0.00  0.00|                    |
```



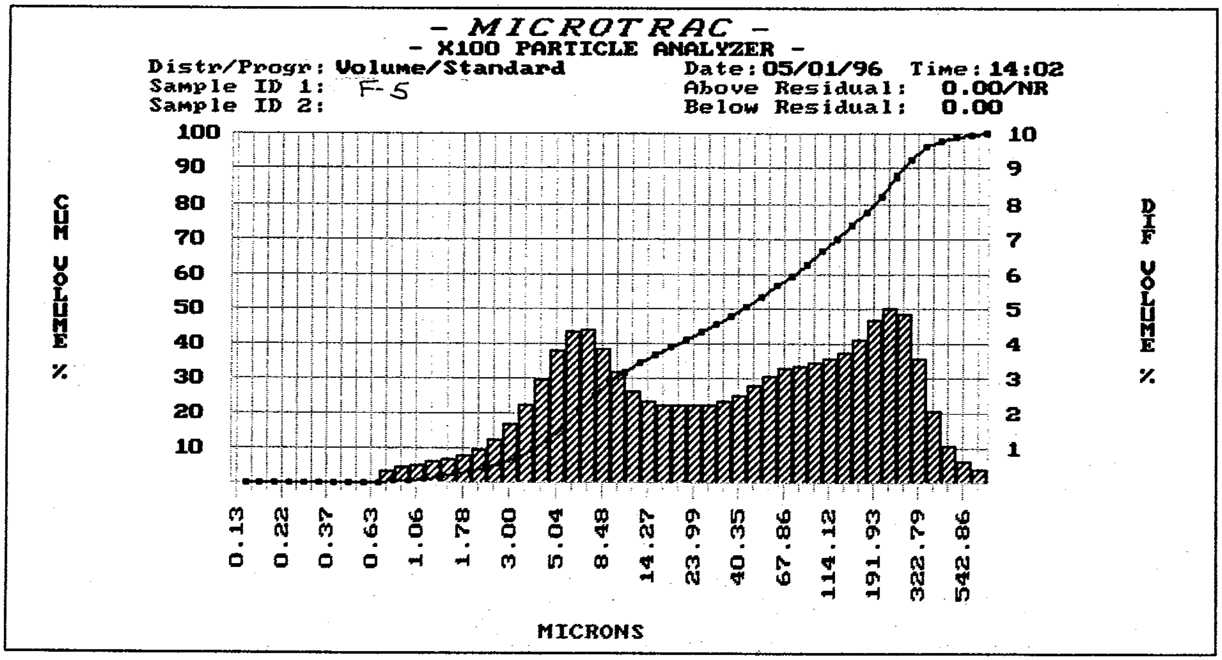
- MICROTRAC -

- X100 PARTICLE ANALYZER -

Distr/Progr: Volume/Standard Date: 05/01/96 Time: 13:50

Sample ID 1: F-5 Above Residual: 0.00/NR

Sample ID 2: Below Residual: 0.00



- MICROTRAC -

- X100 PARTICLE ANALYZER -

Distr/Progr: Volume/Standard Date: 05/01/96 Time: 14:02
Sample ID 1: F-5 Above Residual: 0.00/NR
Sample ID 2: Below Residual: 0.00

- MICROTRAC X100 PARTICLE ANALYZER -
Percent Passing Data
Version 4.13

F-5 Treatment plant
Fresh sludge

ASVR Parameters

Flow Rate: n/a ml/sec
Ultrasonic Power: n/a watts
Ultrasonic Time: n/a seconds

Id #1:
Distrib. Format: Volume
Filter: On
Run Time: 60 seconds
Run Number: 1 of 1 runs
Transmission: 0.96
Laser Int: 1.021/1.005/1.006
Residuals: Disabled
Above Residual: 0.00
Below Residual: 0.00

Summary Data
dv = 0.0738
10% = 4.03
50% = 42.37
90% = 271.24
mv = 96.60
mn = 1.38
ma = 10.25
cs = 0.586
sd = 107.57

Id #2:
Date: 05/01/96 Time: 14:02
Chan. Progression: Standard
Upper Channel Edge: 704.00
Lower Channel Edge: 0.12
Number of Channels: 50
Fluid Refractive Index: n/a
Transparent Particles: No
Spherical Particles: n/a
Part. Refractive Index: n/a

ch top	%pass	%-chn	ch top	%pass	%-chn	ch top	%pass	%-chn	ch top	%pass	%-chn
704.00	100.00	0.40	9.25	28.74	3.85						
591.99	99.60	0.59	7.78	24.89	4.37						
497.80	99.01	1.06	6.54	20.52	4.35						
418.60	97.95	2.06	5.50	16.17	3.77						
352.00	95.89	3.56	4.62	12.40	2.97						
296.00	92.33	4.81	3.89	9.43	2.24						
248.90	87.52	5.02	3.27	7.19	1.66						
209.30	82.50	4.64	2.75	5.53	1.25						
176.00	77.86	4.11	2.31	4.28	0.97						
148.00	73.75	3.75	1.94	3.31	0.79						
124.45	70.00	3.53	1.64	2.52	0.67						
104.65	66.47	3.44	1.38	1.85	0.59						
88.00	63.03	3.36	1.16	1.26	0.51						
74.00	59.67	3.26	0.97	0.75	0.42						
62.23	56.41	3.05	0.82	0.33	0.33						
52.33	53.36	2.79	0.69	0.00	0.00						
44.00	50.57	2.52	0.58	0.00	0.00						
37.00	48.05	2.33	0.49	0.00	0.00						
31.11	45.72	2.22	0.41	0.00	0.00						
26.16	43.50	2.20	0.34	0.00	0.00						
22.00	41.30	2.20	0.29	0.00	0.00						
18.50	39.10	2.23	0.24	0.00	0.00						
15.56	36.87	2.34	0.20	0.00	0.00						
13.08	34.53	2.63	0.17	0.00	0.00						
11.00	31.90	3.16	0.14	0.00	0.00						

```
              - MICROTRAC X100 PARTICLE ANALYZER -
                      Percent Passing Data
                          Version 4.13

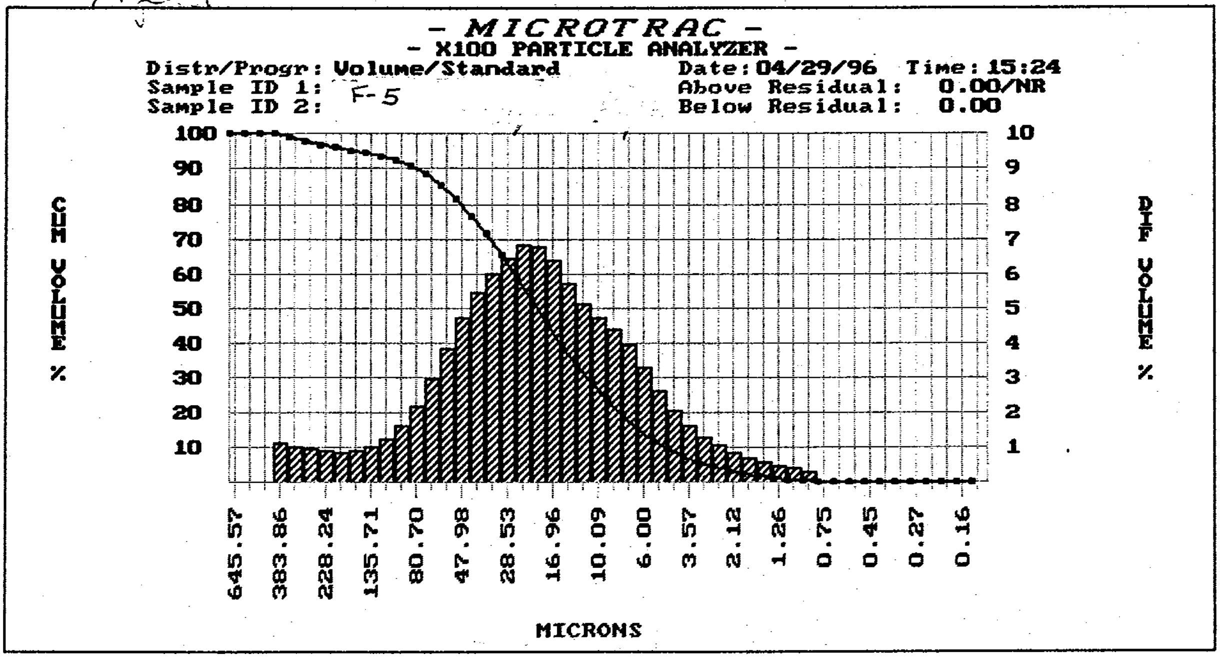
  F-5 Aged Sludge                          ASVR Parameters

  Sample 1                          Flow Rate: n/a ml/sec
                                    Ultrasonic Power: n/a watts
                                    Ultrasonic Time: n/a seconds

 Id #1:                        Summary Data      Id #2:
 Distrib. Format: Volume       dv  = 0.2151      Date: 04/29/96   Time: 15:24
 Filter: On                    10% =   4.89      Chan. Progression: Standard
 Run Time:  60 seconds         50% =  20.83      Upper Channel Edge: 704.00
 Run Number:   1 of  1 runs    90% =  84.16      Lower Channel Edge:   0.12
 Transmission: 0.89            mv  =  40.89      Number of Channels:  50
 Laser Int: 1.020/1.005/1.005  mn  =   1.71      Fluid Refractive Index: n/a
 Residuals: Disabled           ma  =  10.83      Transparent Particles: No
 Above Residual:   0.00        cs  =  0.554      Spherical Particles: n/a
 Below Residual:   0.00        sd  =  25.62      Part. Refractive Index: n/a

ch top  %pass %-chn|ch top  %pass %-chn|ch top  %pass %-chn|ch top  %pass %-chn
704.00 100.00  0.00|  9.25  23.42  4.38|                   |
591.99 100.00  0.00|  7.78  19.04  3.92|                   |
497.80 100.00  0.00|  6.54  15.12  3.29|                   |
418.60 100.00  1.09|  5.50  11.83  2.62|                   |
352.00  98.91  1.02|  4.62   9.21  2.05|                   |
296.00  97.89  0.95|  3.89   7.16  1.62|                   |
248.90  96.94  0.88|  3.27   5.54  1.30|                   |
209.30  96.06  0.86|  2.75   4.24  1.04|                   |
176.00  95.20  0.89|  2.31   3.20  0.83|                   |
148.00  94.31  1.01|  1.94   2.37  0.67|                   |
124.45  93.30  1.22|  1.64   1.70  0.56|                   |
104.65  92.08  1.59|  1.38   1.14  0.46|                   |
 88.00  90.49  2.14|  1.16   0.68  0.38|                   |
 74.00  88.35  2.92|  0.97   0.30  0.30|                   |
 62.23  85.43  3.82|  0.82   0.00  0.00|                   |
 52.33  81.61  4.72|  0.69   0.00  0.00|                   |
 44.00  76.89  5.43|  0.58   0.00  0.00|                   |
 37.00  71.46  6.01|  0.49   0.00  0.00|                   |
 31.11  65.45  6.47|  0.41   0.00  0.00|                   |
 26.16  58.98  6.81|  0.34   0.00  0.00|                   |
 22.00  52.17  6.80|  0.29   0.00  0.00|                   |
 18.50  45.37  6.40|  0.24   0.00  0.00|                   |
 15.56  38.97  5.72|  0.20   0.00  0.00|                   |
 13.08  33.25  5.12|  0.17   0.00  0.00|                   |
 11.00  28.13  4.71|  0.14   0.00  0.00|                   |
```

Acid



- MICROTRAC -
- X100 PARTICLE ANALYZER -

Distr/Progr: Volume/Standard
Sample ID 1: F-5
Sample ID 2:

Date: 04/29/96 Time: 15:24
Above Residual: 0.00/NR
Below Residual: 0.00

```
                    - MICROTRAC X100 PARTICLE ANALYZER -
                            Percent Passing Data
                                Version 4.13

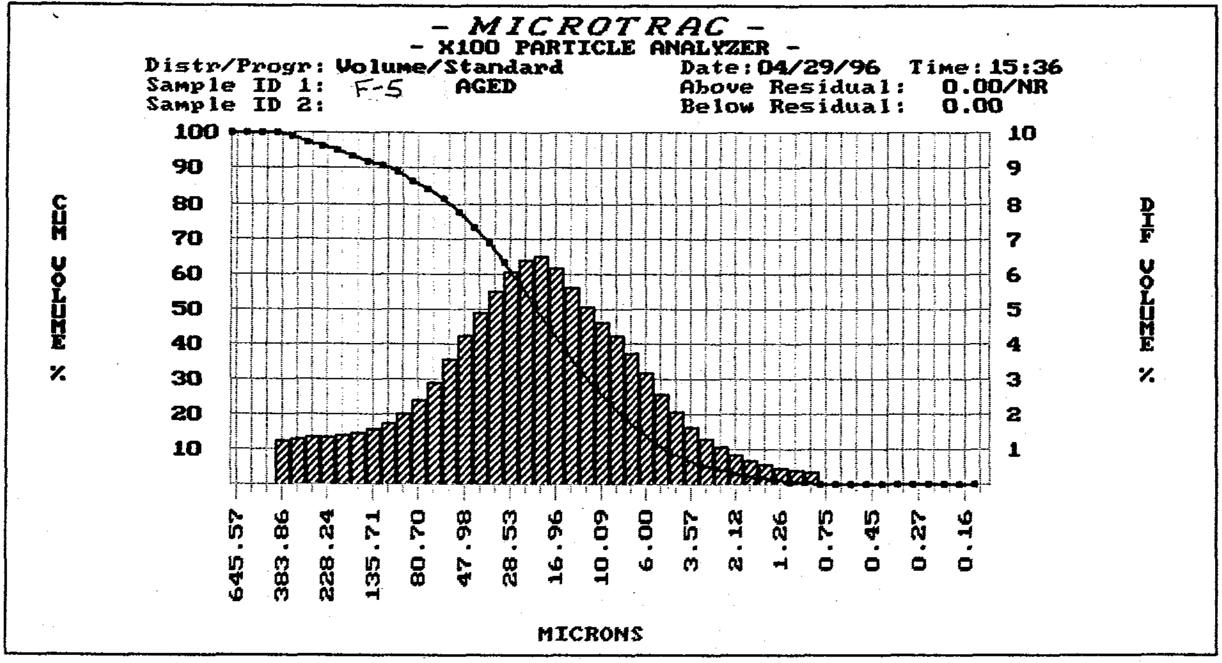
  F-5    Aged Composite                              ASVR Parameters

 Sample 1                                   Flow Rate: n/a ml/sec
                                            Ultrasonic Power: n/a watts
                                            Ultrasonic Time: n/a seconds

 Id #1:                         Summary Data    Id #2:
 Distrib. Format: Volume        dv  = 0.2002    Date: 04/29/96   Time: 15:36
 Filter: On                     10% =   4.92    Chan. Progression: Standard
 Run Time:  60 seconds          50% =  21.57    Upper Channel Edge: 704.00
 Run Number:   1 of  1 runs     90% = 119.44    Lower Channel Edge:   0.12
 Transmission: 0.90             mv  =  47.13    Number of Channels:  50
 Laser Int: 1.020/1.005/1.005   mn  =   1.70    Fluid Refractive Index: n/a
 Residuals: Disabled            ma  =  11.06    Transparent Particles: No
 Above Residual:    0.00        cs  =  0.542    Spherical Particles: n/a
 Below Residual:    0.00        sd  =  32.84    Part. Refractive Index: n/a

ch top  %pass  %-chn ch top   %pass  %-chn ch top   %pass %-chn ch top   %pass %-chn
704.00 100.00   0.00    9.25  22.77   4.20
591.99 100.00   0.00    7.78  18.57   3.72
497.80 100.00   0.00    6.54  14.85   3.14
418.60 100.00   1.25    5.50  11.71   2.55
352.00  98.75   1.30    4.62   9.16   2.03
296.00  97.45   1.34    3.89   7.13   1.61
248.90  96.11   1.35    3.27   5.52   1.29
209.30  94.76   1.38    2.75   4.23   1.03
176.00  93.38   1.44    2.31   3.20   0.83
148.00  91.94   1.55    1.94   2.37   0.67
124.45  90.39   1.72    1.64   1.70   0.55
104.65  88.67   1.99    1.38   1.15   0.46
 88.00  86.68   2.38    1.16   0.69   0.38
 74.00  84.30   2.91    0.97   0.31   0.31
 62.23  81.39   3.54    0.82   0.00   0.00
 52.33  77.85   4.24    0.69   0.00   0.00
 44.00  73.61   4.90    0.58   0.00   0.00
 37.00  68.71   5.52    0.49   0.00   0.00
 31.11  63.19   6.03    0.41   0.00   0.00
 26.16  57.16   6.41    0.34   0.00   0.00
 22.00  50.75   6.48    0.29   0.00   0.00
 18.50  44.27   6.17    0.24   0.00   0.00
 15.56  38.10   5.63    0.20   0.00   0.00
 13.08  32.47   5.08    0.17   0.00   0.00
 11.00  27.39   4.62    0.14   0.00   0.00
```

Aged



- *MICROTRAC* -
- X100 PARTICLE ANALYZER -

Distr/Progr: Volume/Standard Date: 04/29/96 Time: 15:36
Sample ID 1: F-5 AGED Above Residual: 0.00/NR
Sample ID 2: Below Residual: 0.00

Run 1

```
                 - MICROTRAC X100 PARTICLE ANALYZER -
                         Percent Passing Data
                             Version 4.13

                                              ASVR Parameters
   S-6 FRESH
                                            Flow Rate: n/a ml/sec
                                            Ultrasonic Power: n/a watts
                                            Ultrasonic Time: n/a seconds

 Id #1:                       Summary Data   Id #2:
 Distrib. Format: Volume      dv  = 0.0298   Date: 06/19/96   Time:  9:04
 Filter: On                   10% =   1.77   Chan. Progression: Standard
 Run Time:  60 seconds        50% =   6.67   Upper Channel Edge: 704.00
 Run Number:   2 of  3 runs   80% =  12.98   Lower Channel Edge:   0.12
 Transmission: 0.96           mv  =   7.93   Number of Channels:  50
 Laser Int: 1.021/1.003/1.006 mn  =   1.17   Fluid Refractive Index: n/a
 Residuals: Disabled          ma  =   4.14   Transparent Particles: No
 Above Residual:   0.00       cs  =  1.448   Spherical Particles: n/a
 Below Residual:   0.00       sd  =   5.76   Part. Refractive Index: n/a

ch top  %pass %-chn|ch top  %pass %-chn|ch top  %pass %-chn|ch top  %pass %-chn
704.00 100.00  0.00|  9.25  65.89  7.91|                   |
591.99 100.00  0.00|  7.78  57.98  9.04|                   |
497.80 100.00  0.00|  6.54  48.94  9.18|                   |
418.60 100.00  0.00|  5.50  39.76  7.91|                   |
352.00 100.00  0.00|  4.62  31.85  6.22|                   |
296.00 100.00  0.00|  3.89  25.63  4.84|                   |
248.90 100.00  0.00|  3.27  20.79  3.86|                   |
209.30 100.00  0.00|  2.75  16.93  3.12|                   |
176.00 100.00  0.00|  2.31  13.81  2.58|                   |
148.00 100.00  0.00|  1.94  11.23  2.27|                   |
124.45 100.00  0.00|  1.64   8.96  2.15|                   |
104.65 100.00  0.00|  1.38   6.81  2.09|                   |
 88.00 100.00  0.00|  1.16   4.72  1.87|                   |
 74.00 100.00  0.00|  0.97   2.85  1.42|                   |
 62.23 100.00  0.00|  0.82   1.43  0.91|                   |
 52.33 100.00  0.00|  0.69   0.52  0.52|                   |
 44.00 100.00  0.00|  0.58   0.00  0.00|                   |
 37.00 100.00  0.00|  0.49   0.00  0.00|                   |
 31.11 100.00  0.00|  0.41   0.00  0.00|                   |
 26.16 100.00  0.41|  0.34   0.00  0.00|                   |
 22.00  99.59  3.29|  0.29   0.00  0.00|                   |
 18.50  96.30  7.58|  0.24   0.00  0.00|                   |
 15.56  88.72  8.37|  0.20   0.00  0.00|                   |
 13.08  80.35  7.37|  0.17   0.00  0.00|                   |
 11.00  72.98  7.09|  0.14   0.00  0.00|                   |
```

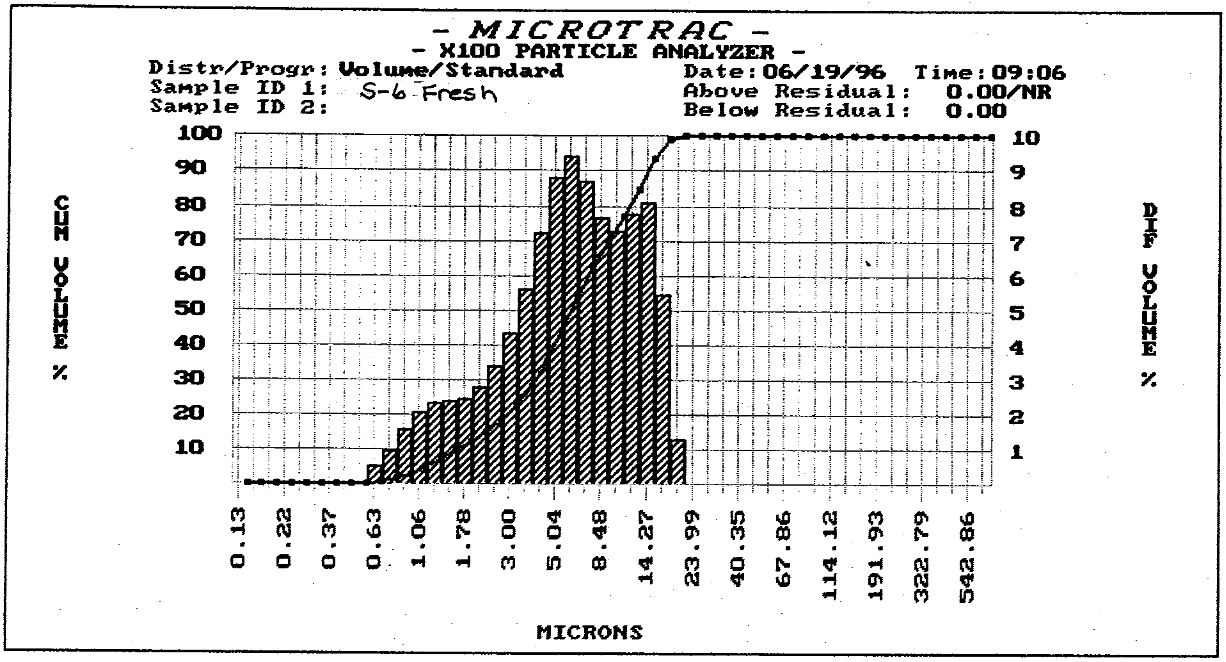
Run 2

```
                 - MICROTRAC X100 PARTICLE ANALYZER -
                        Percent Passing Data
                            Version 4.13

 S-6                                       ASVR Parameters
 fresh sludge 96
                                   Flow Rate: n/a ml/sec
                                   Ultrasonic Power: n/a watts
                                   Ultrasonic Time: n/a seconds

 Id #1:                       Summary Data      Id #2:
 Distrib. Format: Volume      dv  = 0.0274      Date: 06/19/96   Time:  9:06
 Filter: On                   10% =   1.67      Chan. Progression: Standard
 Run Time:  60 seconds        50% =   6.11      Upper Channel Edge: 704.00
 Run Number:  3 of  3 runs    80% =  11.68      Lower Channel Edge:   0.12
 Transmission: 0.96           mv  =   7.21      Number of Channels:  50
 Laser Int: 1.021/1.003/1.006 mn  =   1.18      Fluid Refractive Index: n/a
 Residuals: Disabled          ma  =   3.90      Transparent Particles: No
 Above Residual:   0.00       cs  =  1.539      Spherical Particles: n/a
 Below Residual:   0.00       sd  =   5.16      Part. Refractive Index: n/a

ch top  %pass %-chn|ch top  %pass %-chn|ch top  %pass %-chn|ch top  %pass %-chn
704.00 100.00  0.00|  9.25  70.15  7.69|                   |
591.99 100.00  0.00|  7.78  62.46  8.74|                   |
497.80 100.00  0.00|  6.54  53.72  9.38|                   |
418.60 100.00  0.00|  5.50  44.34  8.78|                   |
352.00 100.00  0.00|  4.62  35.56  7.24|                   |
296.00 100.00  0.00|  3.89  28.32  5.62|                   |
248.90 100.00  0.00|  3.27  22.70  4.32|                   |
209.30 100.00  0.00|  2.75  18.38  3.38|                   |
176.00 100.00  0.00|  2.31  15.00  2.76|                   |
148.00 100.00  0.00|  1.94  12.24  2.46|                   |
124.45 100.00  0.00|  1.64   9.78  2.38|                   |
104.65 100.00  0.00|  1.38   7.40  2.35|                   |
 88.00 100.00  0.00|  1.16   5.05  2.08|                   |
 74.00 100.00  0.00|  0.97   2.97  1.53|                   |
 62.23 100.00  0.00|  0.82   1.44  0.94|                   |
 52.33 100.00  0.00|  0.69   0.50  0.50|                   |
 44.00 100.00  0.00|  0.58   0.00  0.00|                   |
 37.00 100.00  0.00|  0.49   0.00  0.00|                   |
 31.11 100.00  0.00|  0.41   0.00  0.00|                   |
 26.16 100.00  0.00|  0.34   0.00  0.00|                   |
 22.00 100.00  1.25|  0.29   0.00  0.00|                   |
 18.50  98.75  5.45|  0.24   0.00  0.00|                   |
 15.56  93.30  8.12|  0.20   0.00  0.00|                   |
 13.08  85.18  7.77|  0.17   0.00  0.00|                   |
 11.00  77.41  7.26|  0.14   0.00  0.00|                   |
```



- *MICROTRAC* -

- X100 PARTICLE ANALYZER -

Distr/Progr: Volume/Standard
Sample ID 1: S-6 Fresh
Sample ID 2:

Date: 06/19/96 Time: 09:06
Above Residual: 0.00/NR
Below Residual: 0.00

```
                 - MICROTRAC X100 PARTICLE ANALYZER -
                         Percent Passing Data
                             Version 4.13

  S-6                                           ASVR Parameters
 Composite adged sludge
                                           Flow Rate: n/a ml/sec
                                           Ultrasonic Power: n/a watts
                                           Ultrasonic Time: n/a seconds

 Id #1:                         Summary Data     Id #2:
 Distrib. Format: Volume        dv  = 0.0915     Date: 04/30/96   Time:  8:53
 Filter: On                     10% =   5.74     Chan. Progression: Standard
 Run Time:  60 seconds          50% =  22.40     Upper Channel Edge: 704.00
 Run Number:   1 of  1 runs     90% = 197.24     Lower Channel Edge:   0.12
 Transmission: 0.95             mv  =  60.98     Number of Channels:  50
 Laser Int: 1.021/1.006/1.006   mn  =   2.07     Fluid Refractive Index: n/a
 Residuals: Disabled            ma  =  12.68     Transparent Particles: No
 Above Residual:   0.00         cs  =  0.473     Spherical Particles: n/a
 Below Residual:   0.00         sd  =  53.06     Part. Refractive Index: n/a

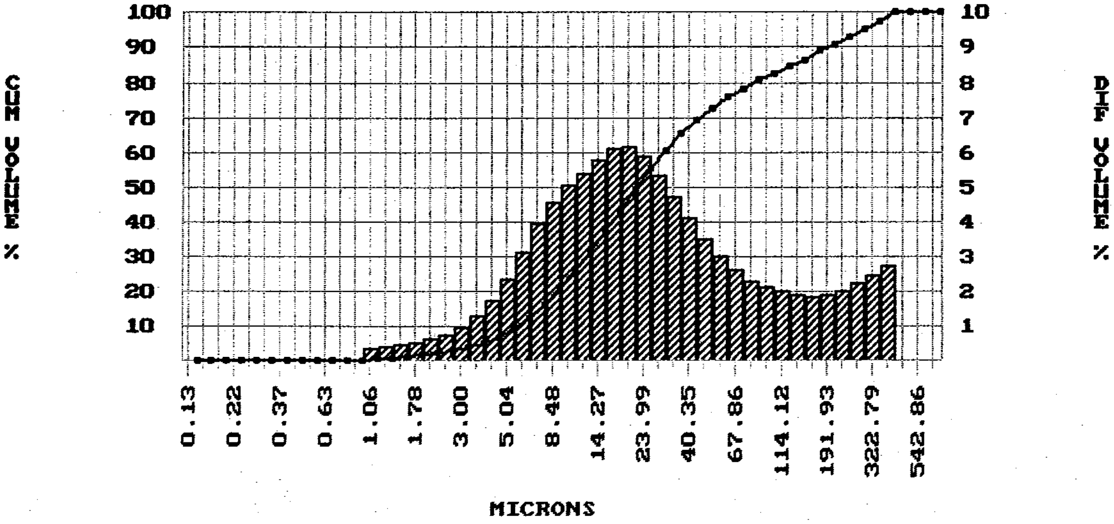
ch top  %pass %-chn|ch top  %pass %-chn|ch top  %pass %-chn|ch top  %pass %-chn
704.00 100.00  0.00|  9.25  20.90  4.57|                   |
591.99 100.00  0.00|  7.78  16.33  3.92|                   |
497.80 100.00  0.00|  6.54  12.41  3.13|                   |
418.60 100.00  2.70|  5.50   9.28  2.35|                   |
352.00  97.30  2.47|  4.62   6.93  1.73|                   |
296.00  94.83  2.20|  3.89   5.20  1.28|                   |
248.90  92.63  1.98|  3.27   3.92  0.96|                   |
209.30  90.65  1.87|  2.75   2.96  0.75|                   |
176.00  88.78  1.85|  2.31   2.21  0.60|                   |
148.00  86.93  1.91|  1.94   1.61  0.49|                   |
124.45  85.02  2.00|  1.64   1.12  0.42|                   |
104.65  83.02  2.13|  1.38   0.70  0.37|                   |
 88.00  80.89  2.30|  1.16   0.33  0.33|                   |
 74.00  78.59  2.59|  0.97   0.00  0.00|                   |
 62.23  76.00  3.00|  0.82   0.00  0.00|                   |
 52.33  73.00  3.52|  0.69   0.00  0.00|                   |
 44.00  69.48  4.10|  0.58   0.00  0.00|                   |
 37.00  65.38  4.74|  0.49   0.00  0.00|                   |
 31.11  60.64  5.36|  0.41   0.00  0.00|                   |
 26.16  55.28  5.91|  0.34   0.00  0.00|                   |
 22.00  49.37  6.18|  0.29   0.00  0.00|                   |
 18.50  43.19  6.09|  0.24   0.00  0.00|                   |
 15.56  37.10  5.77|  0.20   0.00  0.00|                   |
 13.08  31.33  5.40|  0.17   0.00  0.00|                   |
 11.00  25.93  5.03|  0.14   0.00  0.00|                   |
```

[illegible]

- MICROTRAC -

- X100 PARTICLE ANALYZER -

Distr/Progr: Volume/Standard Date: 04/30/96 Time: 08:53
Sample ID 1: 5-6 Above Residual: 0.00/NR
Sample ID 2: Below Residual: 0.00



- MICROTRAC X100 PARTICLE ANALYZER -
Percent Passing Data
Version 4.13

S-6
Composite a ged sludge

ASVR Parameters

Flow Rate: n/a ml/sec
Ultrasonic Power: n/a watts
Ultrasonic Time: n/a seconds

Id #1:
Distrib. Format: Volume
Filter: On
Run Time: 60 seconds
Run Number: 1 of 1 runs
Transmission: 0.96
Laser Int: 1.021/1.006/1.007
Residuals: Disabled
Above Residual: 0.00
Below Residual: 0.00

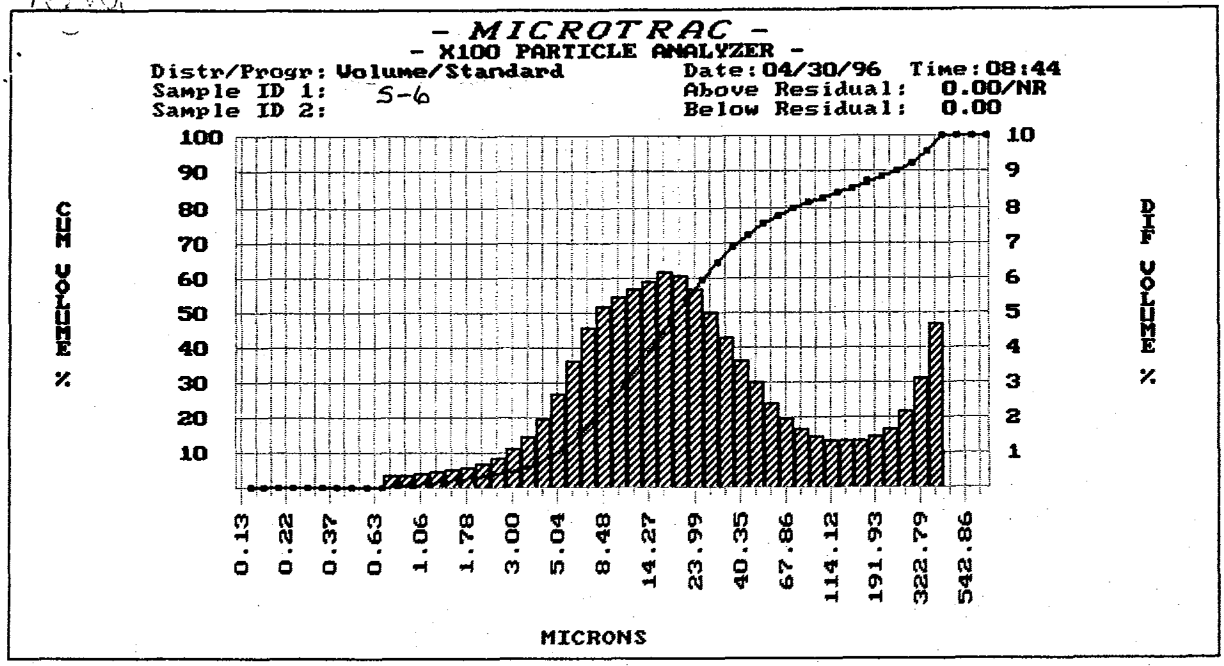
Summary Data
dv = 0.0658
10% = 5.07
50% = 19.67
90% = 247.02
mv = 64.23
mn = 1.37
ma = 10.58
cs = 0.567
sd = 56.60

Id #2:
Date: 04/30/96 Time: 8:44
Chan. Progression: Standard
Upper Channel Edge: 704.00
Lower Channel Edge: 0.12
Number of Channels: 50
Fluid Refractive Index: n/a
Transparent Particles: No
Spherical Particles: n/a
Part. Refractive Index: n/a

ch top	%pass	%-chn
704.00	100.00	0.00
591.99	100.00	0.00
497.80	100.00	0.00
418.60	100.00	4.69
352.00	95.31	3.09
296.00	92.22	2.14
248.90	90.08	1.67
209.30	88.41	1.45
176.00	86.96	1.35
148.00	85.61	1.33
124.45	84.28	1.36
104.65	82.92	1.46
88.00	81.46	1.65
74.00	79.81	1.96
62.23	77.85	2.41
52.33	75.44	2.99
44.00	72.45	3.62
37.00	68.83	4.30
31.11	64.53	4.99
26.16	59.54	5.65
22.00	53.89	6.05
18.50	47.84	6.15
15.56	41.69	5.91
13.08	35.78	5.66
11.00	30.12	5.47
9.25	24.65	5.16
7.78	19.49	4.53
6.54	14.96	3.61
5.50	11.35	2.68
4.62	8.67	1.95
3.89	6.72	1.45
3.27	5.27	1.10
2.75	4.17	0.86
2.31	3.31	0.69
1.94	2.62	0.58
1.64	2.04	0.51
1.38	1.53	0.45
1.16	1.08	0.41
0.97	0.67	0.36
0.82	0.31	0.31
0.69	0.00	0.00
0.58	0.00	0.00
0.49	0.00	0.00
0.41	0.00	0.00
0.34	0.00	0.00
0.29	0.00	0.00
0.24	0.00	0.00
0.20	0.00	0.00
0.17	0.00	0.00
0.14	0.00	0.00

ch top	%pass	%-chn	ch top	%pass	%-chn

A20d



- *MICROTRAC* -

- X100 PARTICLE ANALYZER -

Distr/Progr: Volume/Standard
Sample ID 1: S-6
Sample ID 2:

Date: 04/30/96 Time: 08:44
Above Residual: 0.00/NR
Below Residual: 0.00

- MICROTRAC X100 PARTICLE ANALYZER -
Percent Passing Data
Version 4.13

J-7 (fresh sludge)

ASVR Parameters

Flow Rate: n/a ml/sec
Ultrasonic Power: n/a watts
Ultrasonic Time: n/a seconds

Id #1:
Distrib. Format: Volume
Filter: On
Run Time: 60 seconds
Run Number: 1 of 1 runs
Transmission: 0.87
Laser Int: 1.021/1.006/1.006
Residuals: Disabled
Above Residual: 0.00
Below Residual: 0.00

Summary Data
dv = 0.0909
10% = 1.18
50% = 4.12
90% = 151.61
mv = 29.72
mn = 0.951
ma = 2.82
cs = 2.126
sd = 12.66

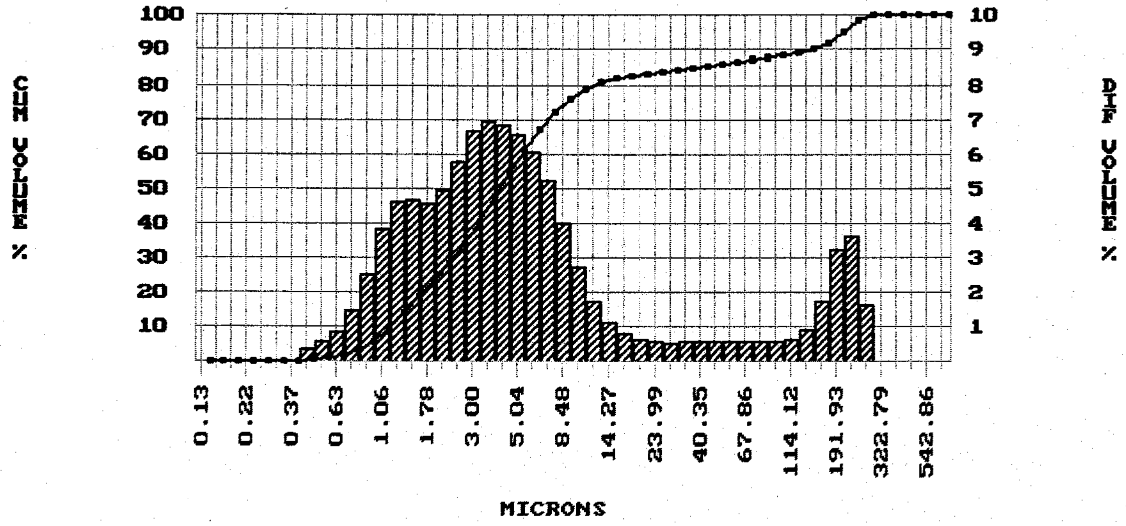
Id #2:
Date: 04/26/96 Time: 9:03
Chan. Progression: Standard
Upper Channel Edge: 704.00
Lower Channel Edge: 0.12
Number of Channels: 50
Fluid Refractive Index: n/a
Transparent Particles: No
Spherical Particles: n/a
Part. Refractive Index: n/a

ch top	%pass	%-chn
704.00	100.00	0.00
591.99	100.00	0.00
497.80	100.00	0.00
418.60	100.00	0.00
352.00	100.00	0.00
296.00	100.00	1.59
248.90	98.41	3.63
209.30	94.78	3.23
176.00	91.55	1.73
148.00	89.82	0.91
124.45	88.91	0.61
104.65	88.30	0.54
88.00	87.76	0.54
74.00	87.22	0.56
62.23	86.66	0.57
52.33	86.09	0.57
44.00	85.52	0.55
37.00	84.97	0.53
31.11	84.44	0.51
26.16	83.93	0.53
22.00	83.40	0.61
18.50	82.79	0.79
15.56	82.00	1.13
13.08	80.87	1.75
11.00	79.12	2.74
9.25	76.38	4.01
7.78	72.37	5.24
6.54	67.13	6.08
5.50	61.05	6.53
4.62	54.52	6.84
3.89	47.68	6.95
3.27	40.73	6.64
2.75	34.09	5.80
2.31	28.29	4.95
1.94	23.34	4.57
1.64	18.77	4.65
1.38	14.12	4.61
1.16	9.51	3.81
0.97	5.70	2.51
0.82	3.19	1.45
0.69	1.74	0.85
0.58	0.89	0.53
0.49	0.36	0.36
0.41	0.00	0.00
0.34	0.00	0.00
0.29	0.00	0.00
0.24	0.00	0.00
0.20	0.00	0.00
0.17	0.00	0.00
0.14	0.00	0.00

- MICROTRAC -

- X100 PARTICLE ANALYZER -

Distr/Progr: Volume/Standard Date: 04/26/96 Time: 09:03
Sample ID 1: J-7 fresh Above Residual: 0.00/NR
Sample ID 2: Below Residual: 0.00



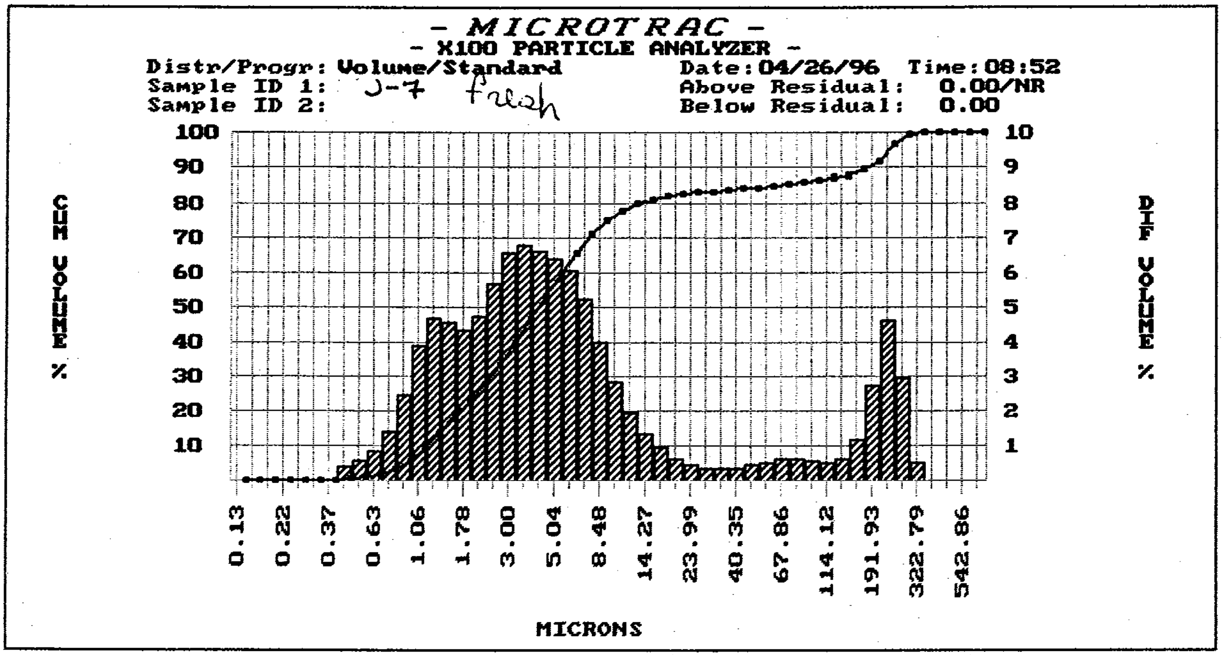
```
                 - MICROTRAC X100 PARTICLE ANALYZER -
                          Percent Passing Data
                              Version 4.13

   J-7        (fresh sludge)                         ASVR Parameters

                                               Flow Rate: n/a ml/sec
                                               Ultrasonic Power: n/a watts
                                               Ultrasonic Time: n/a seconds

 Id #1:                          Summary Data    Id #2:
 Distrib. Format: Volume         dv  = 0.1087    Date: 04/26/96   Time:  8:52
 Filter: On                      10% =   1.18    Chan. Progression: Standard
 Run Time:  60 seconds           50% =   4.23    Upper Channel Edge: 704.00
 Run Number:   1 of  1 runs      90% = 187.81    Lower Channel Edge:   0.12
 Transmission: 0.85              mv  =  34.74    Number of Channels:  50
 Laser Int: 1.021/1.006/1.006    mn  =  0.948    Fluid Refractive Index: n/a
 Residuals: Disabled             ma  =   2.87    Transparent Particles: No
 Above Residual:    0.00         cs  =  2.094    Spherical Particles: n/a
 Below Residual:    0.00         sd  =  19.24    Part. Refractive Index: n/a

ch top  %pass %-chn ch top   %pass %-chn ch top   %pass %-chn ch top   %pass %-chn
704.00 100.00  0.00   9.25   75.01  4.00
591.99 100.00  0.00   7.78   71.01  5.20
497.80 100.00  0.00   6.54   65.81  6.04
418.60 100.00  0.00   5.50   59.77  6.41
352.00 100.00  0.51   4.62   53.36  6.63
296.00  99.49  2.96   3.89   46.73  6.79
248.90  96.53  4.60   3.27   39.94  6.55
209.30  91.93  2.74   2.75   33.39  5.67
176.00  89.19  1.17   2.31   27.72  4.72
148.00  88.02  0.62   1.94   23.00  4.33
124.45  87.40  0.50   1.64   18.67  4.54
104.65  86.90  0.54   1.38   14.13  4.69
 88.00  86.36  0.61   1.16    9.44  3.89
 74.00  85.75  0.61   0.97    5.55  2.46
 62.23  85.14  0.52   0.82    3.09  1.37
 52.33  84.62  0.42   0.69    1.72  0.81
 44.00  84.20  0.35   0.58    0.91  0.54
 37.00  83.85  0.33   0.49    0.37  0.37
 31.11  83.52  0.36   0.41    0.00  0.00
 26.16  83.16  0.44   0.34    0.00  0.00
 22.00  82.72  0.62   0.29    0.00  0.00
 18.50  82.10  0.92   0.24    0.00  0.00
 15.56  81.18  1.35   0.20    0.00  0.00
 13.08  79.83  1.97   0.17    0.00  0.00
 11.00  77.86  2.85   0.14    0.00  0.00
```



- MICROTRAC -
- X100 PARTICLE ANALYZER -

Distr/Progr: Volume/Standard
Date: 04/26/96 Time: 08:52
Sample ID 1: J-7 fresh
Above Residual: 0.00/NR
Sample ID 2:
Below Residual: 0.00

- MICROTRAC X100 PARTICLE ANALYZER -
Percent Passing Data
Version 4.13

J-7
Composite aged sludge

ASVR Parameters

Flow Rate: n/a ml/sec
Ultrasonic Power: n/a watts
Ultrasonic Time: n/a seconds

Id #1:
Distrib. Format: Volume
Filter: On
Run Time: 60 seconds
Run Number: 1 of 1 runs
Transmission: 0.94
Laser Int: 1.020/1.005/1.005
Residuals: Disabled
Above Residual: 0.00
Below Residual: 0.00

Summary Data
dv = 0.0532
10% = 1.72
50% = 9.58
90% = 51.65
mv = 18.95
mn = 0.851
ma = 4.51
cs = 1.330
sd = 17.93

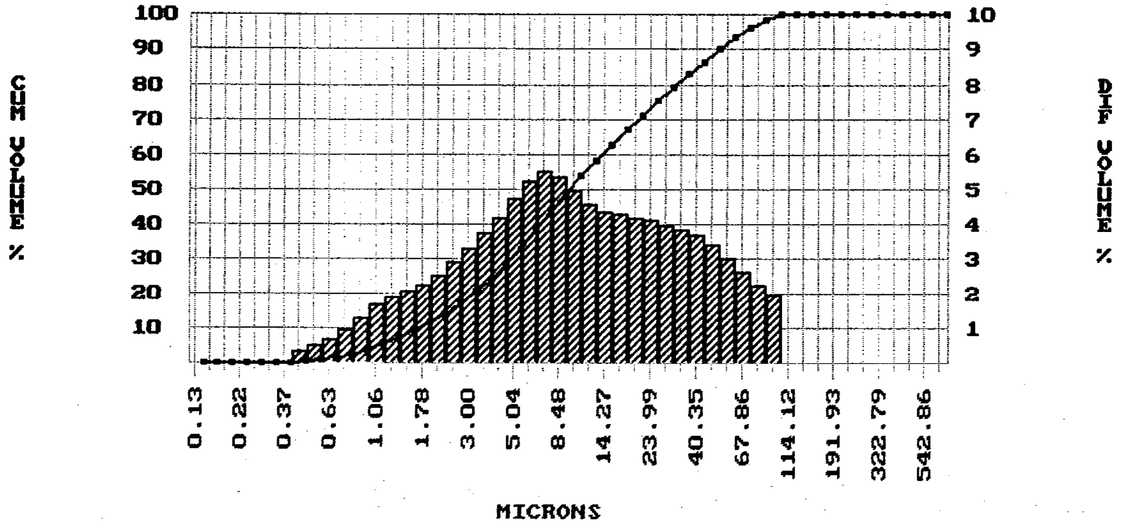
Id #2:
Date: 05/01/96 Time: 14:17
Chan. Progression: Standard
Upper Channel Edge: 704.00
Lower Channel Edge: 0.12
Number of Channels: 50
Fluid Refractive Index: n/a
Transparent Particles: No
Spherical Particles: n/a
Part. Refractive Index: n/a

ch top	%pass	%-chn	ch top	%pass	%-chn	ch top	%pass	%-chn	ch top	%pass	%-chn
704.00	100.00	0.00	9.25	48.98	5.36						
591.99	100.00	0.00	7.78	43.62	5.52						
497.80	100.00	0.00	6.54	38.10	5.25						
418.60	100.00	0.00	5.50	32.85	4.70						
352.00	100.00	0.00	4.62	28.15	4.15						
296.00	100.00	0.00	3.89	24.00	3.70						
248.90	100.00	0.00	3.27	20.30	3.30						
209.30	100.00	0.00	2.75	17.00	2.88						
176.00	100.00	0.00	2.31	14.12	2.51						
148.00	100.00	0.00	1.94	11.61	2.23						
124.45	100.00	0.00	1.64	9.38	2.07						
104.65	100.00	1.94	1.38	7.31	1.91						
88.00	98.06	2.22	1.16	5.40	1.66						
74.00	95.84	2.59	0.97	3.74	1.30						
62.23	93.25	3.00	0.82	2.44	0.94						
52.33	90.25	3.40	0.69	1.50	0.66						
44.00	86.85	3.68	0.58	0.84	0.48						
37.00	83.17	3.85	0.49	0.36	0.36						
31.11	79.32	3.96	0.41	0.00	0.00						
26.16	75.36	4.09	0.34	0.00	0.00						
22.00	71.27	4.19	0.29	0.00	0.00						
18.50	67.08	4.27	0.24	0.00	0.00						
15.56	62.81	4.34	0.20	0.00	0.00						
13.08	58.47	4.55	0.17	0.00	0.00						
11.00	53.92	4.94	0.14	0.00	0.00						

- MICROTRAC -

- X100 PARTICLE ANALYZER -

Distr/Progr: Volume/Standard Date: 05/01/96 Time: 14:17
Sample ID 1: J-7 - aged Above Residual: 0.00/NR
Sample ID 2: Below Residual: 0.00

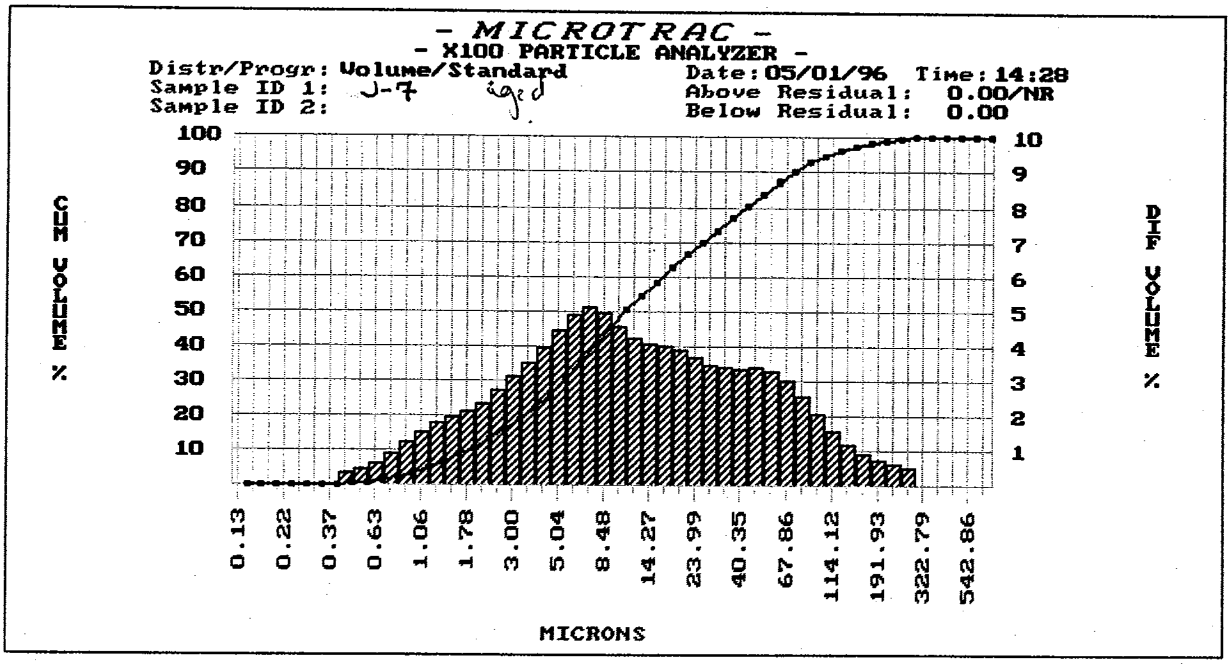


```
                 - MICROTRAC X100 PARTICLE ANALYZER -
                         Percent Passing Data
                             Version 4.13

 J-7                                          ASVR Parameters
 Composite aged sludge
                                           Flow Rate: n/a ml/sec
                                           Ultrasonic Power: n/a watts
                                           Ultrasonic Time: n/a seconds

 Id #1:                     Summary Data   Id #2:
 Distrib. Format: Volume    dv  = 0.0579   Date: 05/01/96   Time: 14:28
 Filter: On                 10% =   1.82   Chan. Progression: Standard
 Run Time:  60 seconds      50% =  10.87   Upper Channel Edge: 704.00
 Run Number:   1 of  1 runs 90% =  74.14   Lower Channel Edge:   0.12
 Transmission: 0.94         mv  =  27.77   Number of Channels:  50
 Laser Int: 1.020/1.005/1.005 mn = 0.847   Fluid Refractive Index: n/a
 Residuals: Disabled        ma  =   4.82   Transparent Particles: No
 Above Residual:   0.00     cs  =  1.244   Spherical Particles: n/a
 Below Residual:   0.00     sd  =  25.21   Part. Refractive Index: n/a

ch top  %pass %-chn ch top  %pass %-chn ch top  %pass %-chn ch top  %pass %-chn
704.00 100.00  0.00   9.25  45.74  4.95
591.99 100.00  0.00   7.78  40.79  5.12
497.80 100.00  0.00   6.54  35.67  4.90
418.60 100.00  0.00   5.50  30.77  4.42
352.00 100.00  0.00   4.62  26.35  3.92
296.00 100.00  0.50   3.89  22.43  3.49
248.90  99.50  0.61   3.27  18.94  3.10
209.30  98.89  0.73   2.75  15.84  2.70
176.00  98.16  0.90   2.31  13.14  2.35
148.00  97.26  1.17   1.94  10.79  2.09
124.45  96.09  1.55   1.64   8.70  1.92
104.65  94.54  2.03   1.38   6.78  1.76
 88.00  92.51  2.54   1.16   5.02  1.52
 74.00  89.97  3.00   0.97   3.50  1.20
 62.23  86.97  3.28   0.82   2.30  0.88
 52.33  83.69  3.38   0.69   1.42  0.62
 44.00  80.31  3.36   0.58   0.80  0.45
 37.00  76.95  3.37   0.49   0.35  0.35
 31.11  73.58  3.47   0.41   0.00  0.00
 26.16  70.11  3.68   0.34   0.00  0.00
 22.00  66.43  3.87   0.29   0.00  0.00
 18.50  62.56  3.99   0.24   0.00  0.00
 15.56  58.57  4.05   0.20   0.00  0.00
 13.08  54.52  4.22   0.17   0.00  0.00
 11.00  50.30  4.56   0.14   0.00  0.00
```



- MICROTRAC -
- X100 PARTICLE ANALYZER -

Distr/Progr: Volume/Standard
Sample ID 1: J-7 ig'd
Sample ID 2:

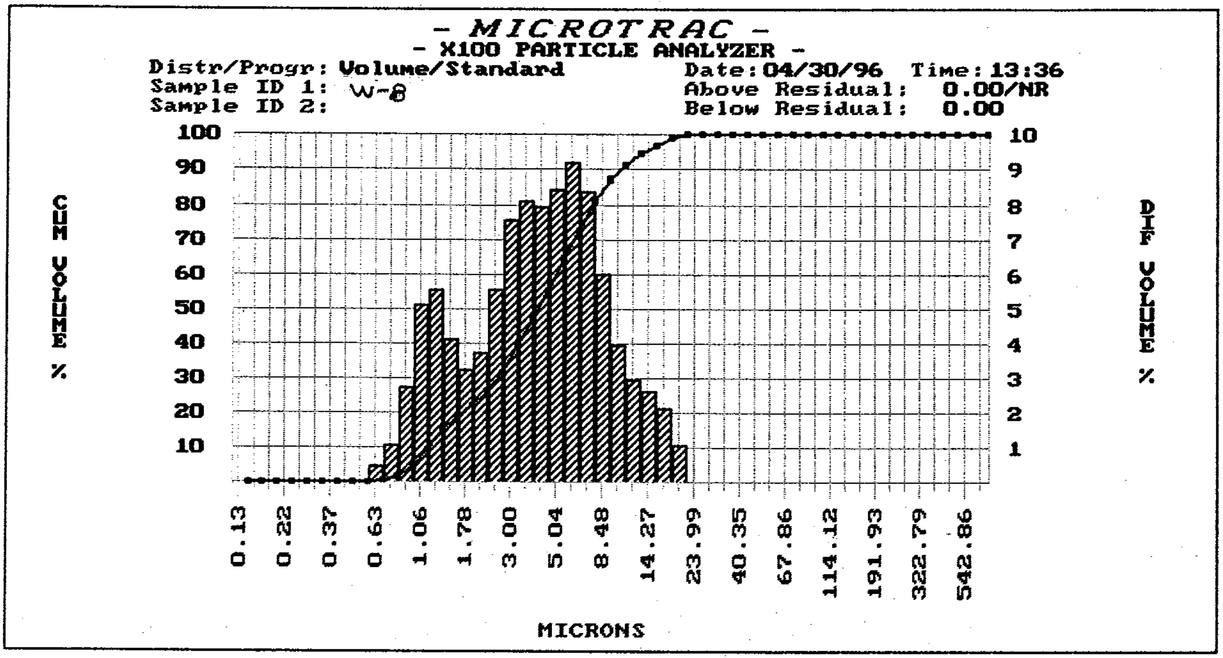
Date: 05/01/96 Time: 14:28
Above Residual: 0.00/NR
Below Residual: 0.00

```
                  - MICROTRAC X100 PARTICLE ANALYZER -
                          Percent Passing Data
                              Version 4.13

 W-8                                              ASVR Parameters
 Fresh sludge
                                               Flow Rate: n/a ml/sec
                                               Ultrasonic Power: n/a watts
                                               Ultrasonic Time: n/a seconds

 Id #1: W-8                    Summary Data    Id #2:
 Distrib. Format: Volume       dv  = 0.0285    Date: 04/30/96   Time: 13:36
 Filter: On                    10% =   1.18    Chan. Progression: Standard
 Run Time:  60 seconds         50% =   4.12    Upper Channel Edge: 704.00
 Run Number:   1 of  1 runs    90% =  10.34    Lower Channel Edge:   0.12
 Transmission: 0.95            mv  =   5.13    Number of Channels:  50
 Laser Int: 1.020/1.005/1.006  mn  =   1.19    Fluid Refractive Index: n/a
 Residuals: Disabled           ma  =   2.80    Transparent Particles: No
 Above Residual:   0.00        cs  =  2.141    Spherical Particles: n/a
 Below Residual:   0.00        sd  =   3.45    Part. Refractive Index: n/a

ch top  %pass  %-chn|ch top  %pass  %-chn|ch top  %pass %-chn|ch top  %pass %-chn
704.00 100.00  0.00 |  9.25  87.32  6.02 |                    |
591.99 100.00  0.00 |  7.78  81.30  8.41 |                    |
497.80 100.00  0.00 |  6.54  72.89  9.17 |                    |
418.60 100.00  0.00 |  5.50  63.72  8.44 |                    |
352.00 100.00  0.00 |  4.62  55.28  7.97 |                    |
296.00 100.00  0.00 |  3.89  47.31  8.13 |                    |
248.90 100.00  0.00 |  3.27  39.18  7.57 |                    |
209.30 100.00  0.00 |  2.75  31.61  5.57 |                    |
176.00 100.00  0.00 |  2.31  26.04  3.75 |                    |
148.00 100.00  0.00 |  1.94  22.29  3.25 |                    |
124.45 100.00  0.00 |  1.64  19.04  4.11 |                    |
104.65 100.00  0.00 |  1.38  14.93  5.58 |                    |
 88.00 100.00  0.00 |  1.16   9.35  5.13 |                    |
 74.00 100.00  0.00 |  0.97   4.22  2.72 |                    |
 62.23 100.00  0.00 |  0.82   1.50  1.07 |                    |
 52.33 100.00  0.00 |  0.69   0.43  0.43 |                    |
 44.00 100.00  0.00 |  0.58   0.00  0.00 |                    |
 37.00 100.00  0.00 |  0.49   0.00  0.00 |                    |
 31.11 100.00  0.00 |  0.41   0.00  0.00 |                    |
 26.16 100.00  0.00 |  0.34   0.00  0.00 |                    |
 22.00 100.00  1.06 |  0.29   0.00  0.00 |                    |
 18.50  98.94  2.12 |  0.24   0.00  0.00 |                    |
 15.56  96.82  2.61 |  0.20   0.00  0.00 |                    |
 13.08  94.21  2.95 |  0.17   0.00  0.00 |                    |
 11.00  91.26  3.94 |  0.14   0.00  0.00 |                    |
```



- MICROTRAC -

- X100 PARTICLE ANALYZER -

Distr/Progr: Volume/Standard Date: 04/30/96 Time: 13:36

Sample ID 1: W-8 Above Residual: 0.00/NR

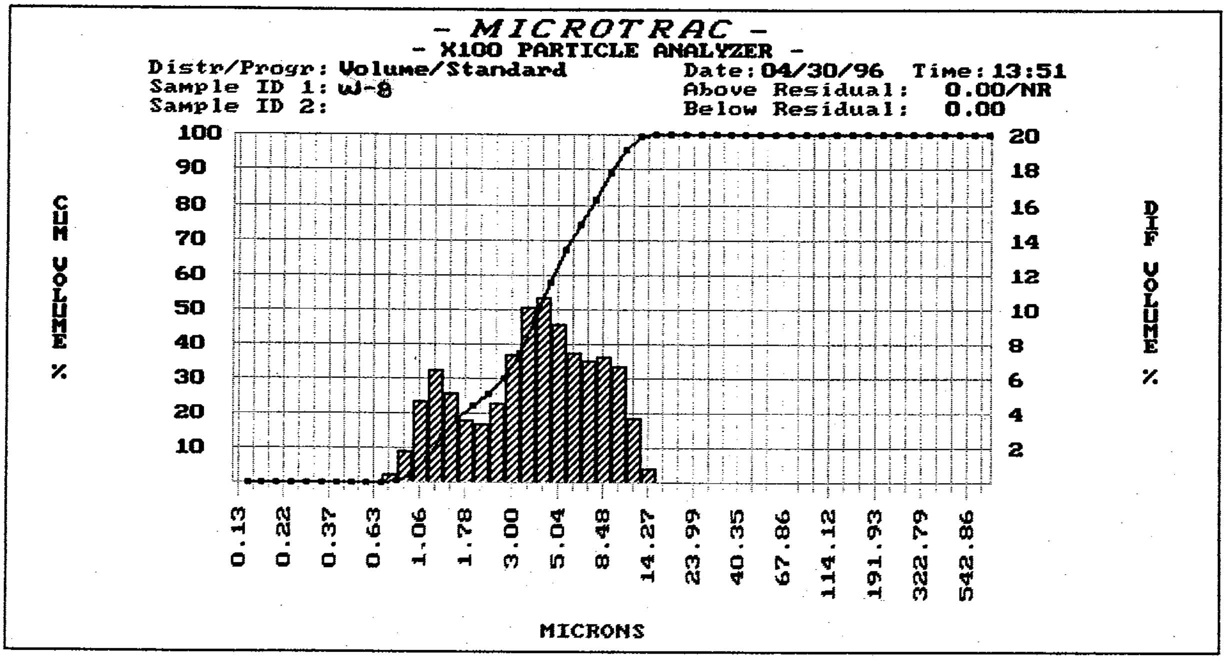
Sample ID 2: Below Residual: 0.00

```
                 - MICROTRAC X100 PARTICLE ANALYZER -
                         Percent Passing Data
                             Version 4.13

W-8                                           ASVR Parameters
Fresh sludge
                                         Flow Rate: n/a ml/sec
                                         Ultrasonic Power: n/a watts
                                         Ultrasonic Time: n/a seconds

Id #1:                          Summary Data     Id #2:
Distrib. Format: Volume         dv  = 0.0290     Date: 04/30/96   Time: 13:51
Filter: On                      10% =   1.26     Chan. Progression: Standard
Run Time:  60 seconds           50% =   4.06     Upper Channel Edge: 704.00
Run Number:   1 of  1 runs      90% =   9.51     Lower Channel Edge:   0.12
Transmission: 0.95              mv  =   4.75     Number of Channels:  50
Laser Int: 1.020/1.005/1.005    mn  =   1.35     Fluid Refractive Index: n/a
Residuals: Disabled             ma  =   2.89     Transparent Particles: No
Above Residual:   0.00          cs  =  2.075     Spherical Particles: n/a
Below Residual:   0.00          sd  =   3.37     Part. Refractive Index: n/a

ch top  %pass %-chn ch top  %pass %-chn ch top  %pass %-chn ch top  %pass %-chn
704.00 100.00  0.00   9.25  88.86  7.27
591.99 100.00  0.00   7.78  81.59  7.01
497.80 100.00  0.00   6.54  74.58  7.50
418.60 100.00  0.00   5.50  67.08  9.07
352.00 100.00  0.00   4.62  58.01 10.66
296.00 100.00  0.00   3.89  47.35 10.14
248.90 100.00  0.00   3.27  37.21  7.29
209.30 100.00  0.00   2.75  29.92  4.53
176.00 100.00  0.00   2.31  25.39  3.30
148.00 100.00  0.00   1.94  22.09  3.55
124.45 100.00  0.00   1.64  18.54  5.12
104.65 100.00  0.00   1.38  13.42  6.48
 88.00 100.00  0.00   1.16   6.94  4.72
 74.00 100.00  0.00   0.97   2.22  1.79
 62.23 100.00  0.00   0.82   0.43  0.43
 52.33 100.00  0.00   0.69   0.00  0.00
 44.00 100.00  0.00   0.58   0.00  0.00
 37.00 100.00  0.00   0.49   0.00  0.00
 31.11 100.00  0.00   0.41   0.00  0.00
 26.16 100.00  0.00   0.34   0.00  0.00
 22.00 100.00  0.00   0.29   0.00  0.00
 18.50 100.00  0.00   0.24   0.00  0.00
 15.56 100.00  0.76   0.20   0.00  0.00
 13.08  99.24  3.71   0.17   0.00  0.00
 11.00  95.53  6.67   0.14   0.00  0.00
```



- MICROTRAC -

- X100 PARTICLE ANALYZER -

Distr/Progr: Volume/Standard
Sample ID 1: W-8
Sample ID 2:

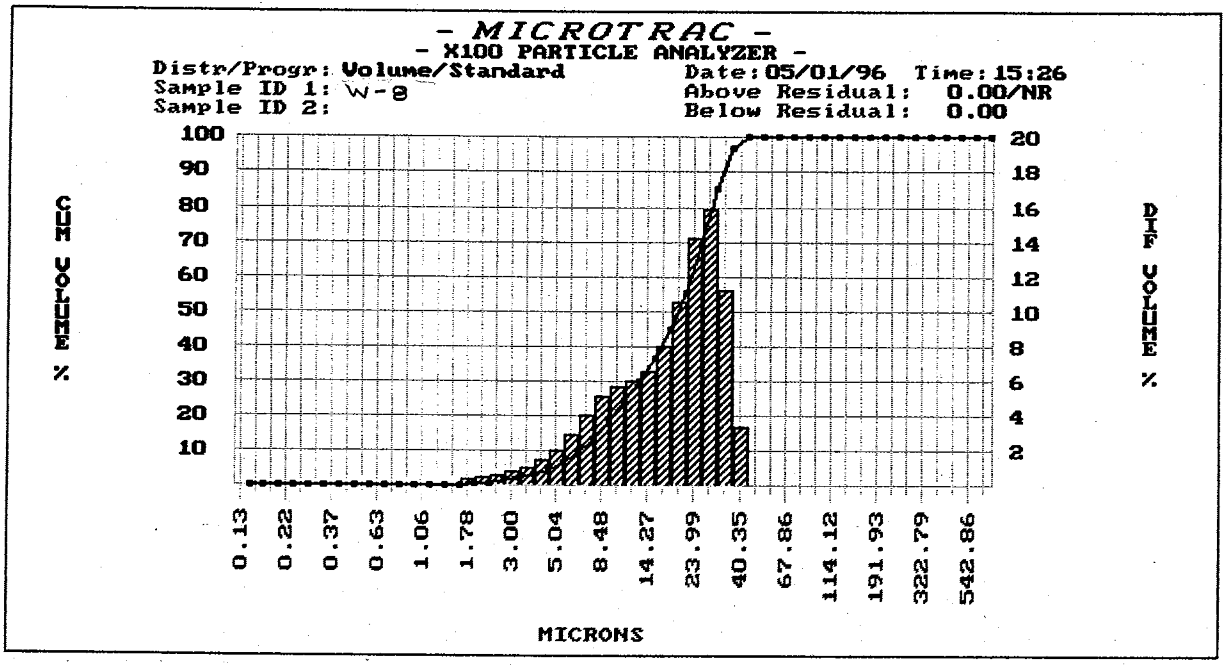
Date: 04/30/96 Time: 13:51
Above Residual: 0.00/NR
Below Residual: 0.00

```
               - MICROTRAC X100 PARTICLE ANALYZER -
                        Percent Passing Data
                            Version 4.13

 W-8                                          ASVR Parameters
 Composite aged sludge
                                         Flow Rate: n/a ml/sec
                                         Ultrasonic Power: n/a watts
                                         Ultrasonic Time: n/a seconds

 Id #1:                      Summary Data   Id #2:
 Distrib. Format: Volume     dv  = 0.0410   Date: 05/01/96   Time: 15:26
 Filter: On                  10% =   6.72   Chan. Progression: Standard
 Run Time:  60 seconds       50% =  20.29   Upper Channel Edge: 704.00
 Run Number:   1 of  1 runs  90% =  32.99   Lower Channel Edge:   0.12
 Transmission: 0.97          mv  =  19.93   Number of Channels:  50
 Laser Int: 1.020/1.005/1.005 mn =   3.92   Fluid Refractive Index: n/a
 Residuals: Disabled         ma  =  13.11   Transparent Particles: No
 Above Residual:   0.00      cs  =  0.458   Spherical Particles: n/a
 Below Residual:   0.00      sd  =  11.04   Part. Refractive Index: n/a

ch top  %pass %-chn ch top  %pass %-chn ch top  %pass %-chn ch top  %pass %-chn
704.00 100.00  0.00   9.25  18.57  5.09
591.99 100.00  0.00   7.78  13.48  4.04
497.80 100.00  0.00   6.54   9.44  2.87
418.60 100.00  0.00   5.50   6.57  1.97
352.00 100.00  0.00   4.62   4.60  1.39
296.00 100.00  0.00   3.89   3.21  1.03
248.90 100.00  0.00   3.27   2.18  0.79
209.30 100.00  0.00   2.75   1.39  0.60
176.00 100.00  0.00   2.31   0.79  0.45
148.00 100.00  0.00   1.94   0.34  0.34
124.45 100.00  0.00   1.64   0.00  0.00
104.65 100.00  0.00   1.38   0.00  0.00
 88.00 100.00  0.00   1.16   0.00  0.00
 74.00 100.00  0.00   0.97   0.00  0.00
 62.23 100.00  0.00   0.82   0.00  0.00
 52.33 100.00  0.00   0.69   0.00  0.00
 44.00 100.00  3.29   0.58   0.00  0.00
 37.00  96.71 11.22   0.49   0.00  0.00
 31.11  85.49 15.92   0.41   0.00  0.00
 26.16  69.57 14.22   0.34   0.00  0.00
 22.00  55.35 10.60   0.29   0.00  0.00
 18.50  44.75  7.96   0.24   0.00  0.00
 15.56  36.79  6.55   0.20   0.00  0.00
 13.08  30.24  5.98   0.17   0.00  0.00
 11.00  24.26  5.69   0.14   0.00  0.00
```



- MICROTRAC -

- X100 PARTICLE ANALYZER -

Distr/Progr: Volume/Standard   Date: 05/01/96 Time: 15:26
Sample ID 1: W-8   Above Residual: 0.00/NR
Sample ID 2:   Below Residual: 0.00

- MICROTRAC X100 PARTICLE ANALYZER -
Percent Passing Data
Version 4.13

W-8
Composite aged sludge

ASVR Parameters

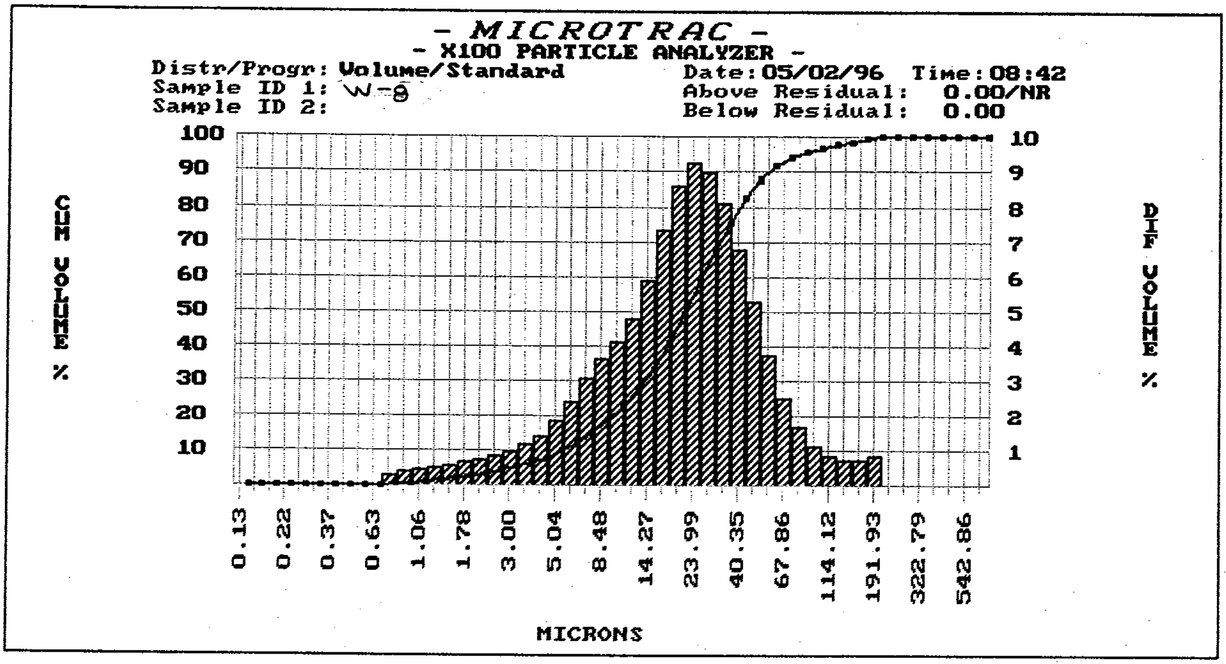
Flow Rate: n/a ml/sec
Ultrasonic Power: n/a watts
Ultrasonic Time: n/a seconds

Id #1:
Distrib. Format: Volume
Filter: On
Run Time: 60 seconds
Run Number: 1 of 1 runs
Transmission: 0.97-
Laser Int: 1.021/1.006/1.006
Residuals: Disabled
Above Residual: 0.00
Below Residual: 0.00

Summary Data
dv = 0.0526
10% = 5.61
50% = 22.21
90% = 57.23
mv = 29.43
mn = 1.32
ma = 10.99
cs = 0.546
sd = 18.88

Id #2:
Date: 05/02/96 Time: 8:42
Chan. Progression: Standard
Upper Channel Edge: 704.00
Lower Channel Edge: 0.12
Number of Channels: 50
Fluid Refractive Index: n/a
Transparent Particles: No
Spherical Particles: n/a
Part. Refractive Index: n/a

ch top	%pass	%-chn	ch top	%pass	%-chn	ch top	%pass	%-chn	ch top	%pass	%-chn
704.00	100.00	0.00	9.25	18.81	3.60						
591.99	100.00	0.00	7.78	15.21	3.04						
497.80	100.00	0.00	6.54	12.17	2.41						
418.60	100.00	0.00	5.50	9.76	1.83						
352.00	100.00	0.00	4.62	7.93	1.41						
296.00	100.00	0.00	3.89	6.52	1.14						
248.90	100.00	0.00	3.27	5.38	0.96						
209.30	100.00	0.82	2.75	4.42	0.83						
176.00	99.18	0.70	2.31	3.59	0.73						
148.00	98.48	0.71	1.94	2.86	0.64						
124.45	97.77	0.84	1.64	2.22	0.58						
104.65	96.93	1.13	1.38	1.64	0.52						
88.00	95.80	1.65	1.16	1.12	0.45						
74.00	94.15	2.51	0.97	0.67	0.37						
62.23	91.64	3.75	0.82	0.30	0.30						
52.33	87.89	5.28	0.69	0.00	0.00						
44.00	82.61	6.80	0.58	0.00	0.00						
37.00	75.81	8.11	0.49	0.00	0.00						
31.11	67.70	8.97	0.41	0.00	0.00						
26.16	58.73	9.22	0.34	0.00	0.00						
22.00	49.51	8.61	0.29	0.00	0.00						
18.50	40.90	7.32	0.24	0.00	0.00						
15.56	33.58	5.88	0.20	0.00	0.00						
13.08	27.70	4.79	0.17	0.00	0.00						
11.00	22.91	4.10	0.14	0.00	0.00						



- *MICROTRAC* -
- X100 PARTICLE ANALYZER -

Distr/Progr: Volume/Standard
Sample ID 1: W-8
Sample ID 2:

Date: 05/02/96 Time: 08:42
Above Residual: 0.00/NR
Below Residual: 0.00

- MICROTRAC X100 PARTICLE ANALYZER -
Percent Passing Data
Version 4.13

N-9 sludge pond
composite aged sludge

ASVR Parameters

Flow Rate: n/a ml/sec
Ultrasonic Power: n/a watts
Ultrasonic Time: n/a seconds

Id #1:
Distrib. Format: Volume
Filter: On
Run Time: 60 seconds
Run Number: 1 of 1 runs
Transmission: 0.94
Laser Int: 1.021/1.006/1.006
Residuals: Disabled
Above Residual: 0.00
Below Residual: 0.00

Summary Data
dv = 0.1076
10% = 4.21
50% = 21.65
90% = 54.00
mv = 38.22
mn = 1.08
ma = 8.96
cs = 0.670
sd = 19.67

Id #2:
Date: 05/01/96 Time: 9:17
Chan. Progression: Standard
Upper Channel Edge: 704.00
Lower Channel Edge: 0.12
Number of Channels: 50
Fluid Refractive Index: n/a
Transparent Particles: No
Spherical Particles: n/a
Part. Refractive Index: n/a

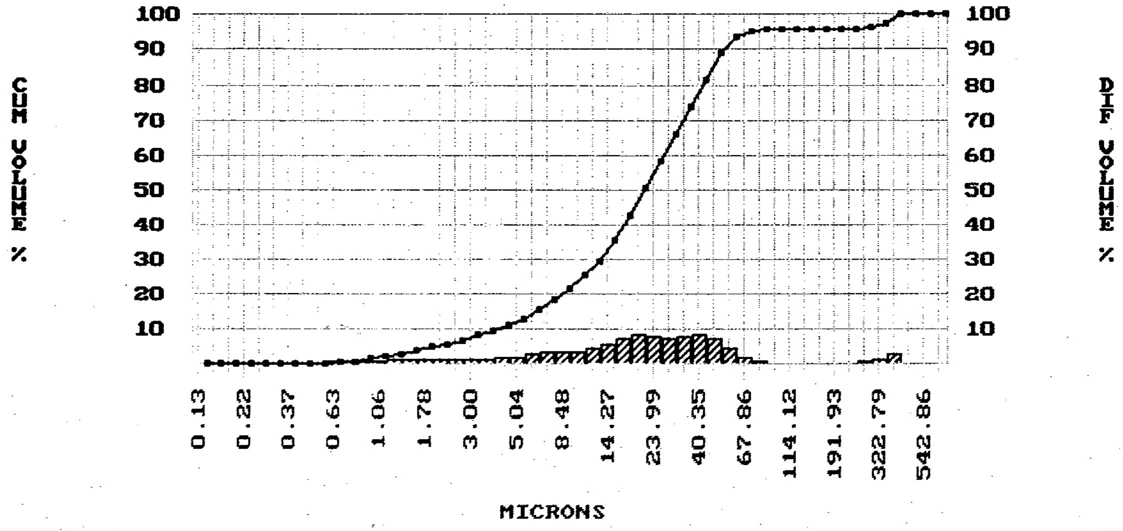
ch top	%pass	%-chn	ch top	%pass	%-chn	ch top	%pass	%-chn	ch top	%pass	%-chn
704.00	100.00	0.00	9.25	21.72	3.34						
591.99	100.00	0.00	7.78	18.38	3.06						
497.80	100.00	0.00	6.54	15.32	2.53						
418.60	100.00	2.79	5.50	12.79	1.93						
352.00	97.21	1.00	4.62	10.86	1.51						
296.00	96.21	0.41	3.89	9.35	1.29						
248.90	95.80	0.00	3.27	8.06	1.18						
209.30	95.80	0.00	2.75	6.88	1.09						
176.00	95.80	0.00	2.31	5.79	1.00						
148.00	95.80	0.00	1.94	4.79	0.92						
124.45	95.80	0.00	1.64	3.87	0.88						
104.65	95.80	0.00	1.38	2.99	0.84						
88.00	95.80	0.65	1.16	2.15	0.75						
74.00	95.15	1.82	0.97	1.40	0.61						
62.23	93.33	4.31	0.82	0.79	0.46						
52.33	89.02	7.17	0.69	0.33	0.33						
44.00	81.85	8.21	0.58	0.00	0.00						
37.00	73.64	7.74	0.49	0.00	0.00						
31.11	65.90	7.38	0.41	0.00	0.00						
26.16	58.52	7.76	0.34	0.00	0.00						
22.00	50.76	8.09	0.29	0.00	0.00						
18.50	42.67	7.35	0.24	0.00	0.00						
15.56	35.32	5.70	0.20	0.00	0.00						
13.08	29.62	4.30	0.17	0.00	0.00						
11.00	25.32	3.60	0.14	0.00	0.00						

- MICROTRAC -

- X100 PARTICLE ANALYZER -

Distr/Progr: Volume/Standard
Sample ID 1:
Sample ID 2: N-9

Date: 05/01/96 Time: 09:17
Above Residual: 0.00/NR
Below Residual: 0.00



- MICROTRAC X100 PARTICLE ANALYZER -
Percent Passing Data
Version 4.13

N-9 sludge pond
composite aged sludge

ASVR Parameters

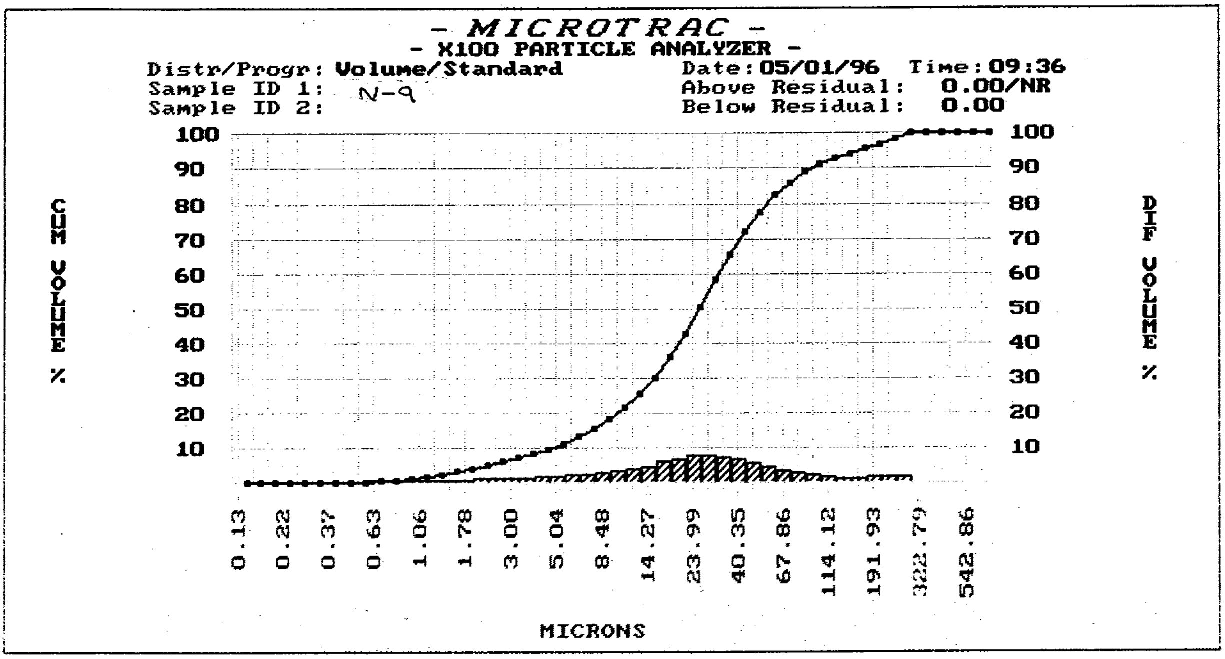
Flow Rate: n/a ml/sec
Ultrasonic Power: n/a watts
Ultrasonic Time: n/a seconds

Id #1:
Distrib. Format: Volume
Filter: On
Run Time: 60 seconds
Run Number: 1 of 1 runs
Transmission: 0.94
Laser Int: 1.020/1.005/1.006
Residuals: Disabled
Above Residual: 0.00
Below Residual: 0.00

Summary Data
dv = 0.1008
10% = 4.82
50% = 25.87
90% = 95.36
mv = 42.61
mn = 1.06
ma = 10.22
cs = 0.587
sd = 28.97

Id #2:
Date: 05/01/96 Time: 9:36
Chan. Progression: Standard
Upper Channel Edge: 704.00
Lower Channel Edge: 0.12
Number of Channels: 50
Fluid Refractive Index: n/a
Transparent Particles: No
Spherical Particles: n/a
Part. Refractive Index: n/a

ch top	%pass	%-chn	ch top	%pass	%-chn	ch top	%pass	%-chn	ch top	%pass	%-chn
704.00	100.00	0.00	9.25	18.48	2.76						
591.99	100.00	0.00	7.78	15.72	2.39						
497.80	100.00	0.00	6.54	13.33	2.02						
418.60	100.00	0.00	5.50	11.31	1.68						
352.00	100.00	0.00	4.62	9.63	1.42						
296.00	100.00	1.70	3.89	8.21	1.23						
248.90	98.30	1.58	3.27	6.98	1.09						
209.30	96.72	1.41	2.75	5.89	0.98						
176.00	95.31	1.32	2.31	4.91	0.89						
148.00	93.99	1.38	1.94	4.02	0.81						
124.45	92.61	1.59	1.64	3.21	0.74						
104.65	91.02	2.02	1.38	2.47	0.67						
88.00	89.00	2.68	1.16	1.80	0.58						
74.00	86.32	3.60	0.97	1.22	0.49						
62.23	82.72	4.68	0.82	0.73	0.40						
52.33	78.04	5.78	0.69	0.33	0.33						
44.00	72.26	6.68	0.58	0.00	0.00						
37.00	65.58	7.35	0.49	0.00	0.00						
31.11	58.23	7.72	0.41	0.00	0.00						
26.16	50.51	7.61	0.34	0.00	0.00						
22.00	42.90	6.94	0.29	0.00	0.00						
18.50	35.96	5.86	0.24	0.00	0.00						
15.56	30.10	4.68	0.20	0.00	0.00						
13.08	25.42	3.77	0.17	0.00	0.00						
11.00	21.65	3.17	0.14	0.00	0.00						



- MICROTRAC -

- X100 PARTICLE ANALYZER -

Distr/Progr: Volume/Standard Date:05/01/96 Time:09:36
Sample ID 1: N-9 Above Residual: 0.00/NR
Sample ID 2: Below Residual: 0.00

```
                 - MICROTRAC X100 PARTICLE ANALYZER -
                        Percent Passing Data
                            Version 4.13

   B-10    (fresh sludge)                        ASVR Parameters

                                           Flow Rate: n/a ml/sec
                                           Ultrasonic Power: n/a watts
                                           Ultrasonic Time: n/a seconds

 Id #1:                         Summary Data      Id #2:
 Distrib. Format: Volume        dv  = 0.0781      Date: 04/25/96   Time: 11:26
 Filter: On                     10% =   5.58      Chan. Progression: Standard
 Run Time:  60 seconds          50% =  14.63      Upper Channel Edge: 704.00
 Run Number:   1 of  1 runs     90% =  47.95      Lower Channel Edge:   0.12
 Transmission: 0.96             mv  =  31.10      Number of Channels:  50
 Laser Int: 1.021/1.006/1.006   mn  =   2.84      Fluid Refractive Index: n/a
 Residuals: Disabled            ma  =  10.62      Transparent Particles: No
 Above Residual:   0.00         cs  =  0.565      Spherical Particles: n/a
 Below Residual:   0.00         sd  =  11.39      Part. Refractive Index: n/a

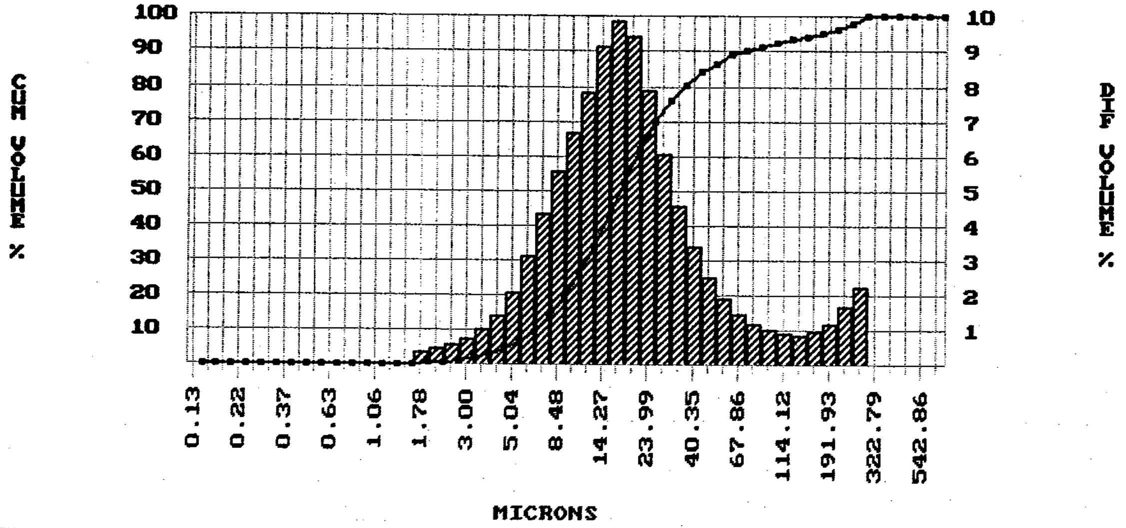
ch top  %pass %-chn  ch top  %pass %-chn  ch top  %pass %-chn  ch top  %pass %-chn
704.00 100.00  0.00    9.25  27.19  7.12
591.99 100.00  0.00    7.78  20.07  5.98
497.80 100.00  0.00    6.54  14.09  4.41
418.60 100.00  0.31    5.50   9.68  2.96
352.00  99.69  0.98    4.62   6.72  1.95
296.00  98.71  1.58    3.89   4.77  1.35
248.90  97.13  1.34    3.27   3.42  0.98
209.30  95.79  0.84    2.75   2.44  0.74
176.00  94.95  0.54    2.31   1.70  0.57
148.00  94.41  0.45    1.94   1.13  0.45
124.45  93.96  0.46    1.64   0.68  0.37
104.65  93.50  0.54    1.38   0.31  0.31
 88.00  92.96  0.64    1.16   0.00  0.00
 74.00  92.32  0.78    0.97   0.00  0.00
 62.23  91.54  0.96    0.82   0.00  0.00
 52.33  90.58  1.24    0.69   0.00  0.00
 44.00  89.34  1.73    0.58   0.00  0.00
 37.00  87.61  2.66    0.49   0.00  0.00
 31.11  84.95  4.35    0.41   0.00  0.00
 26.16  80.60  6.93    0.34   0.00  0.00
 22.00  73.67  9.48    0.29   0.00  0.00
 18.50  64.19 10.54    0.24   0.00  0.00
 15.56  53.65  9.88    0.20   0.00  0.00
 13.08  43.77  8.72    0.17   0.00  0.00
 11.00  35.05  7.86    0.14   0.00  0.00
```

1.

- MICROTRAC -

- X100 PARTICLE ANALYZER -

Distr/Progr: Volume/Standard
Date: 04/25/96 Time: 11:46
Sample ID 1: B-10
Above Residual: 0.00/NR
Sample ID 2:
Below Residual: 0.00



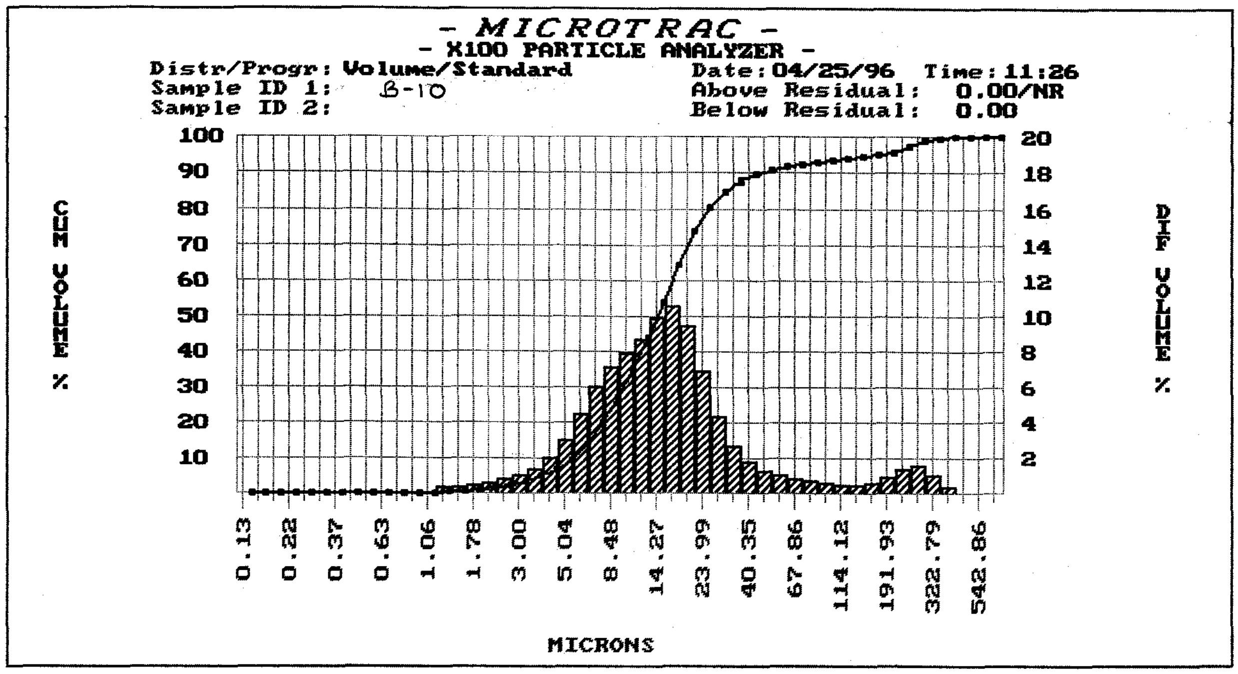
```
                 - MICROTRAC X100 PARTICLE ANALYZER -
                         Percent Passing Data
                             Version 4.13

    B-10     (fresh sludge)                      ASVR Parameters

                                            Flow Rate: n/a ml/sec
                                            Ultrasonic Power: n/a watts
                                            Ultrasonic Time: n/a seconds

 Id #1:                        Summary Data     Id #2:
 Distrib. Format: Volume       dv  = 0.0514     Date: 04/25/96   Time: 11:46
 Filter: On                    10% =   6.67     Chan. Progression: Standard
 Run Time:  60 seconds         50% =  17.59     Upper Channel Edge: 704.00
 Run Number:   1 of  1 runs    90% =  73.34     Lower Channel Edge:   0.12
 Transmission: 0.98            mv  =  35.21     Number of Channels:  50
 Laser Int: 1.020/1.006/1.006  mn  =   4.02     Fluid Refractive Index: n/a
 Residuals: Disabled           ma  =  13.23     Transparent Particles: No
 Above Residual:    0.00       cs  =  0.454     Spherical Particles: n/a
 Below Residual:    0.00       sd  =  17.56     Part. Refractive Index: n/a

ch top  %pass %-chn ch top  %pass %-chn ch top  %pass %-chn ch top  %pass %-chn
704.00 100.00  0.00   9.25  19.42  5.53
591.99 100.00  0.00   7.78  13.89  4.32
497.80 100.00  0.00   6.54   9.57  3.09
418.60 100.00  0.00   5.50   6.48  2.08
352.00 100.00  0.00   4.62   4.40  1.40
296.00 100.00  2.23   3.89   3.00  0.98
248.90  97.77  1.67   3.27   2.02  0.72
209.30  96.10  1.18   2.75   1.30  0.54
176.00  94.92  0.92   2.31   0.76  0.42
148.00  94.00  0.85   1.94   0.34  0.34
124.45  93.15  0.89   1.64   0.00  0.00
104.65  92.26  1.01   1.38   0.00  0.00
 88.00  91.25  1.18   1.16   0.00  0.00
 74.00  90.07  1.45   0.97   0.00  0.00
 62.23  88.62  1.88   0.82   0.00  0.00
 52.33  86.74  2.52   0.69   0.00  0.00
 44.00  84.22  3.39   0.58   0.00  0.00
 37.00  80.83  4.56   0.49   0.00  0.00
 31.11  76.27  6.07   0.41   0.00  0.00
 26.16  70.20  7.89   0.34   0.00  0.00
 22.00  62.31  9.40   0.29   0.00  0.00
 18.50  52.91  9.86   0.24   0.00  0.00
 15.56  43.05  9.11   0.20   0.00  0.00
 13.08  33.94  7.86   0.17   0.00  0.00
 11.00  26.08  6.66   0.14   0.00  0.00
```



- MICROTRAC -

- X100 PARTICLE ANALYZER -

Distr/Progr: Volume/Standard
Sample ID 1: B-10
Sample ID 2:

Date: 04/25/96 Time: 11:26
Above Residual: 0.00/NR
Below Residual: 0.00

```
                 - MICROTRAC X100 PARTICLE ANALYZER -
                          Percent Passing Data
                             Version 4.13

 T-11  Treatment plant                       ASVR Parameters
 Fresh sludge
                                        Flow Rate: n/a ml/sec
                                        Ultrasonic Power: n/a watts
                                        Ultrasonic Time: n/a seconds

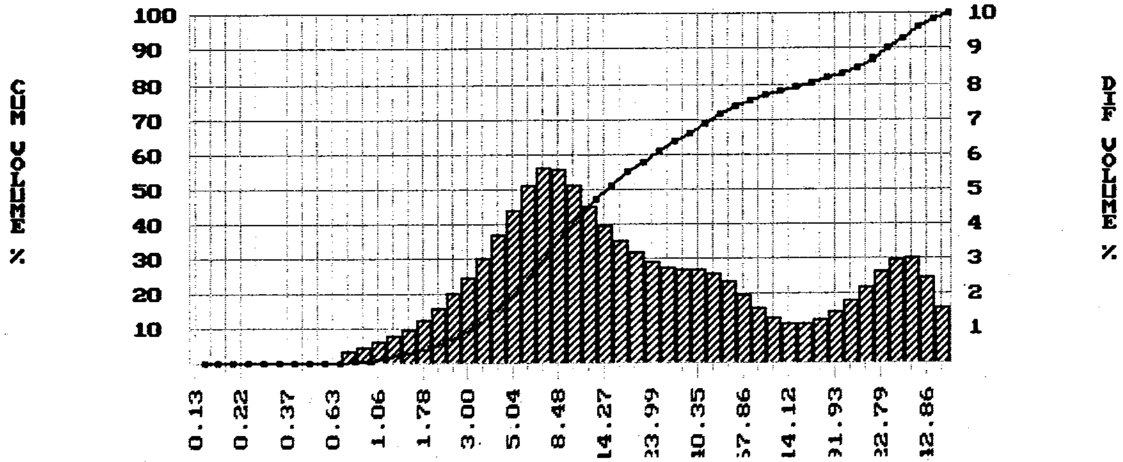
 Id #1:                      Summary Data   Id #2:
 Distrib. Format: Volume     dv  = 0.0781   Date: 04/30/96   Time: 11:15
 Filter: On                  10% =   3.18   Chan. Progression: Standard
 Run Time:  60 seconds       50% =  14.63   Upper Channel Edge: 704.00
 Run Number:   1 of  1 runs  90% = 351.57   Lower Channel Edge:   0.12
 Transmission: 0.94          mv  =  88.76   Number of Channels:  50
 Laser Int: 1.021/1.006/1.007 mn =   1.45   Fluid Refractive Index: n/a
 Residuals: Disabled         ma  =   7.78   Transparent Particles: No
 Above Residual:    0.00     cs  =  0.772   Spherical Particles: n/a
 Below Residual:    0.00     sd  = 108.33   Part. Refractive Index: n/a

ch top  %pass %-chn|ch top  %pass  %-chn|ch top   %pass %-chn|ch top   %pass %-chn
704.00 100.00  1.54|  9.25  37.79  5.55|
591.99  98.46  2.47|  7.78  32.24  5.62|
497.80  95.99  3.00|  6.54  26.62  5.13|
418.60  92.99  2.97|  5.50  21.49  4.40|
352.00  90.02  2.60|  4.62  17.09  3.65|
296.00  87.42  2.17|  3.89  13.44  3.01|
248.90  85.25  1.77|  3.27  10.43  2.46|
209.30  83.48  1.45|  2.75   7.97  1.98|
176.00  82.03  1.22|  2.31   5.99  1.57|
148.00  80.81  1.10|  1.94   4.42  1.23|
124.45  79.71  1.11|  1.64   3.19  0.97|
104.65  78.60  1.27|  1.38   2.22  0.78|
 88.00  77.33  1.57|  1.16   1.44  0.61|
 74.00  75.76  1.95|  0.97   0.83  0.47|
 62.23  73.81  2.31|  0.82   0.36  0.36|
 52.33  71.50  2.56|  0.69   0.00  0.00|
 44.00  68.94  2.64|  0.58   0.00  0.00|
 37.00  66.30  2.68|  0.49   0.00  0.00|
 31.11  63.62  2.74|  0.41   0.00  0.00|
 26.16  60.88  2.89|  0.34   0.00  0.00|
 22.00  57.99  3.14|  0.29   0.00  0.00|
 18.50  54.85  3.50|  0.24   0.00  0.00|
 15.56  51.35  3.95|  0.20   0.00  0.00|
 13.08  47.40  4.51|  0.17   0.00  0.00|
 11.00  42.89  5.10|  0.14   0.00  0.00|
```

- MICROTRAC -

- X100 PARTICLE ANALYZER -

Distr/Progr: Volume/Standard Date:04/30/96 Time:11:15
Sample ID 1: T-11 Above Residual: 0.00/NR
Sample ID 2: Below Residual: 0.00



```
                 - MICROTRAC X100 PARTICLE ANALYZER -
                        Percent Passing Data
                            Version 4.13

 T-11 Treatment plant                      |        ASVR Parameters
 Fresh sludge                              |
                                           | Flow Rate: n/a ml/sec
                                           | Ultrasonic Power: n/a watts
                                           | Ultrasonic Time: n/a seconds

 Id #1:                      | Summary Data    | Id #2:
 Distrib. Format: Volume     | dv  = 0.1162    | Date: 04/30/96   Time: 11:35
 Filter: On                  | 10% =   3.57    | Chan. Progression: Standard
 Run Time:  60 seconds       | 50% =  26.81    | Upper Channel Edge: 704.00
 Run Number:   1 of  1 runs  | 90% = 335.99    | Lower Channel Edge:   0.12
 Transmission: 0.92          | mv  = 101.69    | Number of Channels:  50
 Laser Int: 1.021/1.005/1.006| mn  =   1.44    | Fluid Refractive Index: n/a
 Residuals: Disabled         | ma  =   9.32    | Transparent Particles: No
 Above Residual:   0.00      | cs  =  0.644    | Spherical Particles: n/a
 Below Residual:   0.00      | sd  = 110.37    | Part. Refractive Index: n/a

ch top  %pass %-chn|ch top  %pass %-chn|ch top  %pass %-chn|ch top  %pass %-chn
704.00 100.00  1.48|  9.25  30.22  4.05|                   |
591.99  98.52  2.25|  7.78  26.17  4.18|                   |
497.80  96.27  2.71|  6.54  21.99  4.03|                   |
418.60  93.56  2.82|  5.50  17.96  3.59|                   |
352.00  90.74  2.70|  4.62  14.37  3.05|                   |
296.00  88.04  2.59|  3.89  11.32  2.53|                   |
248.90  85.45  2.59|  3.27   8.79  2.06|                   |
209.30  82.86  2.76|  2.75   6.73  1.66|                   |
176.00  80.10  3.00|  2.31   5.07  1.31|                   |
148.00  77.10  3.23|  1.94   3.76  1.04|                   |
124.45  73.87  3.30|  1.64   2.72  0.83|                   |
104.65  70.57  3.21|  1.38   1.89  0.66|                   |
 88.00  67.36  2.98|  1.16   1.23  0.52|                   |
 74.00  64.38  2.69|  0.97   0.71  0.40|                   |
 62.23  61.69  2.41|  0.82   0.31  0.31|                   |
 52.33  59.28  2.25|  0.69   0.00  0.00|                   |
 44.00  57.03  2.25|  0.58   0.00  0.00|                   |
 37.00  54.78  2.45|  0.49   0.00  0.00|                   |
 31.11  52.33  2.73|  0.41   0.00  0.00|                   |
 26.16  49.60  2.98|  0.34   0.00  0.00|                   |
 22.00  46.62  3.08|  0.29   0.00  0.00|                   |
 18.50  43.54  3.10|  0.24   0.00  0.00|                   |
 15.56  40.44  3.16|  0.20   0.00  0.00|                   |
 13.08  37.28  3.36|  0.17   0.00  0.00|                   |
 11.00  33.92  3.70|  0.14   0.00  0.00|                   |
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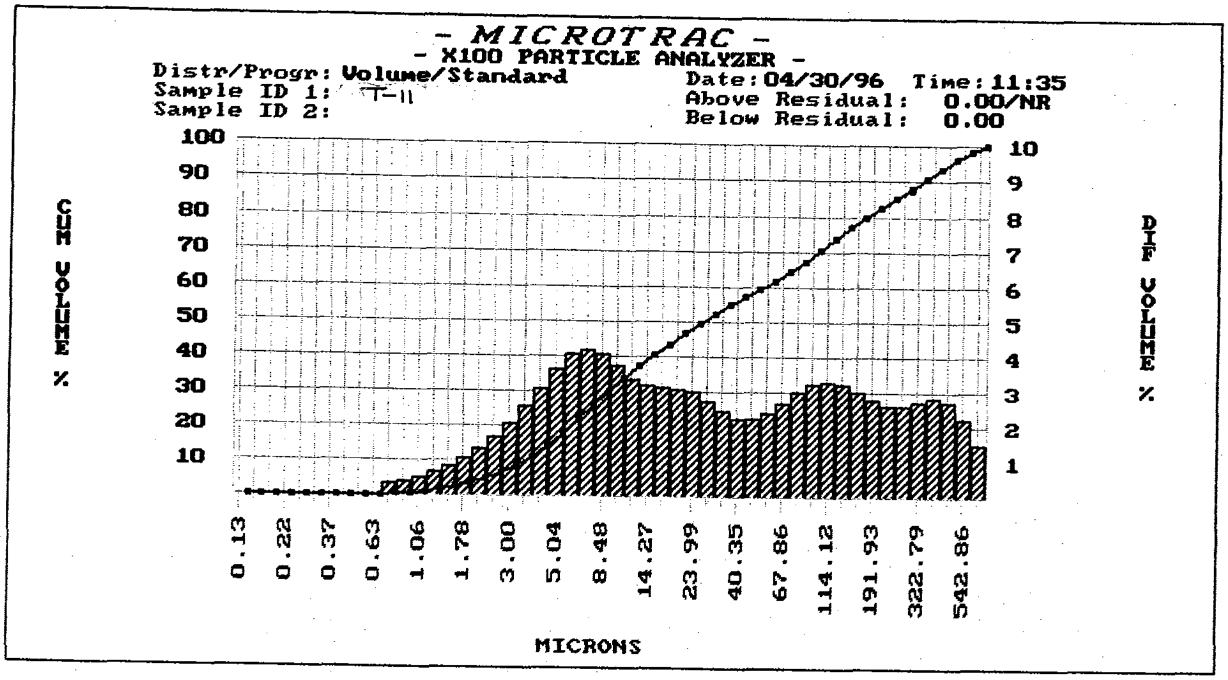


- MICROTRAC -

- X100 PARTICLE ANALYZER -

Distr/Progr: Volume/Standard
Sample ID 1: T-11
Sample ID 2:

Date: 04/30/96 Time: 11:35
Above Residual: 0.00/NR
Below Residual: 0.00

APPENDIX G: MINERALOGICAL ANALYSES

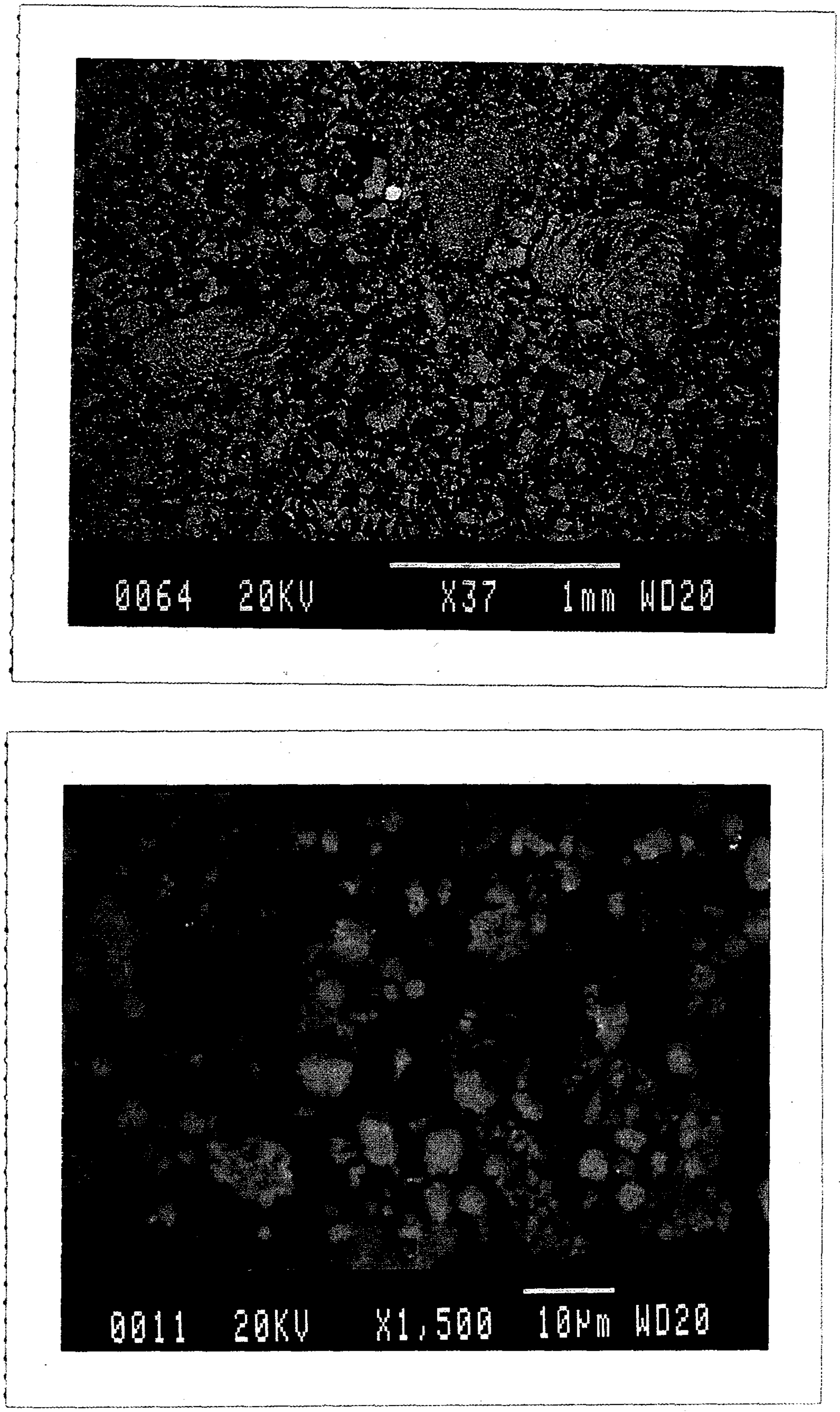
This appendix contains the mineralogical data (SEM / XRD) and is organized by site code. A description of the mineralogical findings for each site is followed by the photomicrograph plates referred to in the text. The plate number is located in the lower left hand corner of the photomicrograph. The XRD patterns for each sample are also included and follow the photomicrographs.

Site D-1

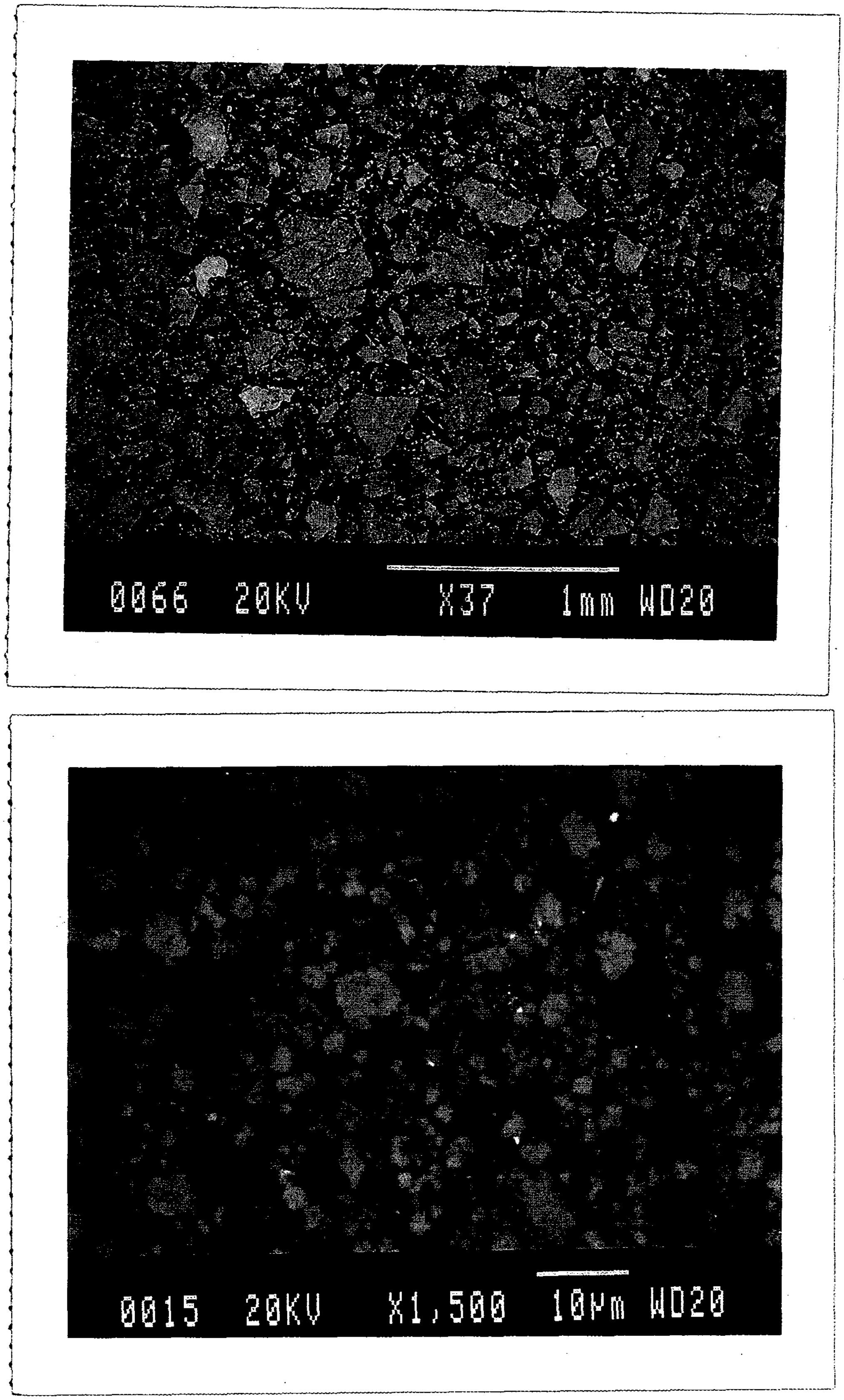
Plate 64 shows the general morphology of the fresh sludges. Except for a few larger calcite (bright) particles, the sludge is fine -grained and uniform. Plate 11 shows that the calcite (<1 - 8μm in size) is the major constituent of the sludge. The fine-grained matrix is an amorphous mass and consists mainly of Ca, Mg, Fe, Si, C, S, O, and traces of Mn, Al. XRD analysis indicates that the sludge is mainly crystalline calcite, only a small amount occurs as amorphous compounds. Therefore, most of the elements, except Ca, are carried in these amorphous compounds. The silica-rich nature of the amorphous compound(s) likely has immobilized the elements in the sludge.

Plate 66 shows the general morphology of the aged sludges. Except for a trace amount of silicate particles (light grey) such as quartz, K-Al silicate, Fe-Mg-Ca silicate and Fe-Mg silicate, the sludge consists of a fine-grained mixture (dark grey) mainly calcite and a minor amorphous phase. Plate 15 shows the details of the fine-grained material. The light-grey particles, <1 to 8μm diameter are calcite (larger) or (Ca, Fe, Mg) CO_3 (tiny). The tiny bright grains are pyrite and Fe oxide most likely originating from the slag/tailings deposited in the area. The fine dark-grey matrix consists of major Fe, Mg, Ca, minor Si, S, O, CO_3 and trace Al, Ni and P; it is likely a silica-carbonate-sulphate rich amorphous compound. Quartz, silicate and pyrite particles are detrital in origin, whereas the remainder are precipitated in nature. XRD analyses indicate the sludge consists of calcite, trace quartz and a minor amount of the amorphous phase.

The comparison of the XRD data indicates that the aged and the fresh sludges contain approximately the same amount of the amorphous phases, indicating that the sludge mineralogy/chemistry at this site does not change significantly with time. Plate 15 and 11 also show that the particle size of the calcite crystals has not changed with aging. Most of the elements, such as Al, Fe, Mg, Na, Zn and SO_4 are captured in the amorphous phase.



Photomicrographs of sludge collected from D-1 (refer to text).



Photomicrographs of sludge collected from D-1 (refer to text).

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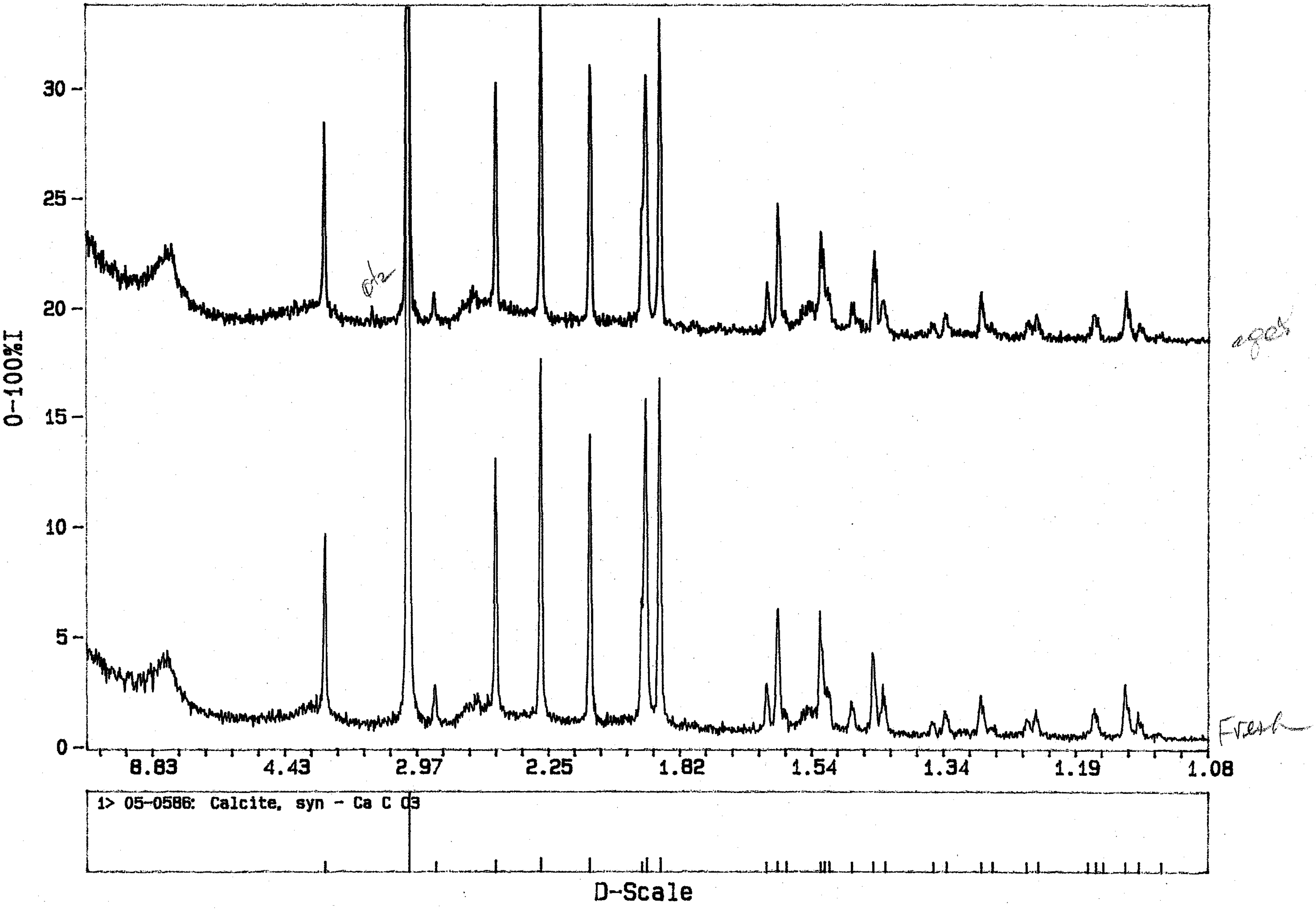
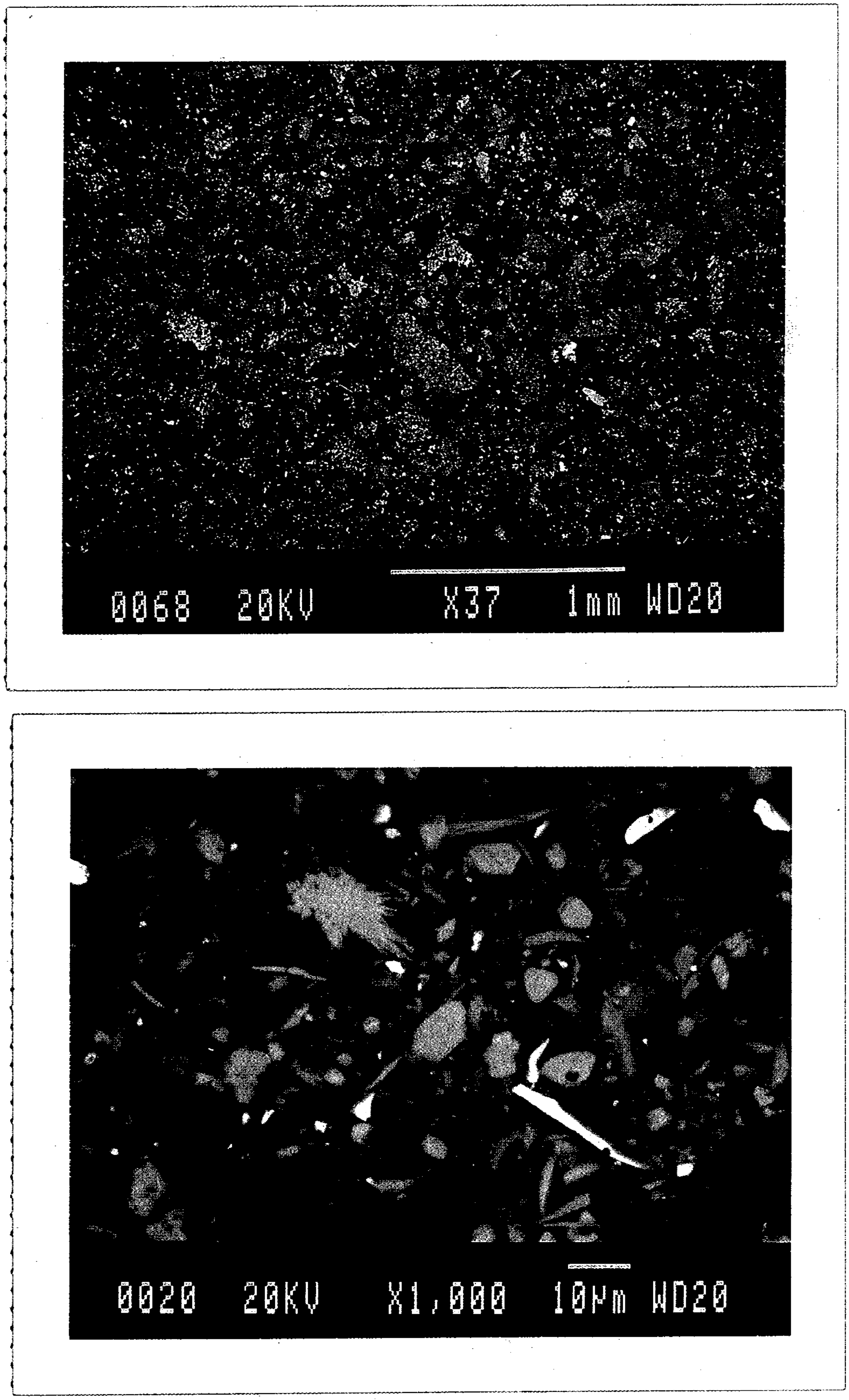


CANMET/MSL

Site Q-2

In addition to the precipitated compounds, the sludge from Q-2 contains a lot (10%) of detrital particles such as pyrite, chalcopyrite and silicates (quartz, clinoclore - Mg-Fe-Al silicate, talc - Mg silicate, feldspar - K-Al silicate). Plate 68 illustrates the general morphology of the sludge. The few "larger" bright particles are quartz, silicates and calcite; the tiny bright particles in the general precipitated masses (grey) are mostly calcite, with traces of pyrite and chalcopyrite. In general, the brighter masses contain more calcite. The details of these precipitated masses are shown in Plate 20. Calcite (coarser, grey crystals) is the major compound, the needle-like particles are silicates (clinoclore and minor talc); the bright angular particles are pyrite and chalcopyrite. The silicates, pyrite and chalcopyrite are detrital in origin. The darker fine grained matrix is mainly Ca, Fe, Mg, CO_3, Si, SO_4, Zn, Na, Cl in composition and is presumably amorphous in nature. Trace gypsum and ankerite (Ca, Fe, Mg, Mn)-carbonate are also present in the precipitated mass. The precipitates are generally $< 1\ \mu m$ in size, the detrital grains vary from <1 to 30 μm; and the calcite crystals range from < 1 to 20 μm.

In contrast to the other sludge samples, XRD analysis of this sludge sample shows the presence of a large amount of detrital phase (quartz, clinoclore, talc and pyrite), in addition to the normal sludge constituents of calcite, ankerite and gypsum. Attempts were made prior to characterization to remove much of this detrital material through centrifugation. The relatively rough background of the XRD pattern indicates a major proportion of the sample is in the amorphous phase, as shown in Plate 20. Most of the elements (Al, Ca, Fe, CO_3, Mg, Na, Si, S^{2-}, SO_4, Zn) occur as carbonates, silicates or sulphide minerals in the sludge. Silicates or sulphide minerals are detrital in origin, whereas carbonates are recrystallized in nature. The fine-grained amorphous precipitates contain minor amounts of silicates and carbonates, presumably the components have stabilized the other minor elements (such as soluble Zn and Cu) in the sludge.



Photomicrographs of sludge collected from Q-2 (refer to text).

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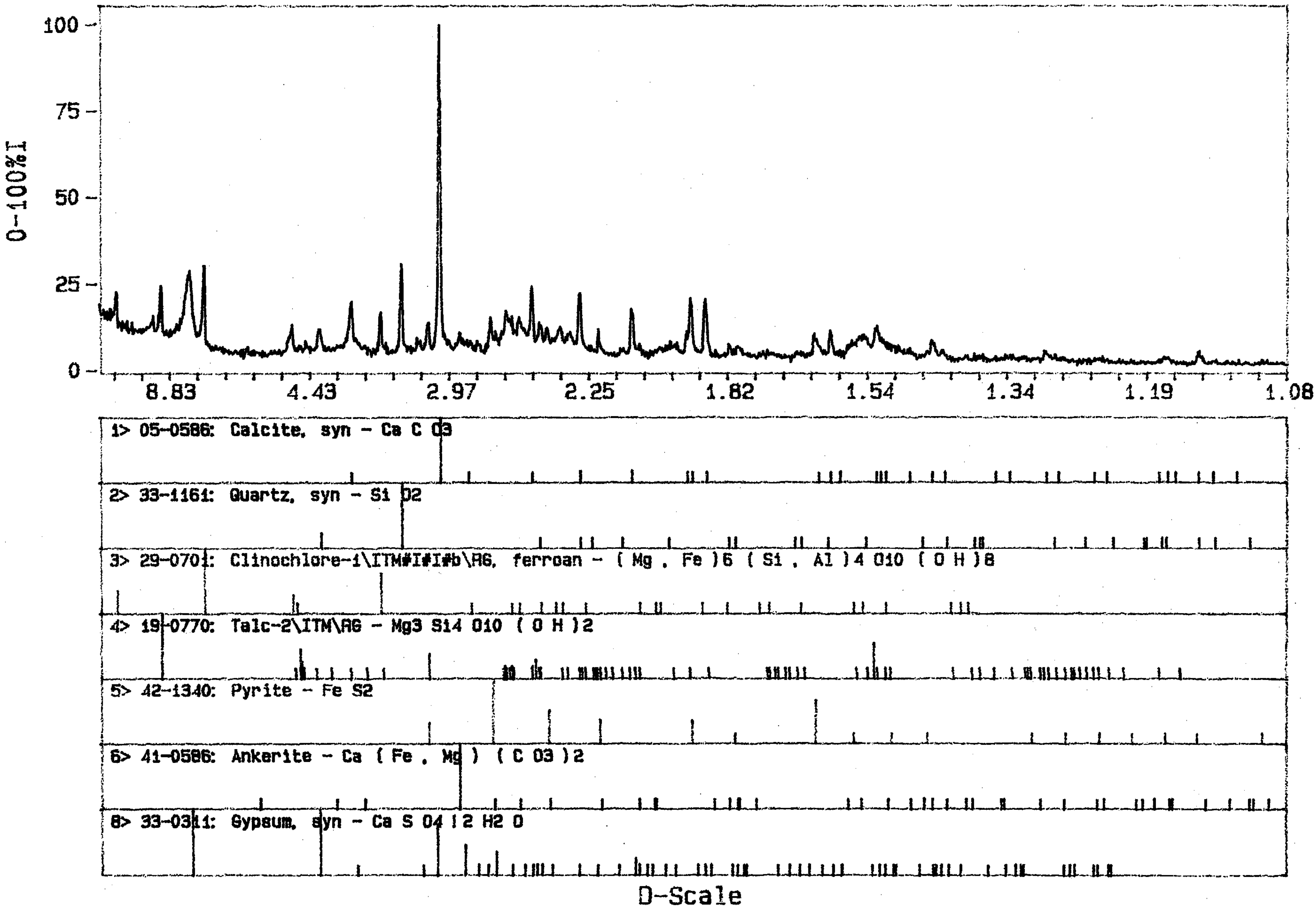
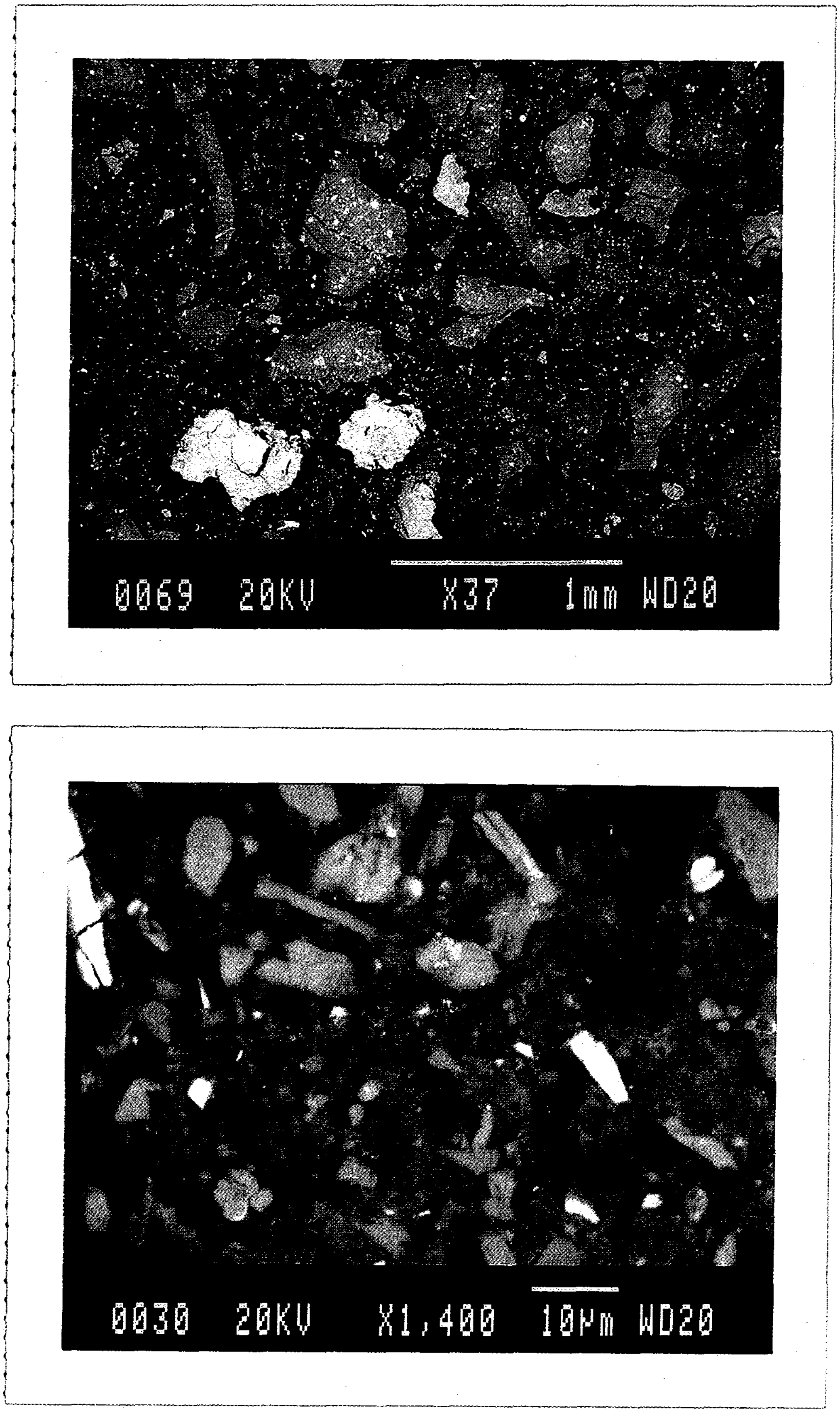


CANMET/MSL

Site M-3

Plate 69 illustrates the general morphology of the sludges. Although the sludge particles show different grey-level they are different mainly in water content; they contain the same elements. The details of the sludge particles are shown in Plate 30. In general, the sludge consists of a minor amount of calcite (light grey, 1 - 9 μm); trace tiny particles of $(Mg,Fe,Ca)CO_3$, quartz, pyrite (bright), ZrSiO4 (bright), Fe oxide and Fe-Ca-Mg-Al silicate in a major very fine-grained matrix major Fe, Mg, O, C, minor Si, Na, Ca, Zn, S, Ni, Al, Mn, Cl in composition. All the silicate and sulphide particles are detrital in origin; the carbonates are recrystallized and the fine-grained matrix is amorphous in nature. XRD analysis also shows the major presence of amorphous compounds in the sludge. Only calcite and trace quartz are detected by X-ray, the other sulphides and silicates are too low in quantity to be detected by X-ray. Bulk analyses indicate that the sludge contains significant amount of Ca, Fe, Mg, Si, S, and Na. A major proportion of the elements (Ca, Fe, Zn, S, Mg, Al) are present as carbonates, silicates and sulphide minerals in the sludge; the remainder occur in the amorphous precipitates. Ni is only detected in the amorphous phase. The leachable nature of nickel may imply its presence as Ni sulphate in the amorphous phase.



Photomicrographs of sludge collected from M-3 (refer to text).

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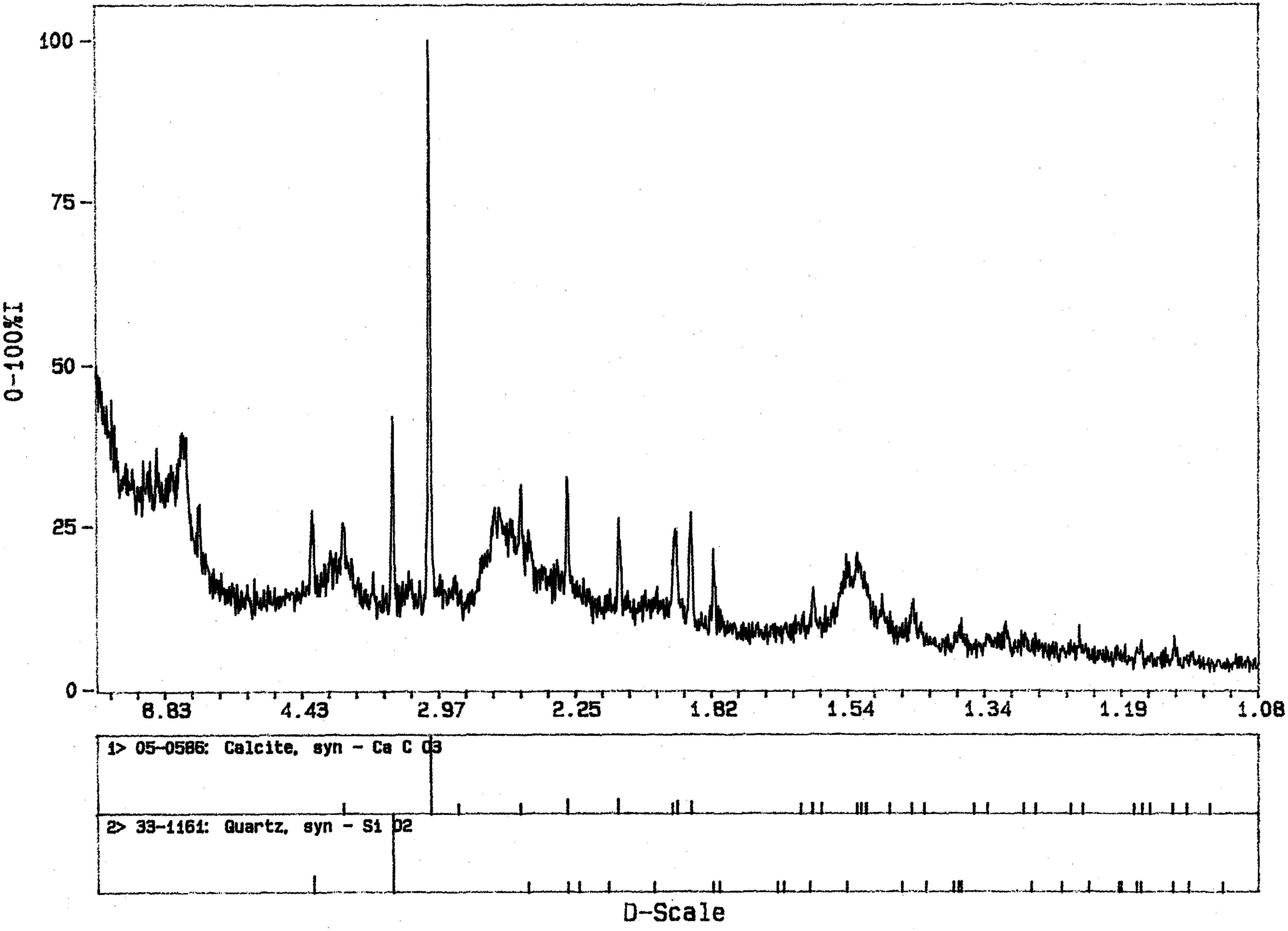


Site: R-4

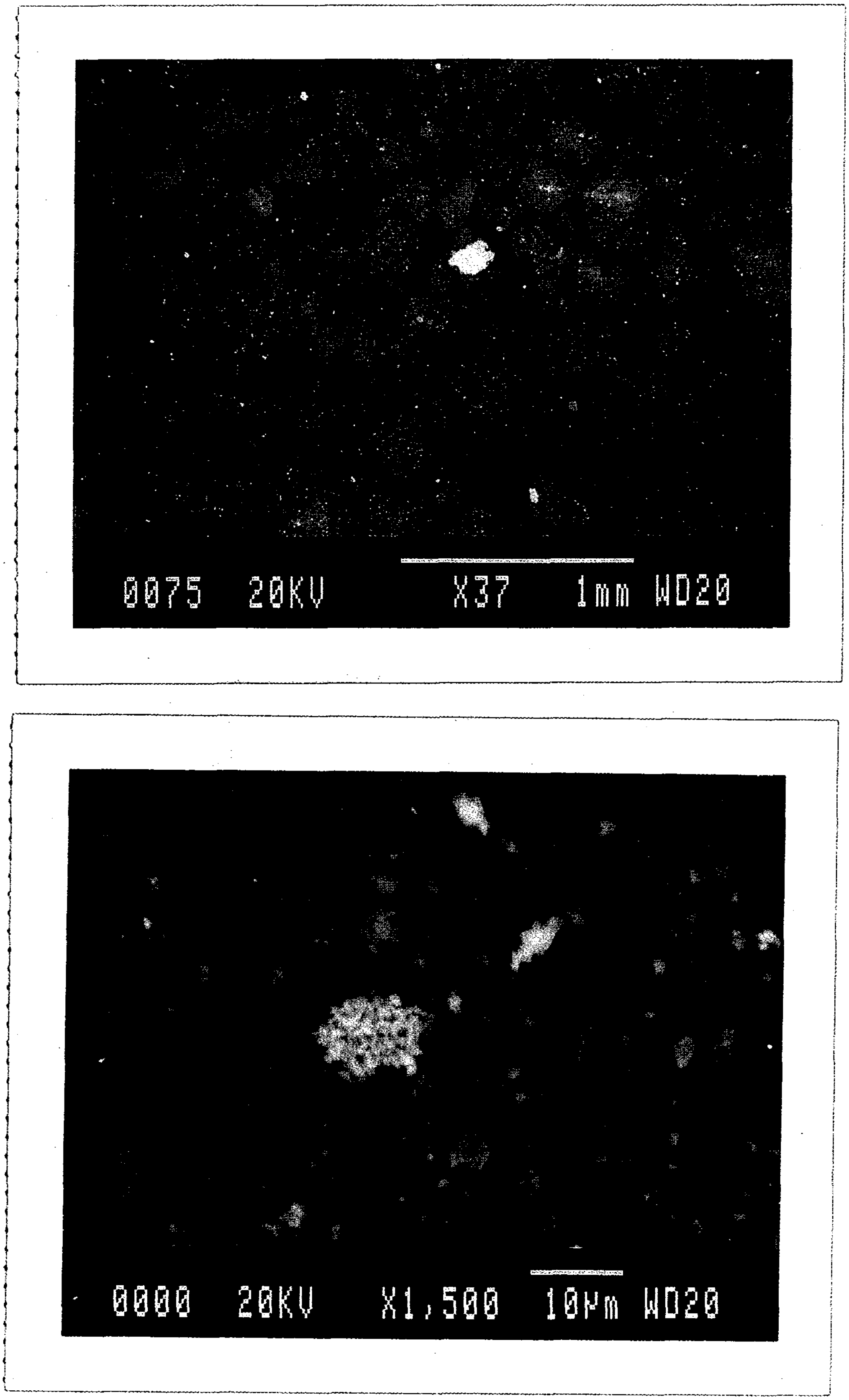
Plate 75 shows the general morphology of the fresh sludge. The material consists of a major fine-grained mixture (grey) and trace calcite particles. XRD analysis indicates that calcite is the only crystalline compound; the fine-grained mixture is mainly amorphous. The fine-grained mixture consists of minor amounts of calcite, Mg-Al-Fe silicate, quartz, Fe oxide, and a major amount of amorphous compound consisting of major Mg, Al, Si, Fe minor S and Ca and trace Zn and Mn. The material is believed to be hydrated. Traces of gypsum may be present in the mixture. All silicate particles are detrital in origin, whereas the remainder are precipitates in nature. The amorphous compounds are usually < 1 μm in size, but tend to agglomerate into a larger porous mass. Wet chemical analysis shows the sludge contains significant amount of Al, Ca, Fe, Mg, Si, SO_4 and Zn. Ca occurs as calcite and in the amorphous phase; Fe, Mg, Al and Si occur as silicates and as amorphous compounds; Zn is mainly in the amorphous phase. Sulphate is present in the amorphous phase, likely as Ca-, or M (metal) - SO4 compounds.

Plate 77 shows the general morphology of the blended aged sludge. The sample consists of major fine-grained compounds and a few larger silicate particles (bright). The large bright grains are Fe-Ca-Mg-Al silicate and Na-Fe-Mg-Al silicate; the tiny bright grains are calcite particles. The fine-grained mixture is mainly Fe-Mg-Al-Si-Ca-S-O and trace Zn, Mn in composition.

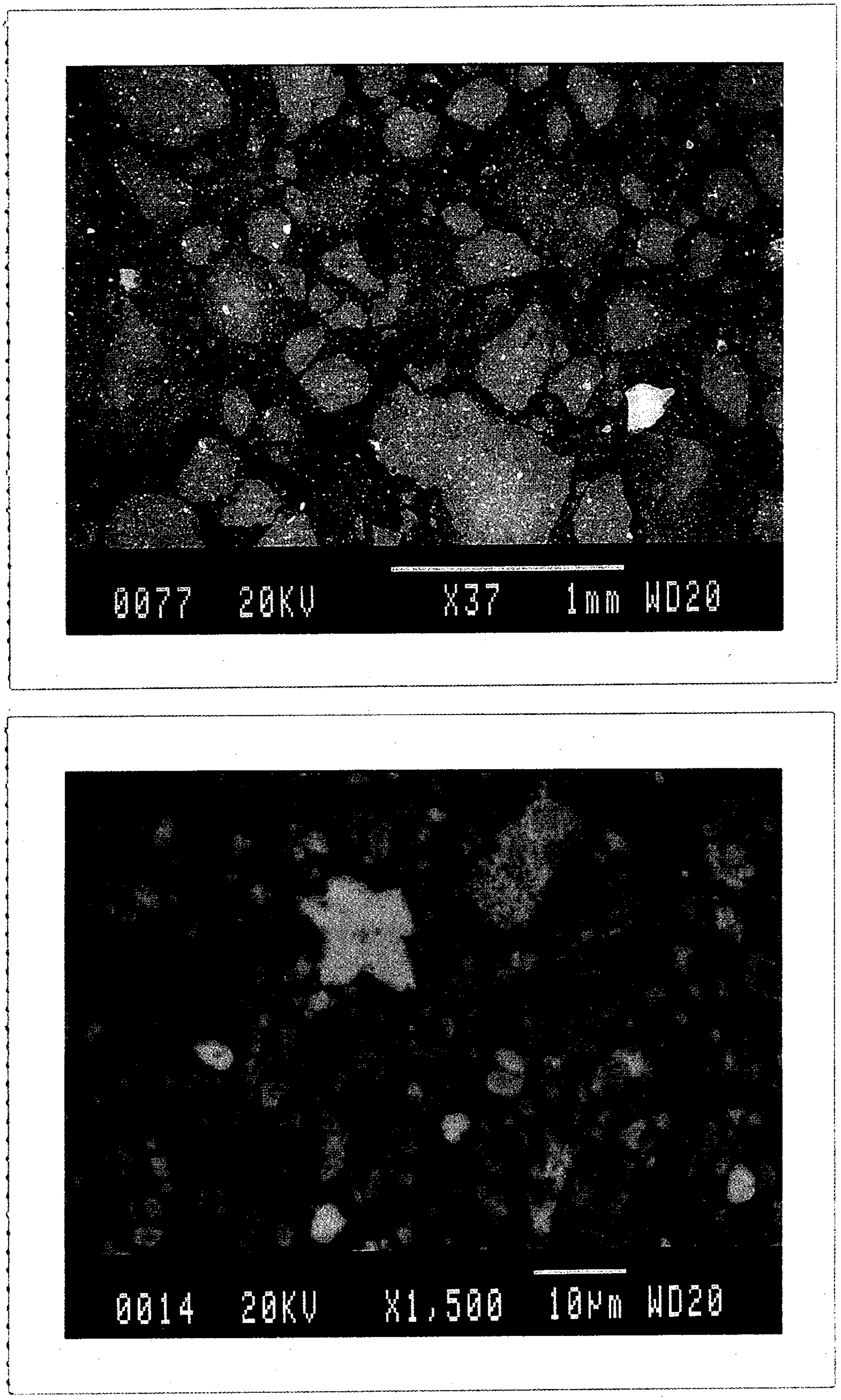
XRD analysis shows that the material contains a trace quantity of calcite and a large amount of an amorphous compound. Silicate phases are too low in quantity to be detected by XRD. Comparison of the XRD patterns indicates that the fresh sludge contains a lot more amorphous material, whereas the aged sludge contains more recrystallized calcite; the quartz is detrital. This implies that the amorphous sludge compounds in the fresh precipitate will gradually recrystallize to form more calcite with time.

Plate 14 shows the details of the fine-grained material. The material consists of "larger" calcite grains, tiny Fe-Mg-Al silicates, quartz particles and a fine-grained mass consisting of Fe-Mg-Al-Si-Ca-S-O and trace Mn and Zn. Presumably all the amorphous compounds are hydrated. The recrystallized nature (star-like) of the calcite particles is obvious. One porous grain, 5 x 10μm size, appears to be a Ca-O-H compound, which may be an aged unreacted lime particle. The particle size of this fine-grained material is approximately 1 μm and up. Comparison of Plate 14 with Plate 00, the particle sizes of the aged material. A comparison of Plate 14 and Plate 00 shows that the particle sizes of the aged material, particularly calcite, appears to be slightly larger. This is likely due to recrystallization and grain growth.

Wet chemical analysis indicates that the sludge contains significant amounts of Al, Ca, Fe, CO_2, Mg, Si, SO_4 and Zn. Fe, Mg, Al, Ca, Si, SO_4, Mn, and Zn are mainly carried in the amorphous phase. A major amount of Fe, Mg, Al and Si are also present in silicates; Ca is present as $CaCO_3$. The high concentrations of Si (4.4 % bulk sludge) and CO_3 (8.2 % bulk sludge) in the amorphous phase likely have stabilized the elements in the sludge.



Photomicrographs of sludge collected from R-4 (refer to text).



Photomicrographs of sludge collected from R-4 (refer to text).

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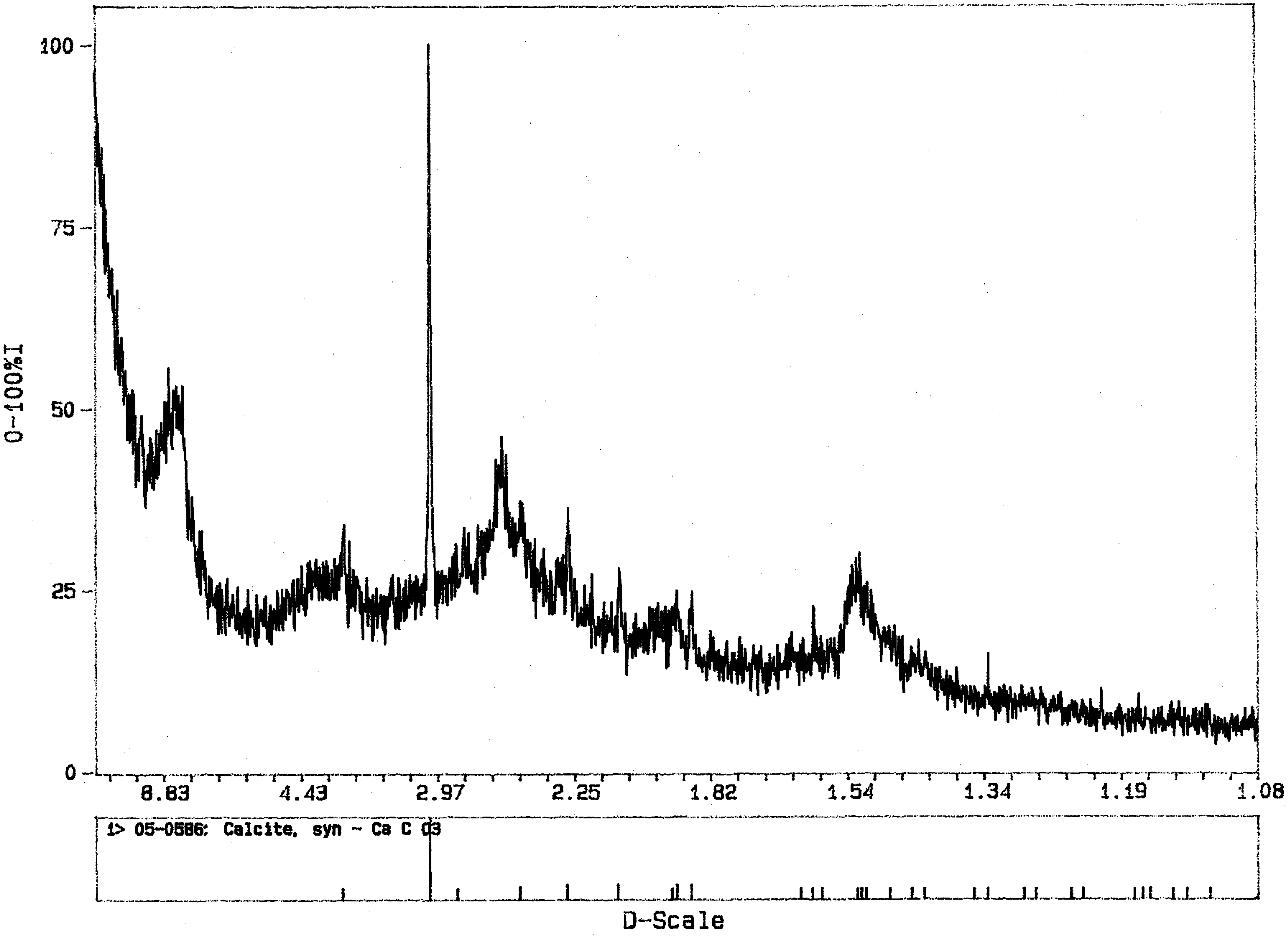

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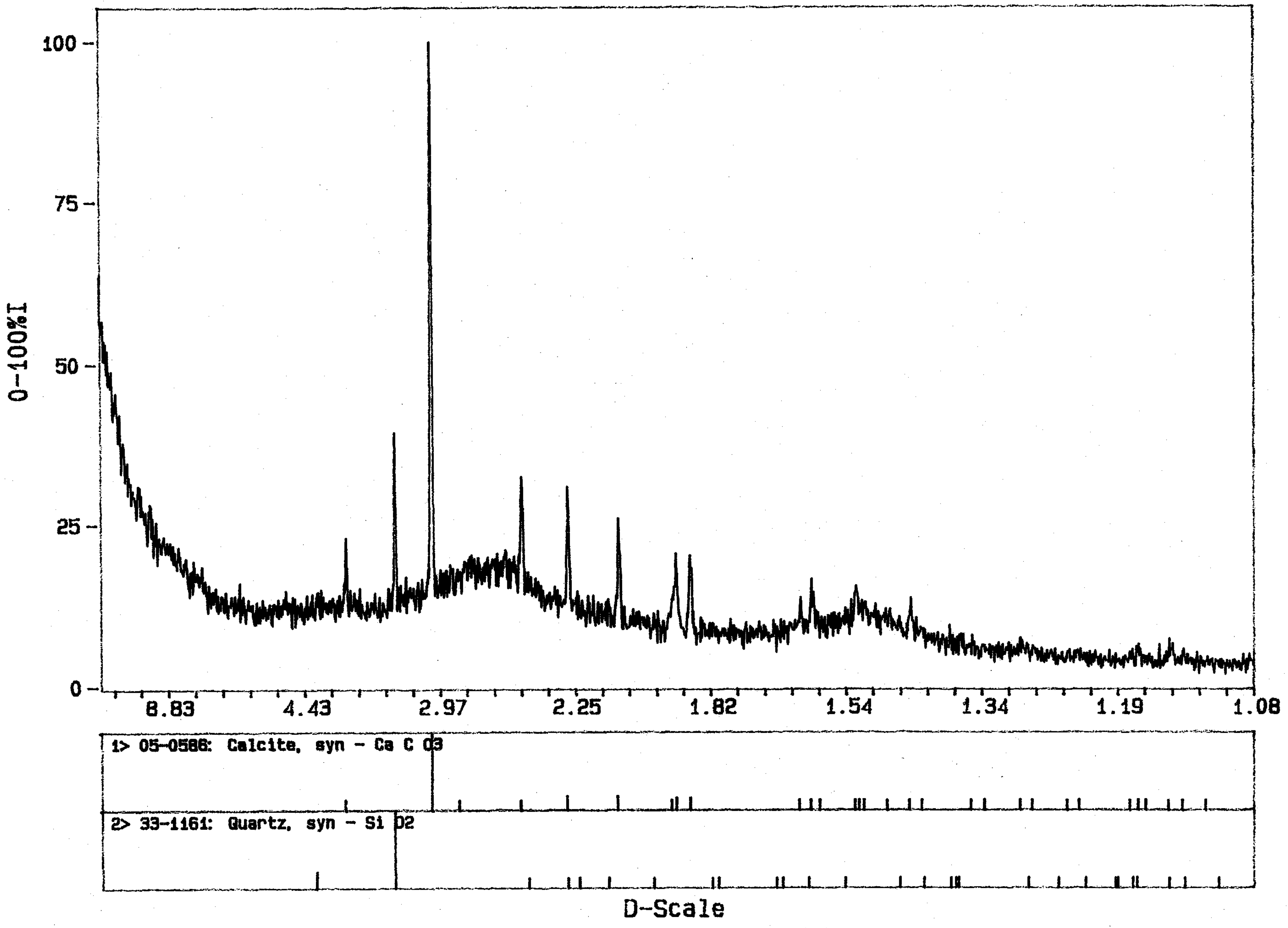

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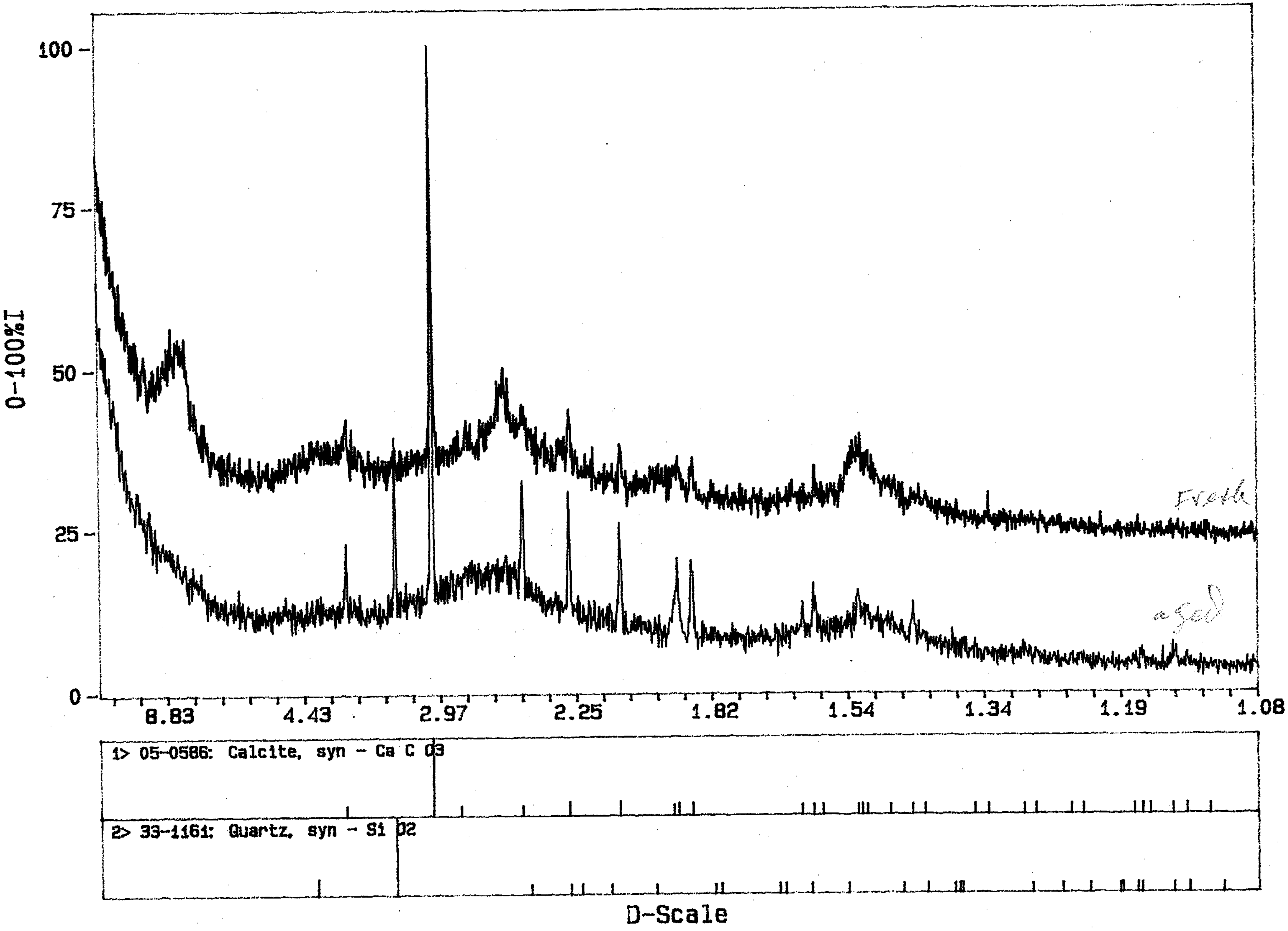


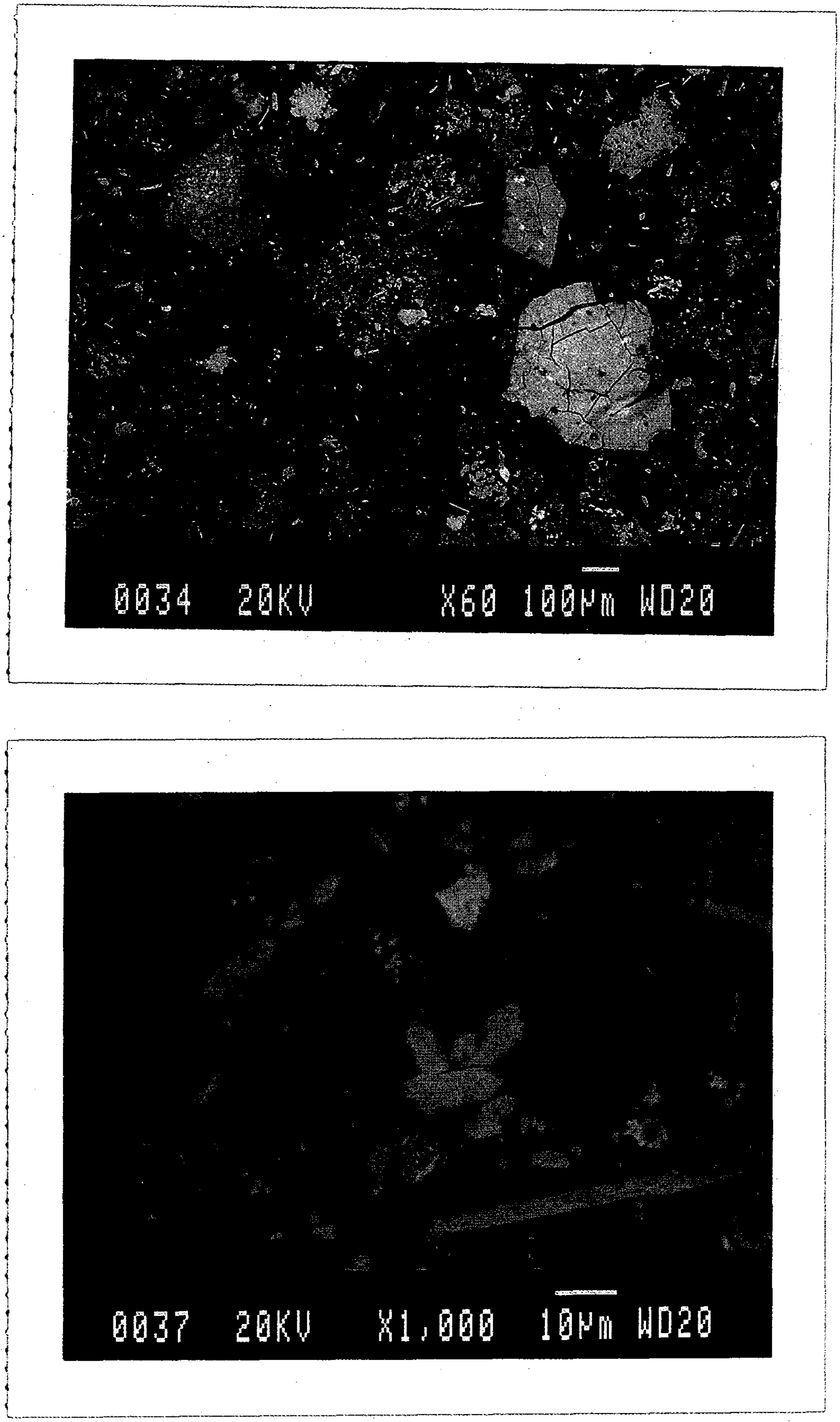
CANMET/MSL

Site F-5

The composition of the sludge from coal AMD is expected to be different from the base metal mine sludges as the mineralogy of the coal beds is quite different from those of base metal ores. Plate 34 shows the general mineralogy of the sludge. The sludge composition is heterogeneous. The large light particle on the right of the photomicrograph is a mass consisting of gypsum crystals in a matrix of major Al-O, minor S, Si, Ca, Fe, C and trace Mg, Mn, and Na. The large dark particle on the left consists of tiny gypsum, quartz and calcite particles in a matrix of mainly Al-O-C with trace Fe, Mn, Ca, S, Si, Mg. The light grey grain at the upper right corner is a cluster of K-Al silicates. In general, all the fine-grained matrix phases contain major Al-O and variable amounts of Fe, Ca, SO_4, C, Si, and trace amounts of Mg, Mn, and Na. The lighter matrix contains more Fe, Ca, and SO_4. Presumably, all the fine-grained matrix in the sludge are hydrated compounds. The tiny bright particles in the plates are mainly gypsum and calcite.

The detailed mineralogy is shown in Plate 37. The sludge consists of major gypsum (needles), minor calcite, trace quartz, Fe oxide and Mn-Al oxide, in a major amorphous fine-grained matrix of Al-O-Fe-Ca-SO_4-C-Si-Mg-Mn-Na in composition. As is shown in Plate 37, the composition of the amorphous matrix phase is very heterogenous. The dark grains are mainly Al-O-H in composition, whereas the lighter regions are richer in Ca, Fe and SO_4.

Again, the XRD analysis shows that the gypsum and calcite are the only crystalline compounds; quartz is detrital in origin. As indicated in Plate 37, the majority of the elements detected by wet chemical analysis are carried in the Al-rich very fine-grained hydrate compounds.



Photomicrographs of sludge collected from F-5 (refer to text).

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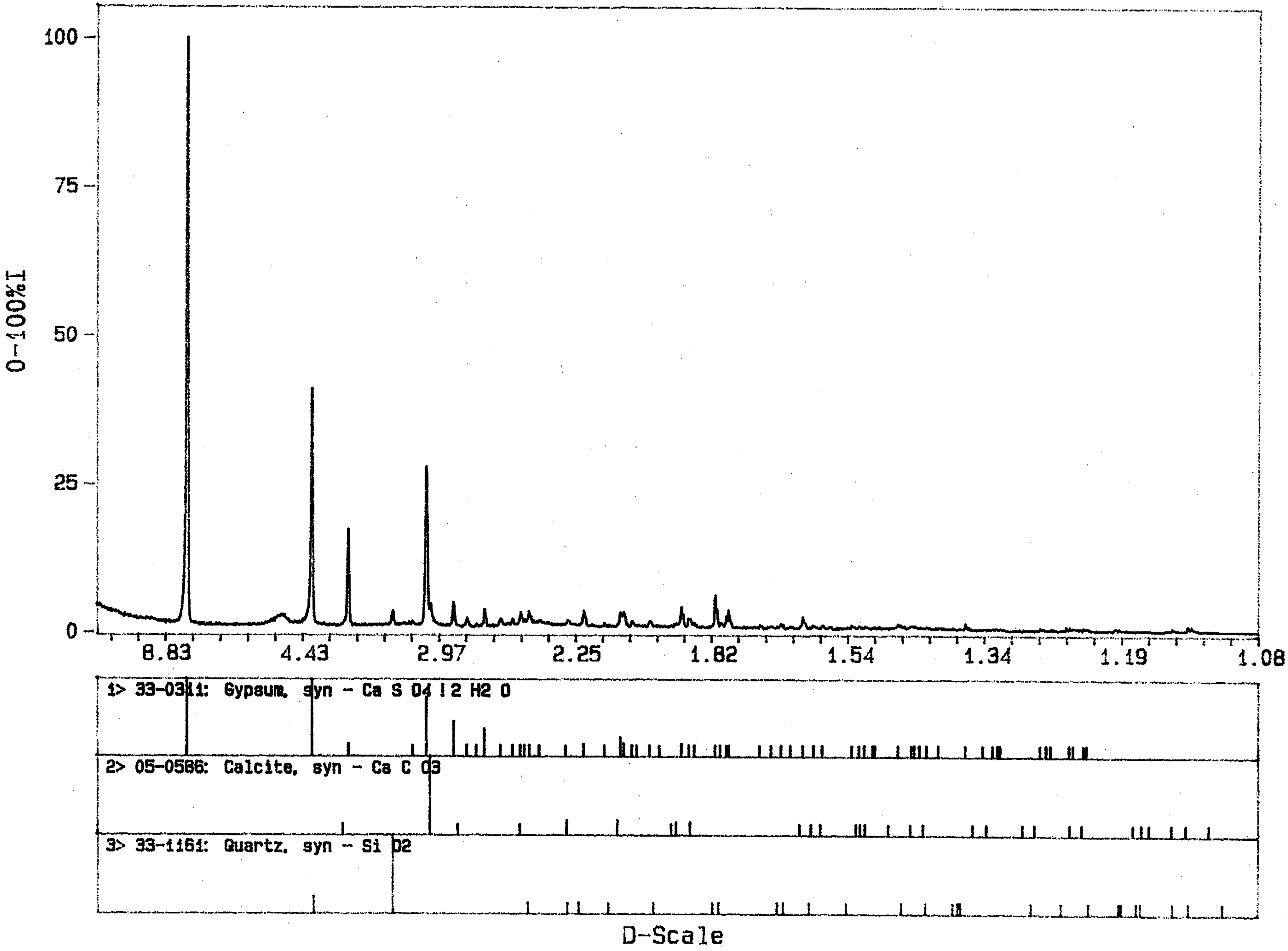


Site S-6

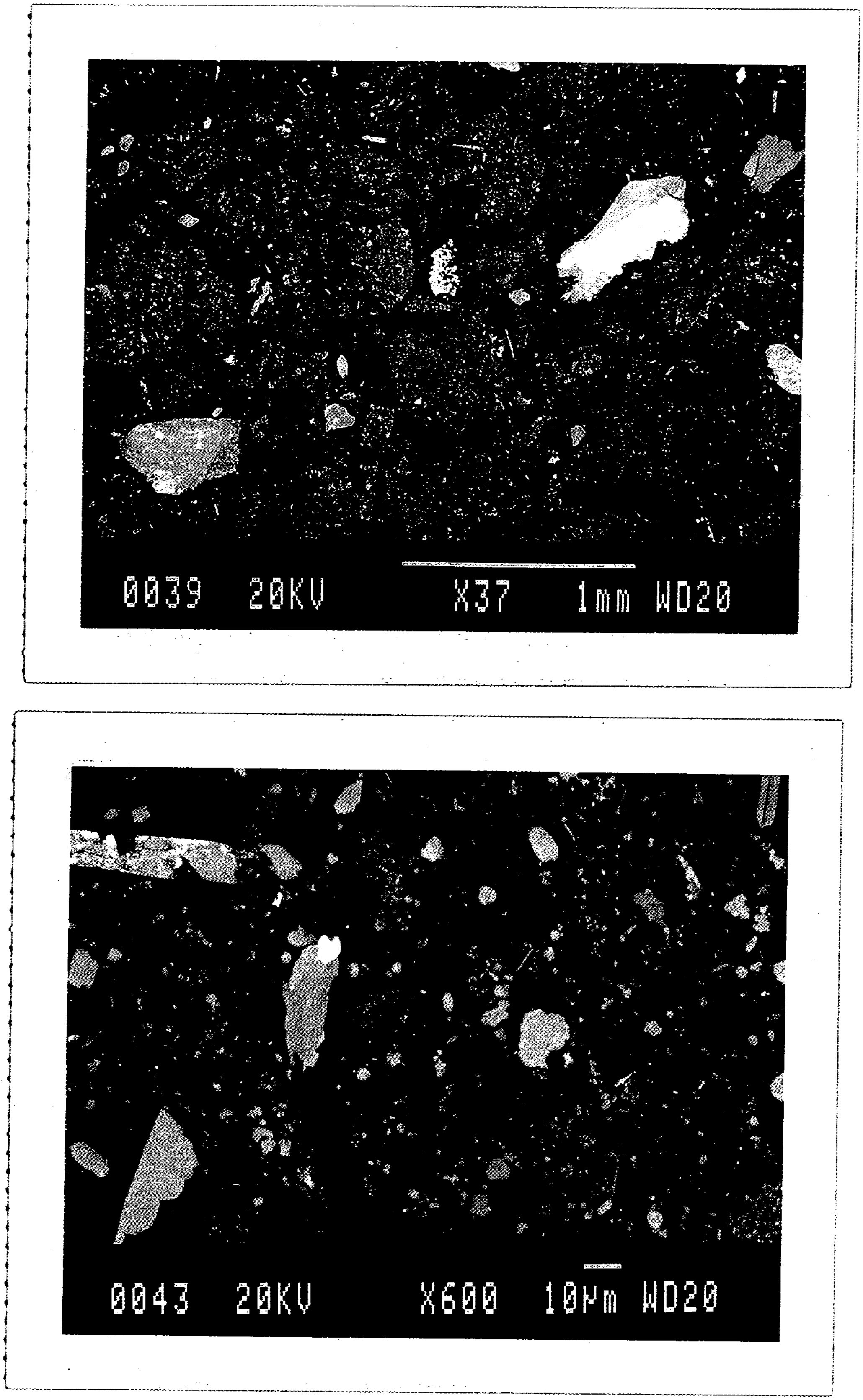
Plate 39 shows the general mineralogy of the fresh sludge. The sludge is heterogeneous, primarily because it contains various silicate particles of detrital origin. The three larger light grains along the upper right to lower left diagonal of the photo are particles of Fe-Mg-Al silicate and K-Al silicate; Al-Mg-Fe-K-Zn silicate and Ca-Na-Al silicate; and Na-Al silicate and Fe-Mg silicate. Most of the medium and tiny bright particles in the photomicrograph are either calcite or gypsum. Most of the sludge consists of amorphous compounds (dark grey) of major Mg, C, O, minor Na, Ca, S, Zn, Si, Mn, and trace Fe in composition. The cracking morphology of the amorphous compound indicates the hydrated nature of the precipitate. The detailed morphology of the sludge is shown in Plate 43. The sludge consists of tiny (grey) particles of $(Ca,Zn,Mg)CO_3$ mixed with hair-like crystals of gypsum, coarser $CaCO_3$ crystals (light grey), and large laths of gypsum (upper left) embedded in the matrix of the amorphous compounds. The bright phase in the upper left of the photomicrograph is a TiO_2 particle attached to a K-Al silicate and Fe-Mg-Al silicate particle. In general, the sludge contains a high amount of detrital particles. Other detrital particles observed include Fe-Mg-K-Na-Zn-Al silicate, Ca-Na-Al silicate, Na-Al silicate, quartz, Fe-Mn oxide and more rarely, pyrite and chalcopyrite.

XRD analysis indicates that much of the silicates are clinoclore, quartz, muscovite and plagioclase. Wet chemical analysis shows a relatively low Fe and relatively high Zn content in the sludges. Most of the Fe is present as silicates and Zn is carried the amorphous phase which is mainly a carbonate-sulphate-hydrate compound.

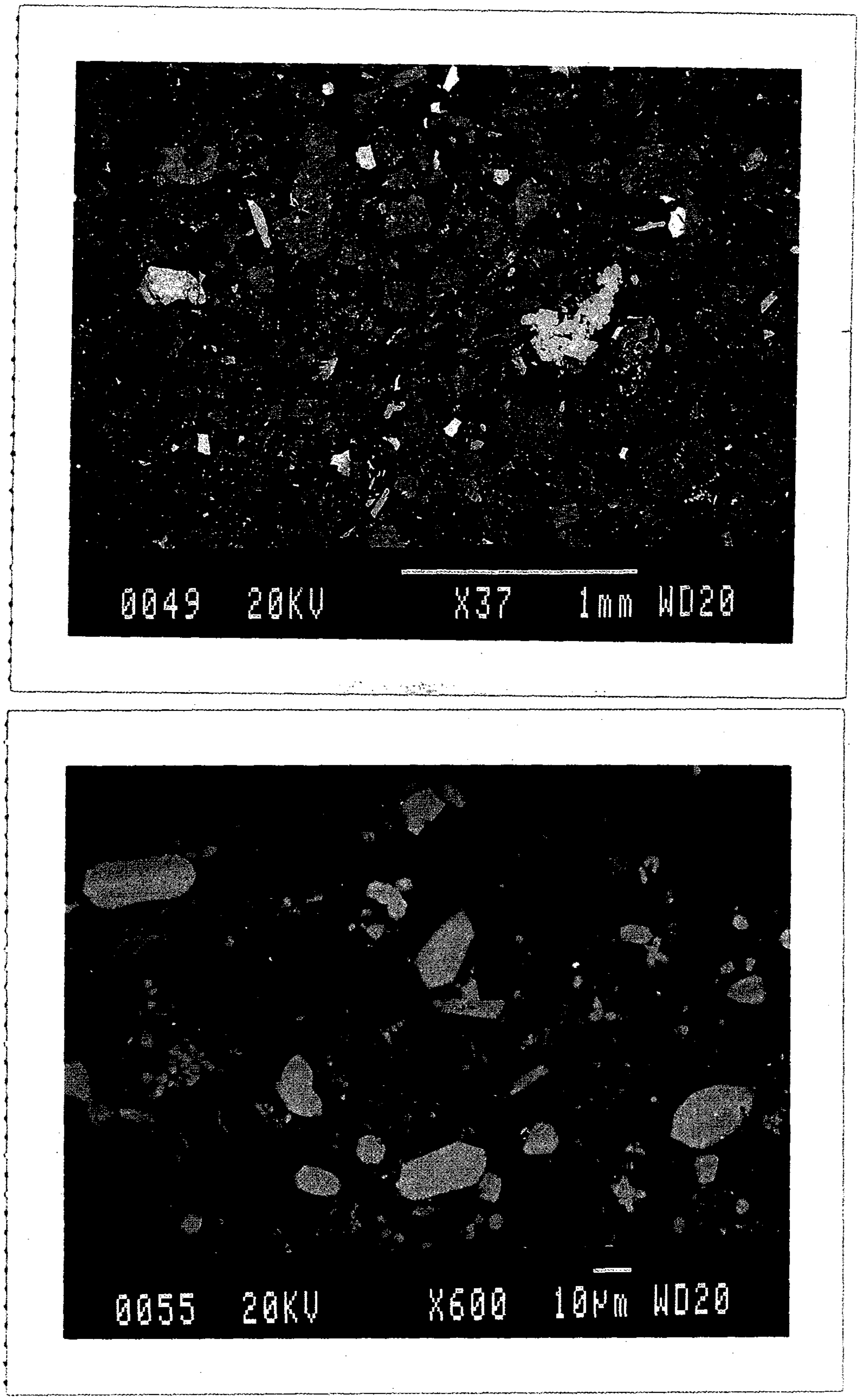
Plate 49 shows the general morphology of the aged S-6 sludge. The larger bright particles are mainly gypsum and a Zn-Mn-Na-Mg-S-O phase containing minor amounts of Ca, Si and Fe. The cracked morphology implies that this latter phase is a hydrated compound. The tiny bright particles in the dark grey matrix are mainly calcite grains. The majority of the sludge consists of an amorphous (dark grey) phase of Mg, O, C and minor Na, Zn, S, Si, Ca and trace Mn and Fe in composition. The detailed mineralogy of this amorphous compound is shown in Plate 55, in which the coarse crystals are calcite, the tiny light particles are calcite and $(Ca,Mg,Zn)CO_3$ with trace gypsum, and the matrix is mainly Mg-C-O, minor Zn, S, Si, Ca, Na and trace Mn and Fe in composition. The composition of the amorphous phase is variable but contains the same elements.

Mineral morphology similar to this is shown for the fresh sludge in Plate 43. The aged sludge consists almost entirely of recrystallized calcite, gypsum and amorphous compounds, whereas, the fresh sludge contains a significant amount of detrital particles such as silicates and pyrite. Plate 58 shows that the aged sludge contains a Zn-Mn-Na-Mg-S-O phase, with minor Si, Ca and Fe content. This phase is absent in the fresh sludge sample. Presumably, this phase contributes to the difference in the leaching characteristic of Zn between the fresh and aged samples.

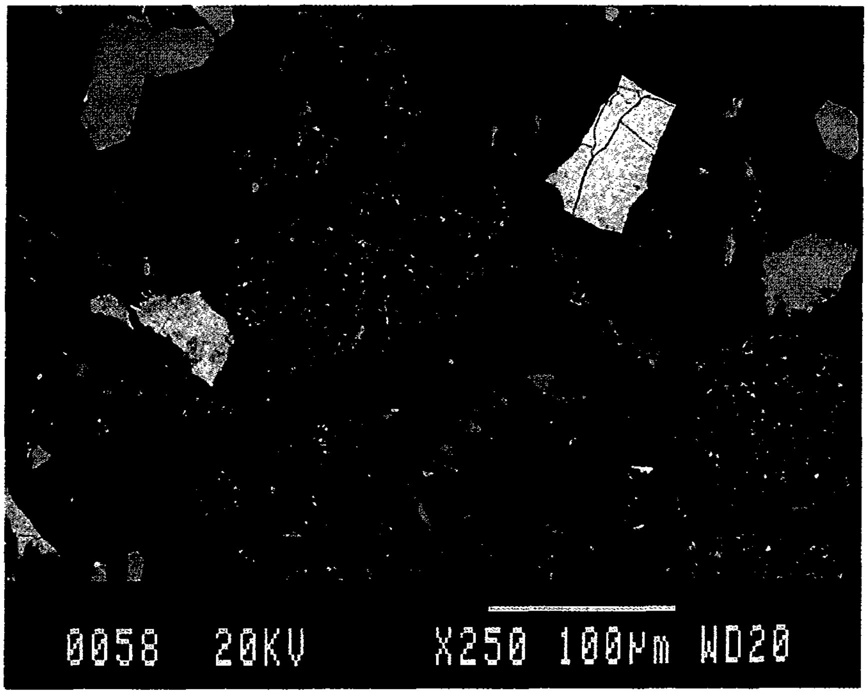
XRD pattern of the aged sludge indicates that the sample contains a quantity of amorphous compound, as observed by the SEM study. Calcite, gypsum and trace quartz are the other compounds detected by XRD. Presumably, most of the elements are carried in the amorphous phase.



Photomicrographs of sludge collected from S-6 (refer to text).



Photomicrographs of sludge collected from S-6 (refer to text).



Photomicrographs of sludge collected from S-6 (refer to text).

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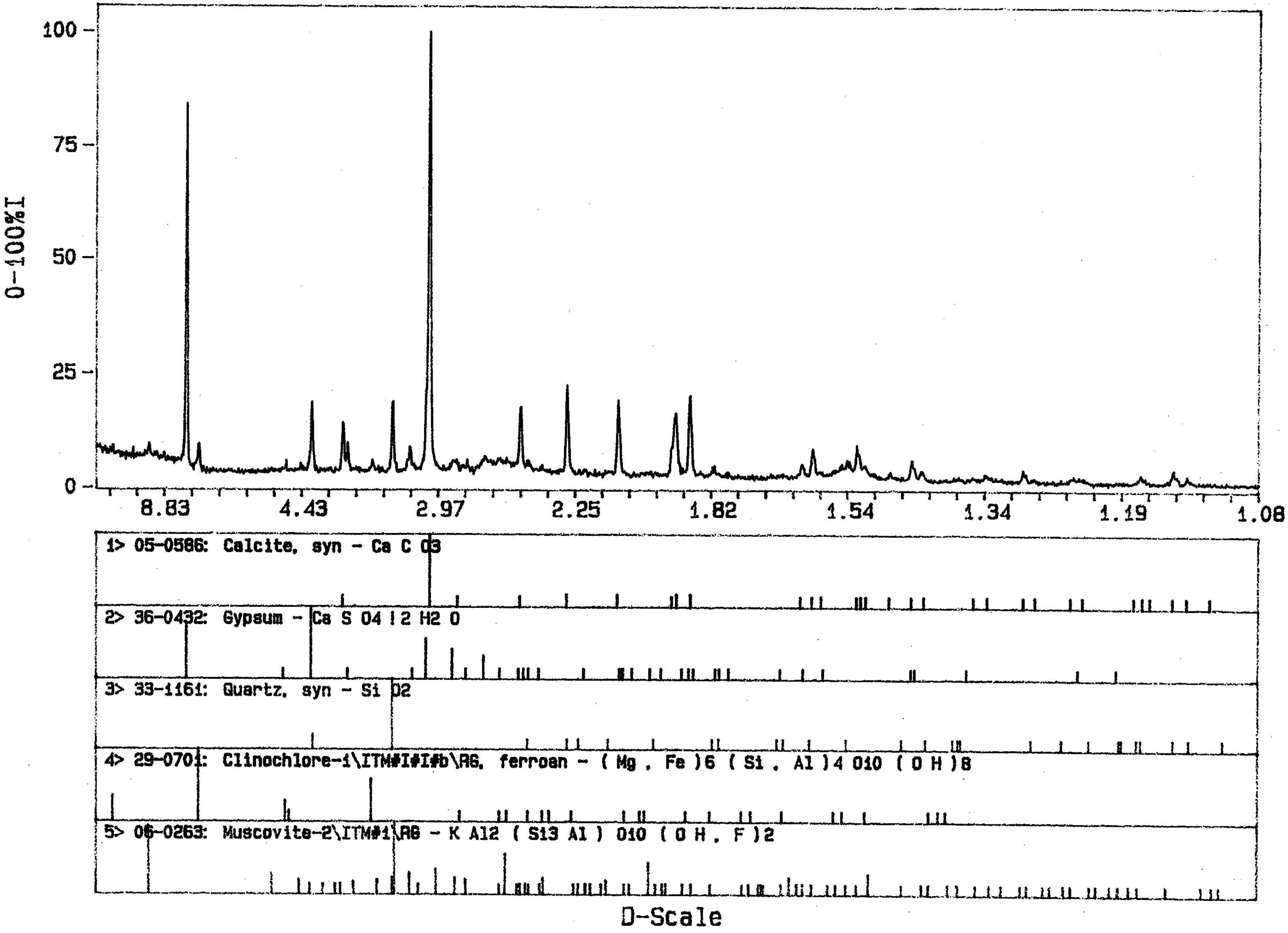

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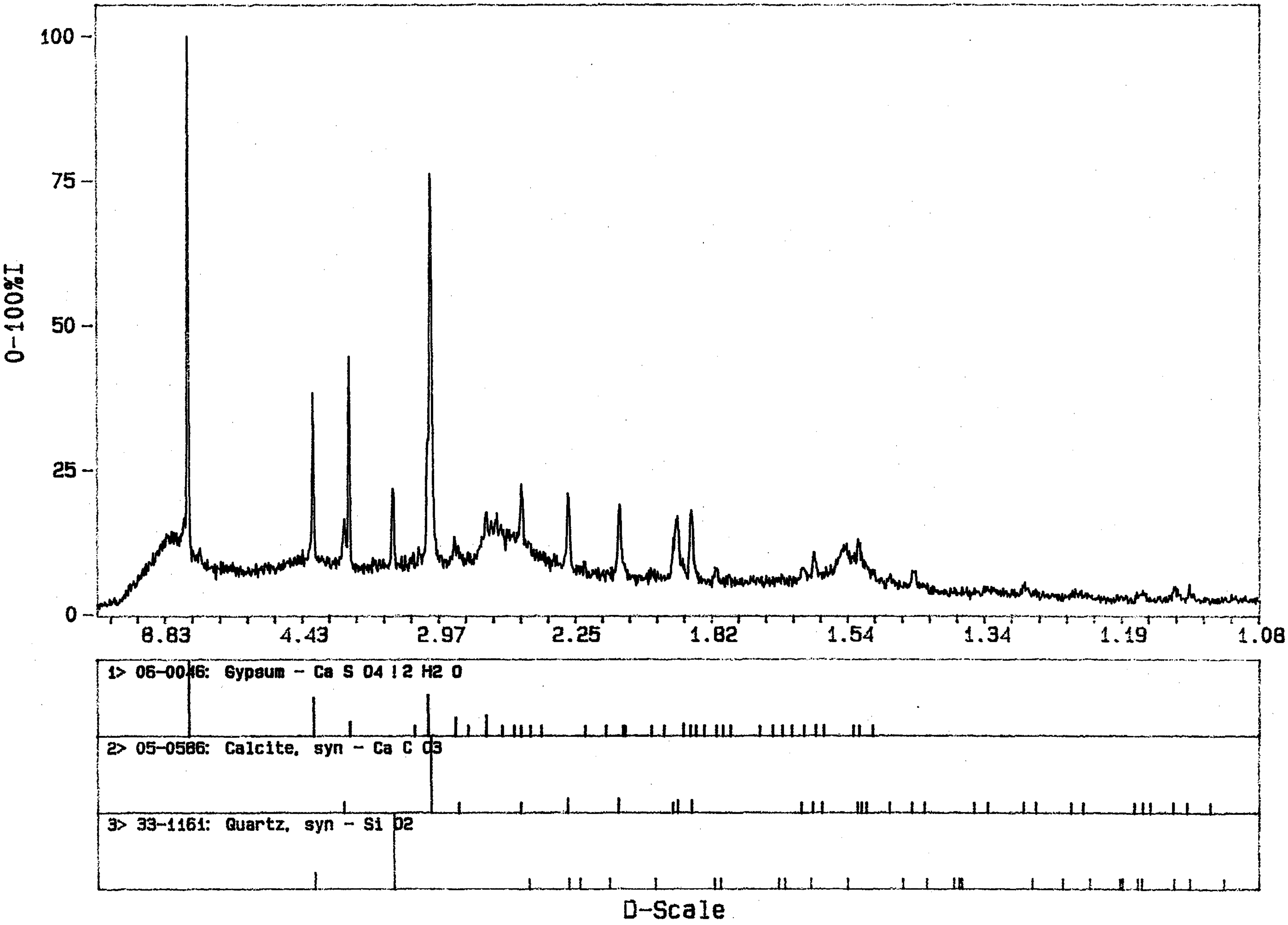
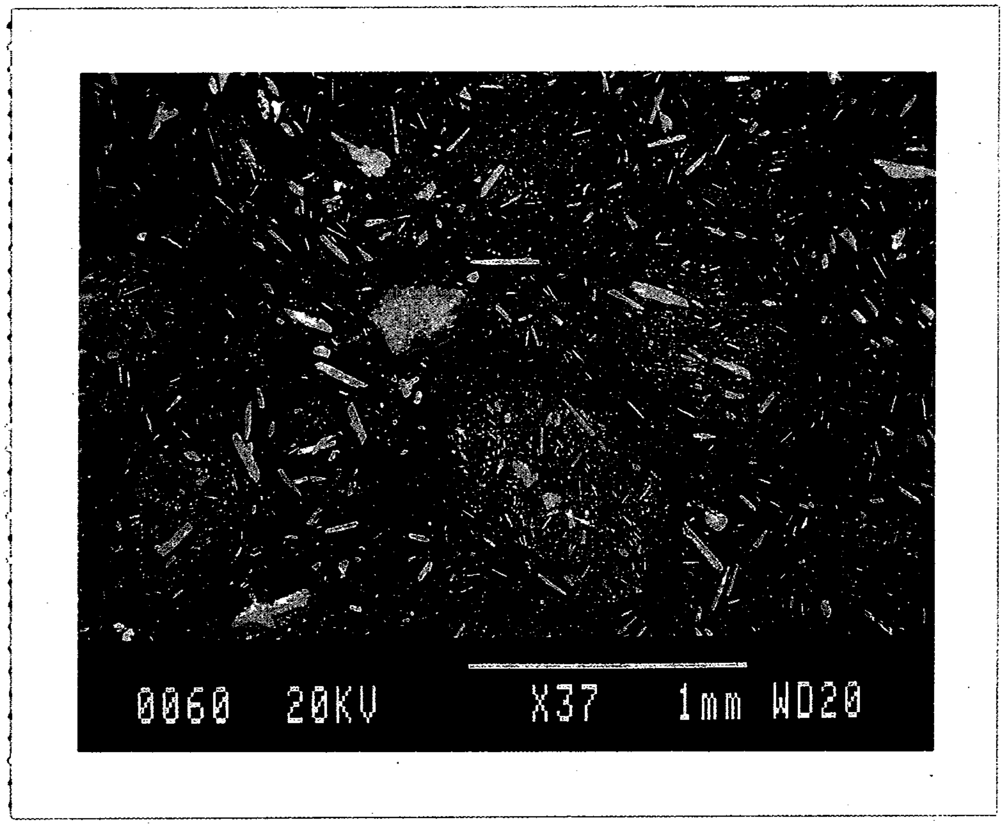
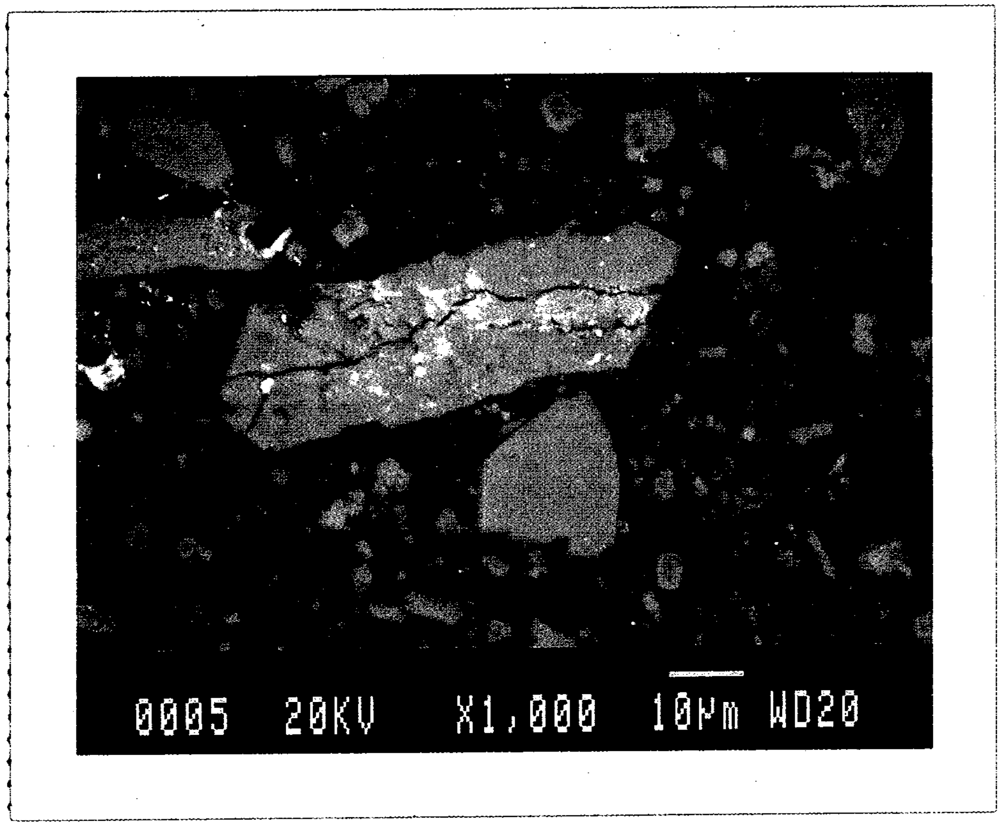


Site J-7

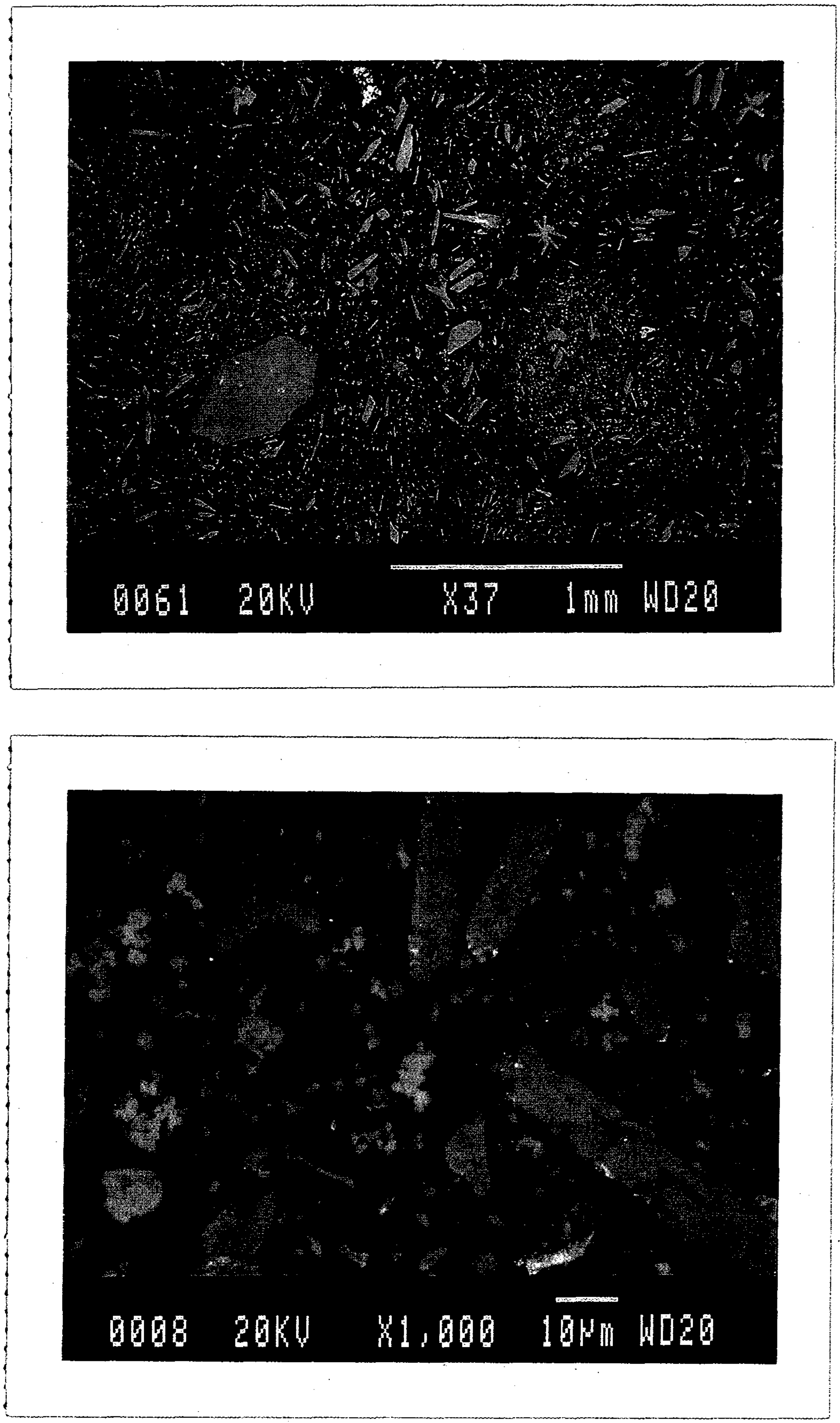
Plate 60 illustrates the general morphology of the J-7 fresh sludge.. The sludge consists of needle-like crystals of gypsum and major fine-grained compounds. The detailed morphologies of the fine-grained phase is shown in Plate 5. The large crystals are gypsum, the fine-grained matrix consists of major Fe, Ca, Al, minor Mg, SO_4, Si and trace Mn, Zn and C in composition. XRD analysis indicates that the sludge consists predominantly of gypsum with only traces of amorphous compounds. This implies that gypsum is the major constituent of the fine-grained material; other elements such as Fe, Al, SO_4, Si, Mn, and Zn are likely to be present as amorphous compounds. Trace Fe-oxide, silicates, quartz particles are also present in the fine-grained matrix. Chemical analysis indicate high Ca and SO_4 contents for the sludge sample; the high Ca and SO_4 contents reflect the presence of dominant amounts of gypsum in the sludge.

Plate 61 shows the general morphology of the aged J-7 sludge. The sludge particles show different degrees of dehydration. Except for the presence of a large quartz grain (detrital origin), the morphology and mineral species are similar to those of the fresh sludge sample (Plate 60). All the light, lath-like crystals are gypsum, most of the tiny particles are gypsum, and the fine-grained matrix consists of major Fe, Ca, S, O, Al and minor Mg, Si, P, and trace Mn and Zn in composition. The detailed morphology of the fine-grained matrix is shown in Plate 8; the large lath-like crystals and tiny needles are gypsum, the brighter small grain aggregates are particles of major Fe, minor Ca, S, Al, O, Mg, Si, P, and trace Mn in composition. Most of the elements, other than Ca and $SO_{4,}$ detected in the sludges are present in these fine-grained particles. Trace Fe oxide, quartz and silicate particles are also present in the fine-grained masses. Comparison of Plate 5 with Plate 8 indicates that the fresh sludge may contain slightly more amorphous phase than the aged sludge. Most of the fine-grained amorphous phase will gradually recrystallize into gypsum crystals, however the particle sizes appear to be similar.





Photomicrographs of sludge collected from J-7 (refer to text).



Photomicrographs of sludge collected from J-7 (refer to text).

ID: CHEN J-7 30-SEP-96@08: 59

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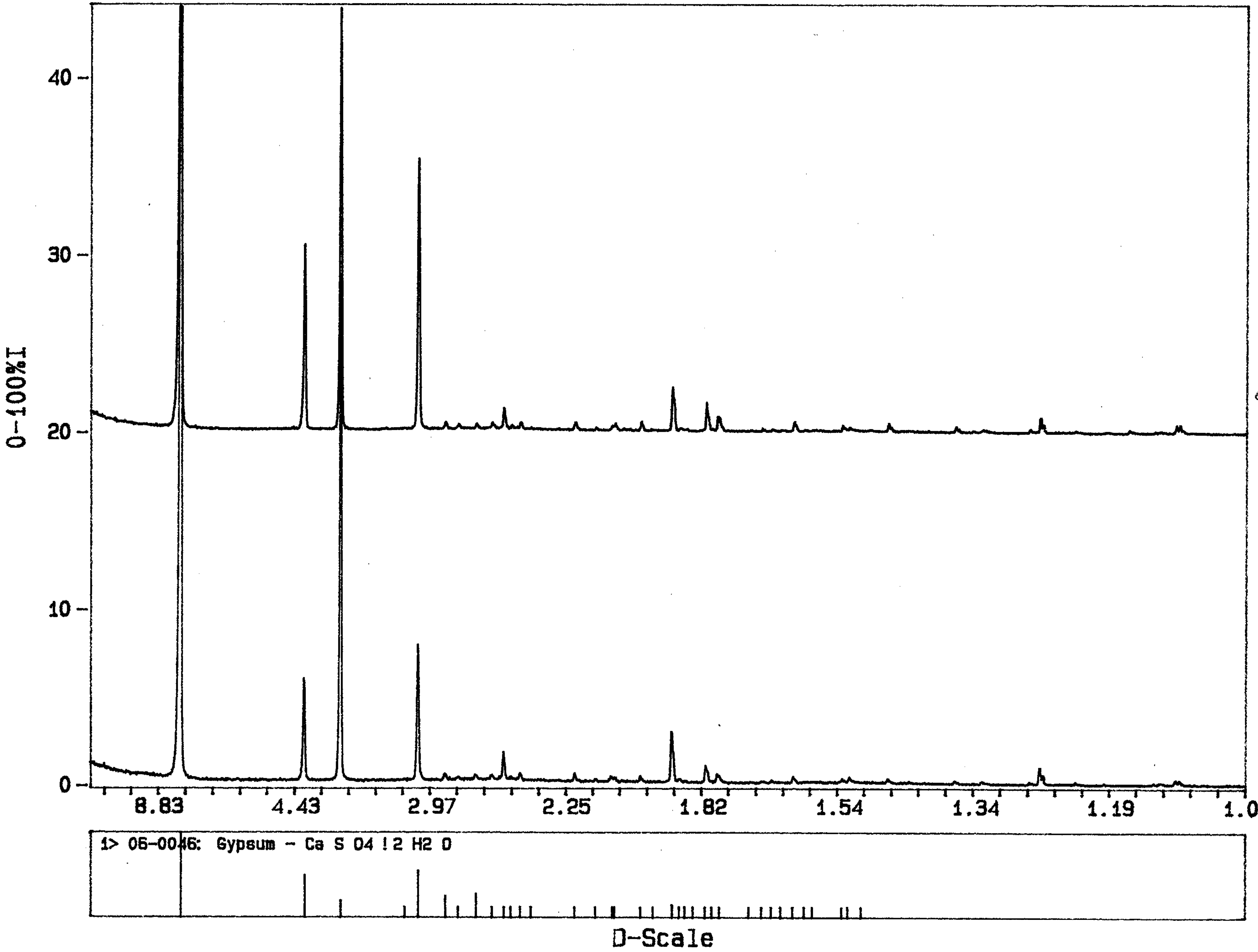


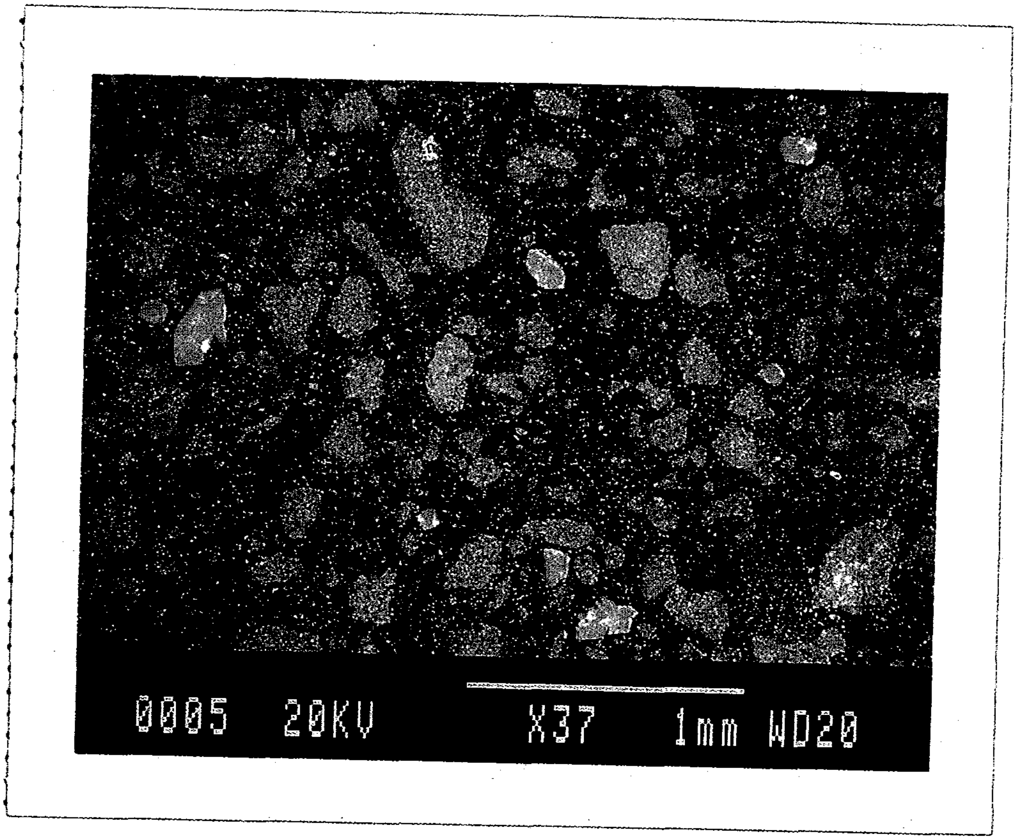
Site: W-8

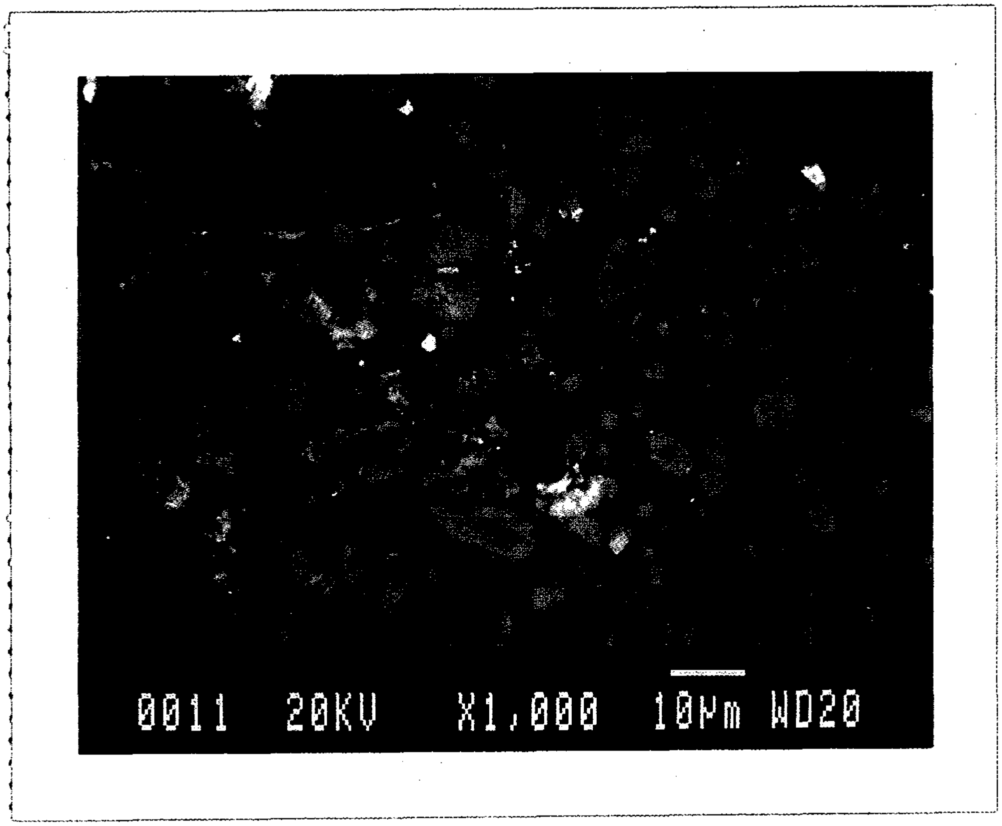
Plate 5 (37 x magnification) shows the general morphology of the fresh sludge produced from the W-8 HDS process. The material is mainly amorphous; only trace gypsum, bassanite ($CaSO_4 \cdot 5H_2O$) and an ettringite-type compound (likely $Ca,Mg)_6(Al,Fe,Mn)_2(SO_4)_3(OH)_{12} \cdot nH_2O$) are detected by XRD analysis. In general, the sample consists of minor amounts of large gypsum crystals and bassanite, and a dominant amount of fine-grained compounds. A trace quartz particle is also present. Apart from the large gypsum crystals, the material is relatively homogeneous. Plate 11 shows the morphology of the fine-grained material. The material consists of larger gypsum crystals and a major small (1 - 10 μm) particles of mainly Fe-Zn-Mn-Ca-S-O with minor Na, Mg, Al and trace Si in composition. Presumably, these particles are mainly amorphous with very low degree of crystallinity, and represent the ettringite-type compound $(Ca, Mg)_6(Al,Fe,Mn)_2(SO_4)_3(OH)_{12} \cdot nH_2O)$ detected by the XRD analysis. The fine-grained (< 1 μm) darker particles are Fe-Zn-Ca-S-Na-Mg-Al-Si-O in composition; these particles contain only trace Mn. Since the Fe-Zn-Mn-Ca-S-minor Al, Mg, Na (ettringite-type) particles are the main component of the sludge, the stability or leachability of the sludge depends on the stability of these compounds.

Plate 1 shows the general morphology of the W-8 aged sludge. XRD analysis indicates the sludge is mainly amorphous, except for small amounts of gypsum/bassanite, calcite and ettringite compounds. The bright particles and the majority of the particles (grey) in Plate 1 are compounds containing Fe-Ca-S-O-Zn-Al-Mn-Na; the lighter particles contain more Fe, Mn and Zn than the darker particles. The tiny bright particles are mostly pyrite, sphalerite, calcite and gypsum (euhedral crystals). The dark grains are a Ca-S-C-O-Si compound. Trace Fe-oxide, quartz and silicate particles, all detrital in origin are also present in the sludge.

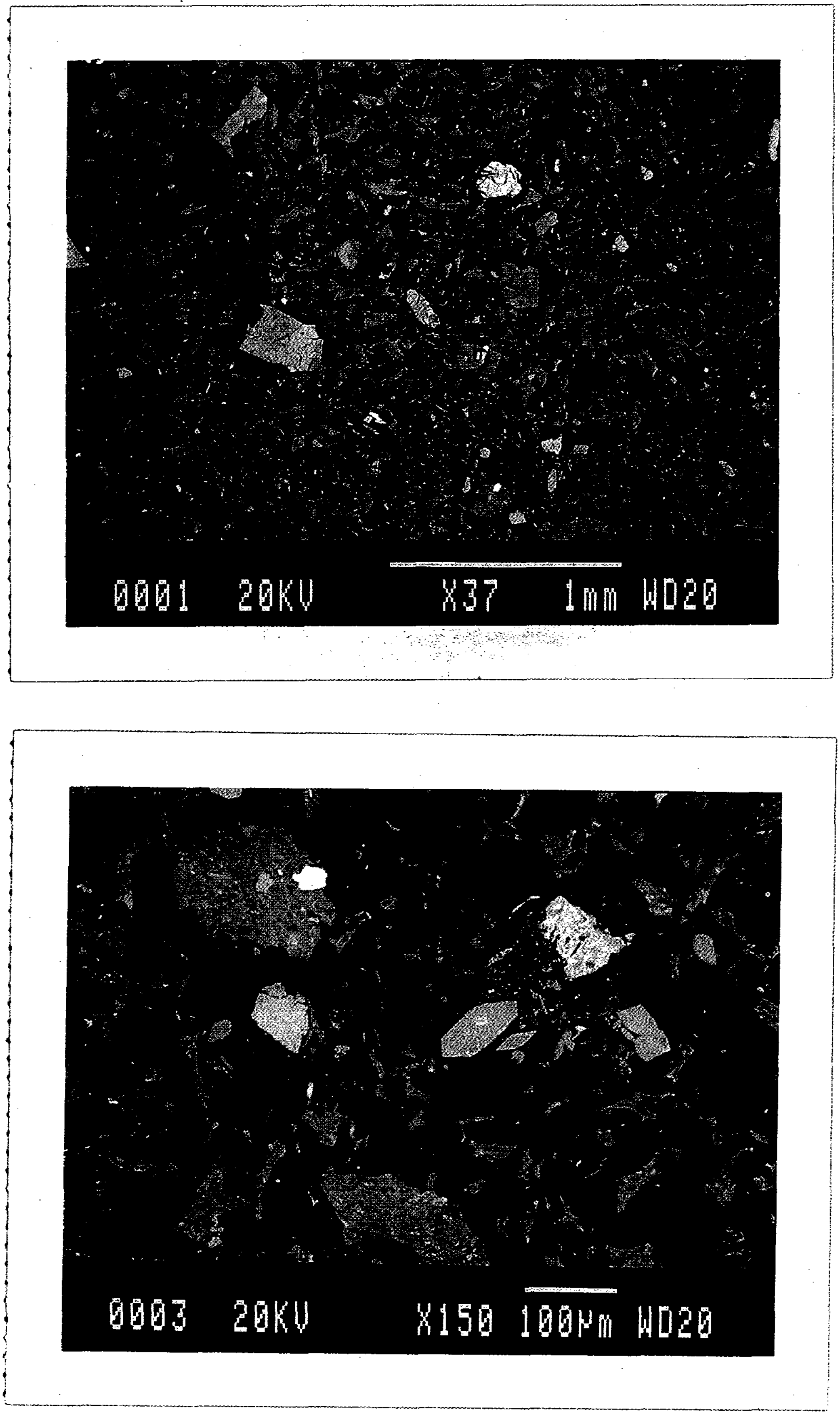
Plate 3 shows the details of the sludge. Gypsum (light) occurs as euhedral crystals; calcite occurs as angular grains; and a small amount of carbon hydrate phase (dark) containing trace S, Ca, O, Si is present. Trace pyrite (bright), sphalerite (bright) and quartz are also present. The major compound in Plate 3 is a fine-grained mixture of Fe-Ca-S-O-Zn-Al-Mg-Si-Mn-Na in composition. Plate 12 shows the details of these mixtures. The mass consists of tiny needle-like crystals of gypsum (left), small irregular particles (light, likely ettringite-type compounds) of Fe-Ca-S-Mg-O-minor Zn, Mn, Al, Si, Na in composition and a fine-grained matrix of Ca-Mg-S-O minor Fe, Zn, Si, Al, Na, C, Mn in composition. Presumably tiny gypsum crystals and poorly crystalline ettringite-type compounds are the main components of this fine-grained matrix. Variable amounts of calcite are also present in this fine-grained mass.

While a direct comparison cannot be made between the fresh and aged sludge samples as they were produced from different treatment process some general observations can be made. The aged sludge contains more detrital material, such as pyrite, chalcopyrite, sphalerite and quartz, than the fresh sludge. In general both the aged and fresh sludge contain only small amounts of recrystallized gypsum, calcite and ettringite-type compounds.

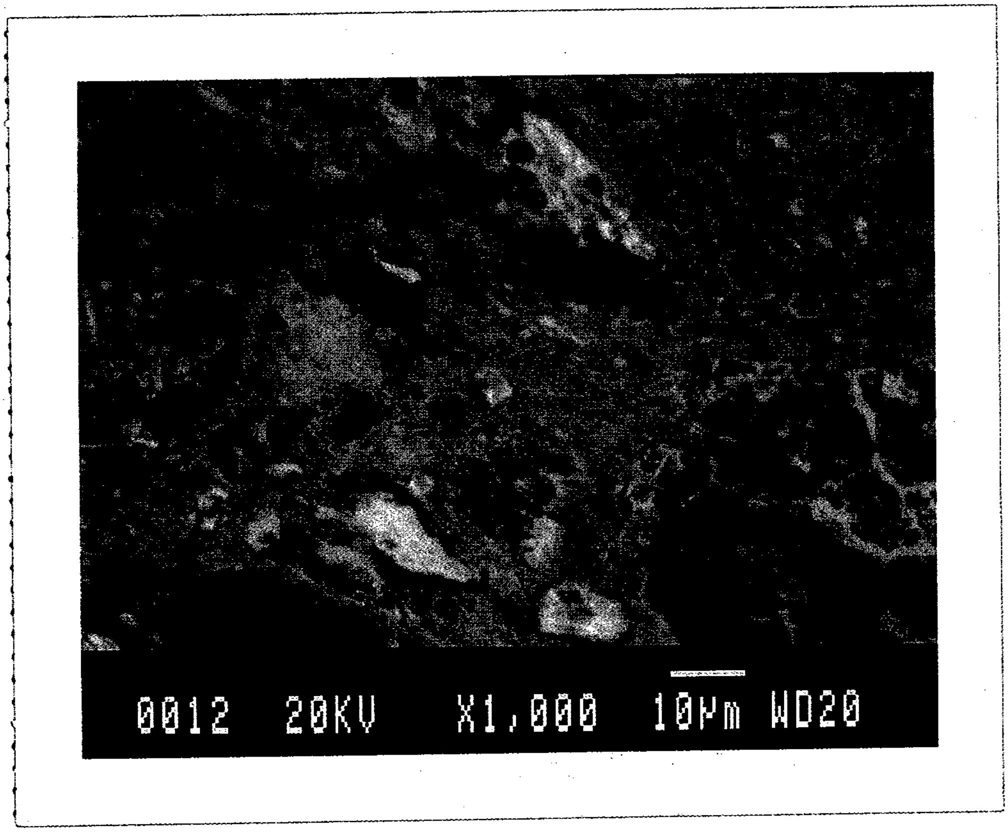




Photomicrographs of sludge collected from W-8 (refer to text).



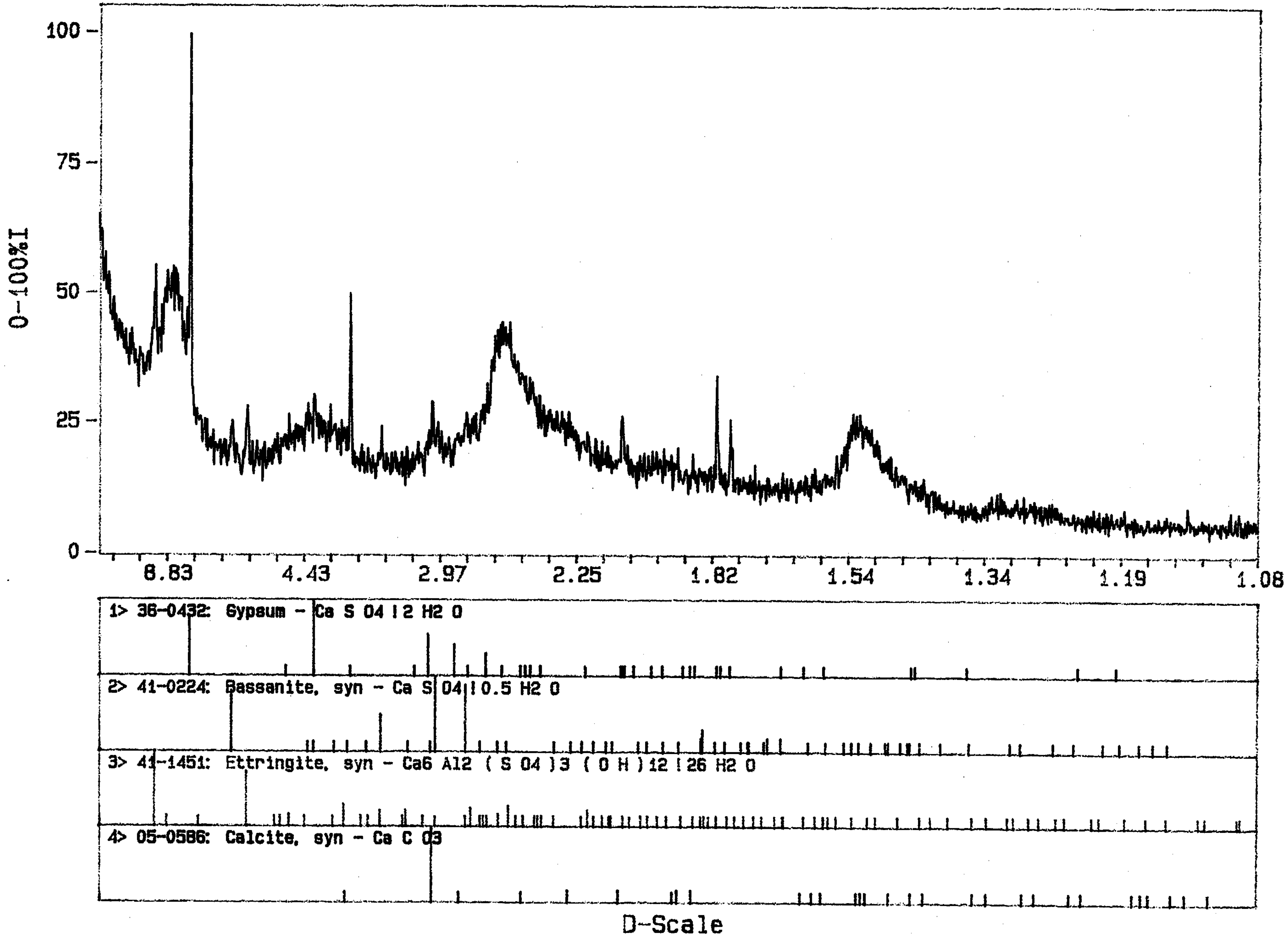
Photomicrographs of sludge collected from W-8 (refer to text).



Photomicrograph of sludge collected from W-8 (refer to text).

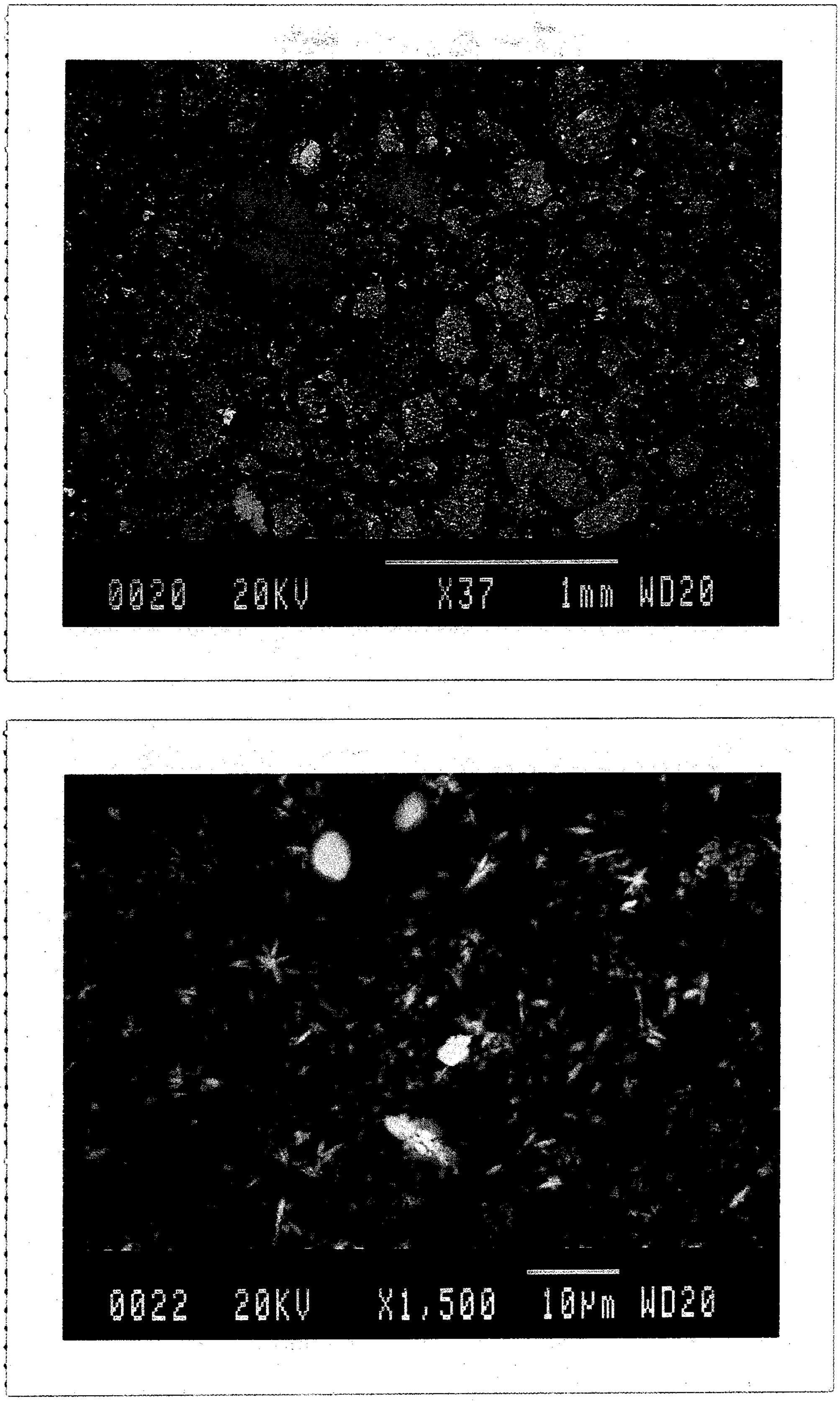
ID: CHEN W-8-F 30-SEP-96@10:24

File: Z02749.RAW Scan: 5-90/.05/ 1/#1701. Anode: CU



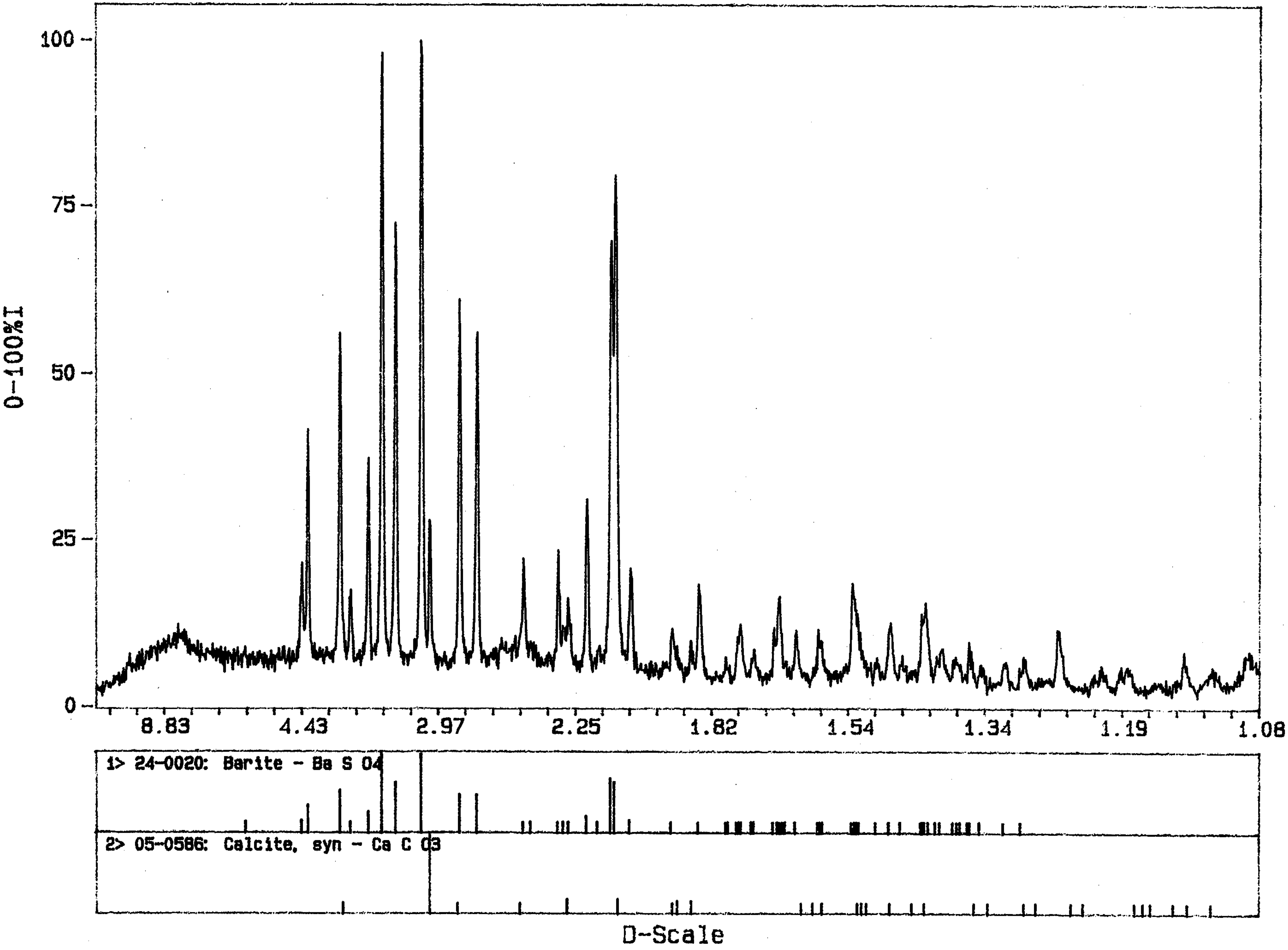
Site N-9

Plate 20 shows the general morphology of the sludge from the N-9 site. The "larger" light-grey particles are calcite; the numerous tiny bright particles are barite ($BaSO_4$), and the grey main mass is an amorphous compound consisting of Mg, Al, Si, Fe, Ba, Ca, S, C, O and trace Mn, Zn, Na in composition. The darker mass contains less barite crystals. The cracked texture of the amorphous mass implies its hydrated nature. The details of the sludge are shown in Plate 22. The star-like and tiny bright particles are $BaSO_4$; the "star" morphology indicates its origin of recrystallization from solution. The two bright spherical particles at the top are Ce-La-Ca-Ba carbonates, the ore is likely rich in rare earth elements. The few sub-round grey particles are $CaCO_3$; gypsum occurs as porous elongate "rings" (grey). The matrix is mainly Fe-S-Si-O-C, minor Ca, S, Mg and Mn in composition. XRD analysis indicates that the barite and calcite are the main crystalline phases, the remainder of the sludge is mostly amorphous. This amorphous material apparently carries most of the elements (other than Ca, Ba and SO_4) found by the wet chemical analysis.



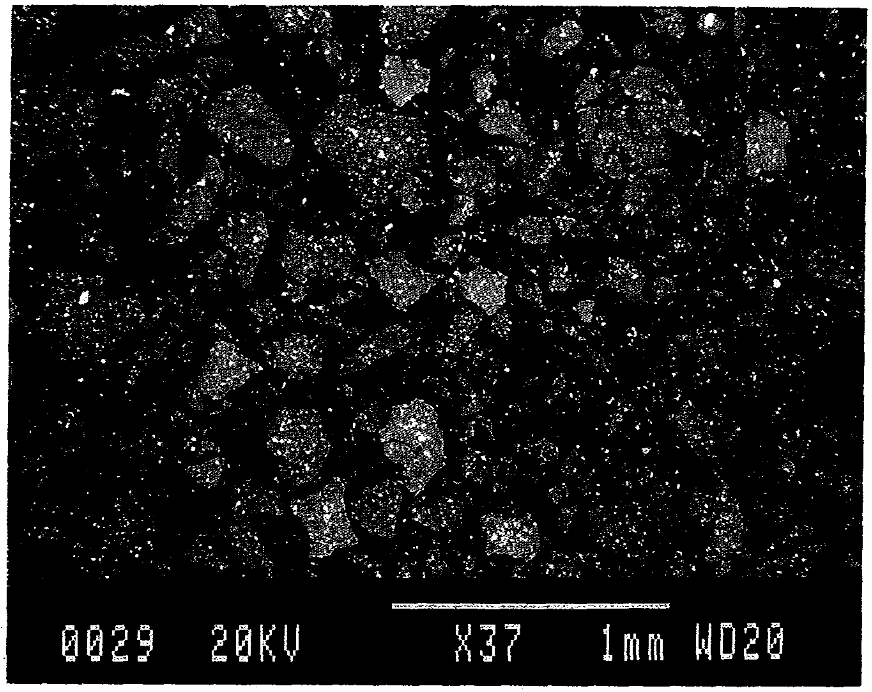
Photomicrographs of sludge collected from N-9 (refer to text).

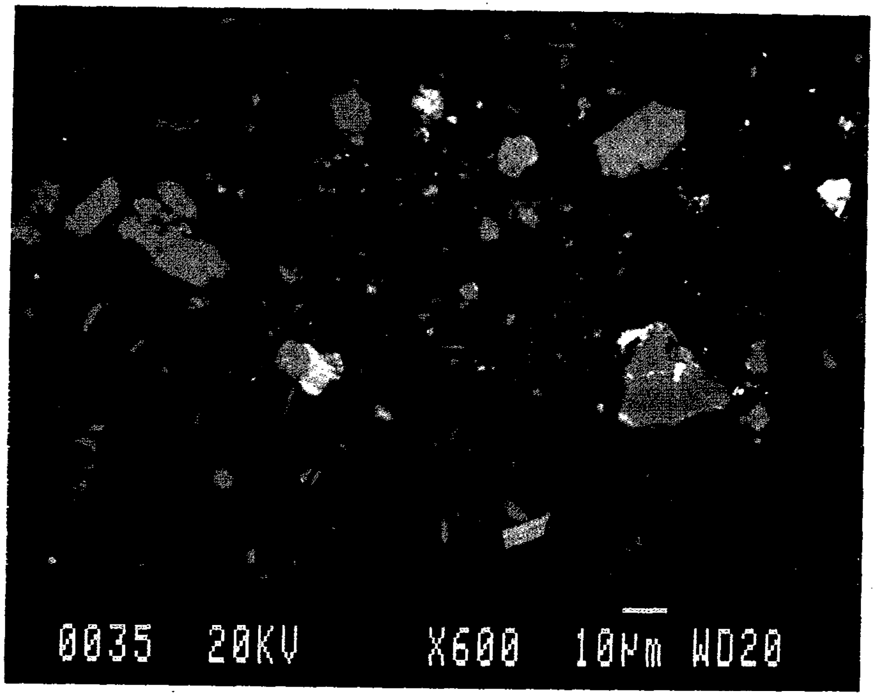
ID: CHEN N-9 30-SEP-96 08: 59
File: Z02743.RAW Scan: 5-90/.05/ 1/#1701, Anode: CU



Site B-10

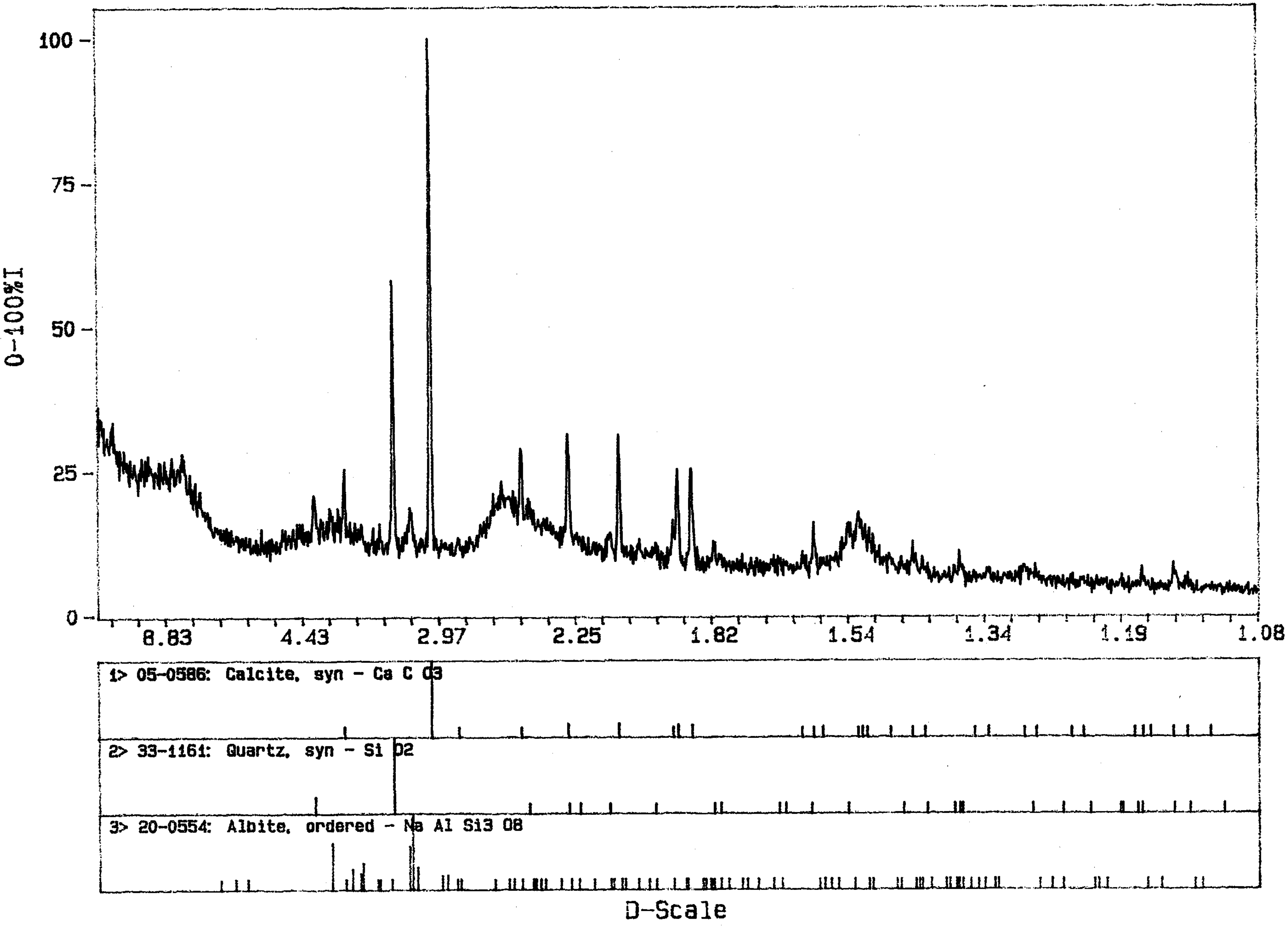
Plate 29 shows the general morphology of the fresh B-10 sludge. The bright particles are mainly quartz, Fe-oxide, calcite and various Fe-Ca-Mg-Al silicates. The main mass (grey) of the sludge is an amorphous precipitate of major Fe, Mg, Al, Si, O, C, minor S, Na, Ni, and trace Na, Cu in composition (see XRD data, Appendix G). All the silicate particles are detrital in origin. Other than the detrital particles, the sludge is fairly uniform. Plate 35 shows the details of this amorphous mixture. Quartz (grey), calcite (grey), Fe-Mg silicate, Fe-Ca-Mg-Al silicate (grey), Fe-oxide (bright), pyrite, commonly albite and Ca-Fe-T-Mg-Al silicate particles of various sizes are present in a fine-grained matrix (dark) of major Fe, Mg, Al, Si, O, C, minor S, Ca, Ni, and trace Na, Cu, in composition.





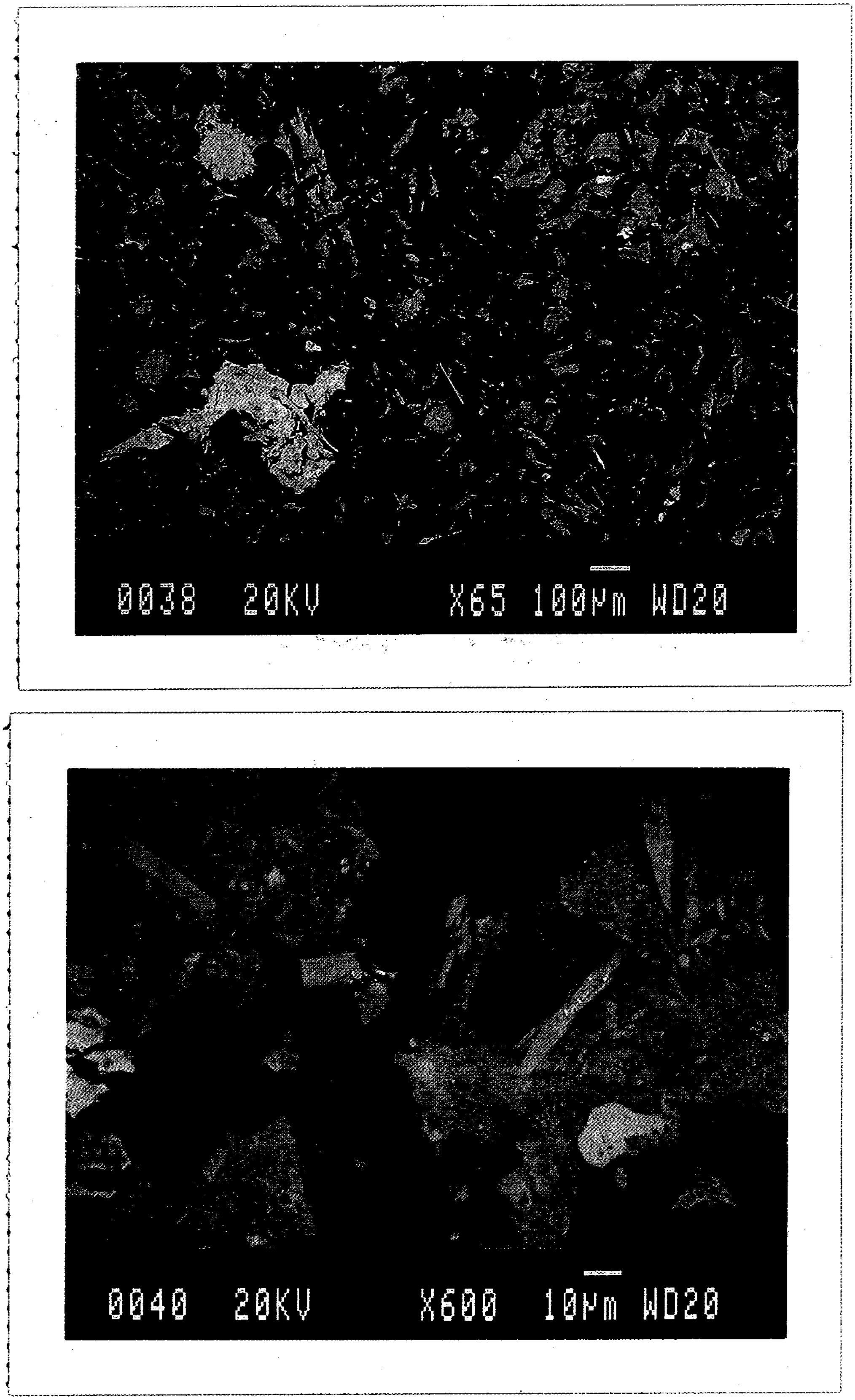
Photomicrographs of sludge collected from B-10 (refer to text).

ID: CHEN B-10 30-SEP-96@10: 24
File: Z02750.RAW Scan: 5-90/.05/ 1/#1701. Anode: CU



Site T-11

Plate 38 shows the general morphology of the sludge from B-11. Apart from small amounts of gypsum (radiating, needles) and calcite (small bright crystals), the sludges are mainly amorphous. Trace quartz and Fe-oxide particles (bright) are also present. Plate 40 shows the details of the amorphous compound. The needle-like crystals are gypsum, the brighter particles are Fe oxide and a few light particles are calcite. The fine-grained matrix is mainly Fe-Ca-Mg-S-O-C with minor Si, Al, Mn in composition. Likely, tiny gypsum crystals and a Fe-Ca-Mg sulphate phase are the main components of this amorphous mass.



Photomicrographs of sludge collected at T-11 (refer to text).

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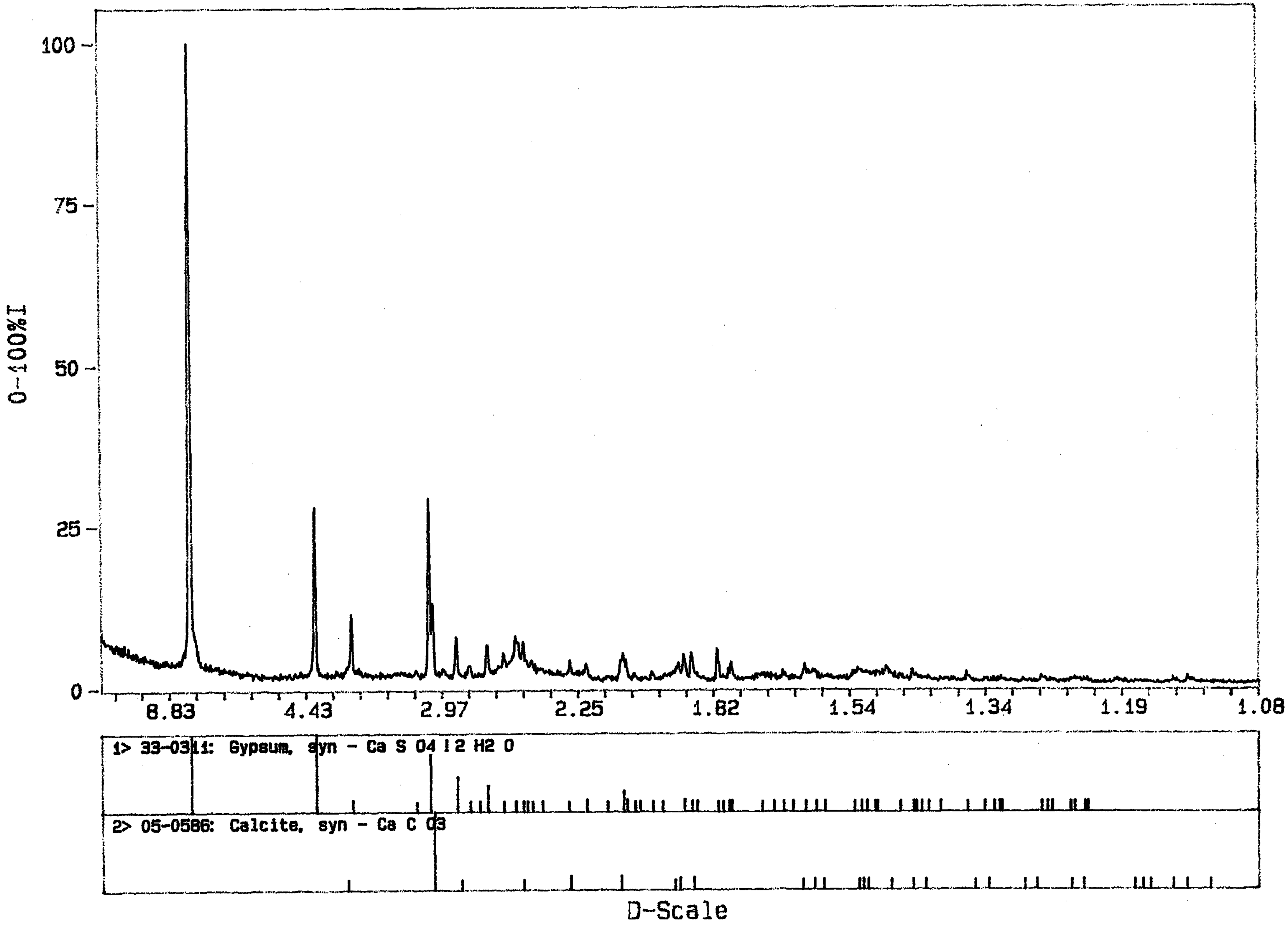
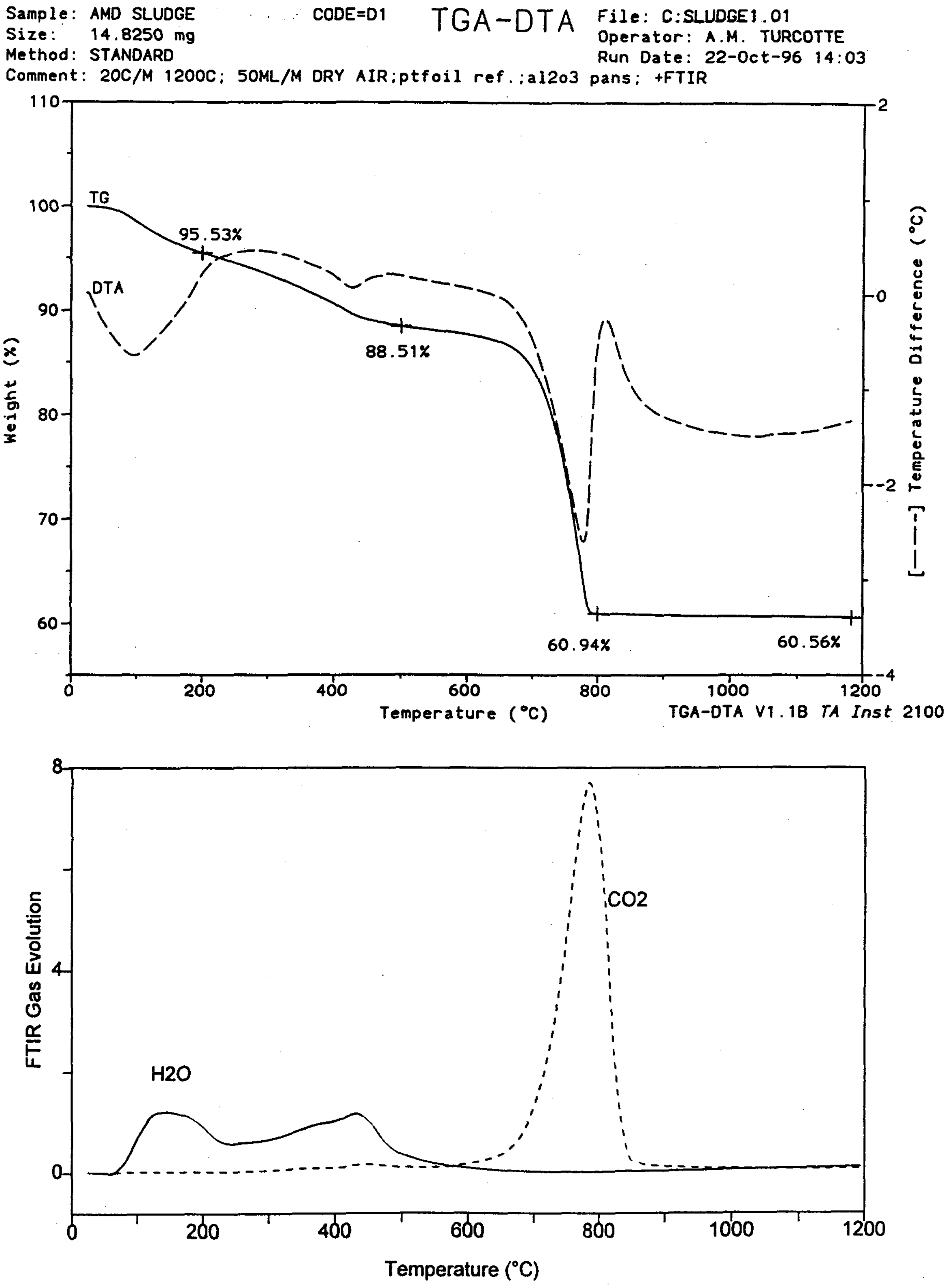
File: Z02751.RAW Scan: 5-90/.05/ 1/#1701, Anode: CU



APPENDIX H: THERMAL ANALYSES



Sample: AMD SLUDGE CODE=D1
Size: 14.8250 mg
Method: STANDARD
Comment: 20C/M 1200C; 50ML/M DRY AIR;ptfoil ref.;al2o3 pans; +FTIR
File: C:SLUDGE1.01
Operator: A.M. TURCOTTE
Run Date: 22-Oct-96 14:03

Figure H-1: TG/DTA/FTIR diagram for D-1 fresh sludge in air.

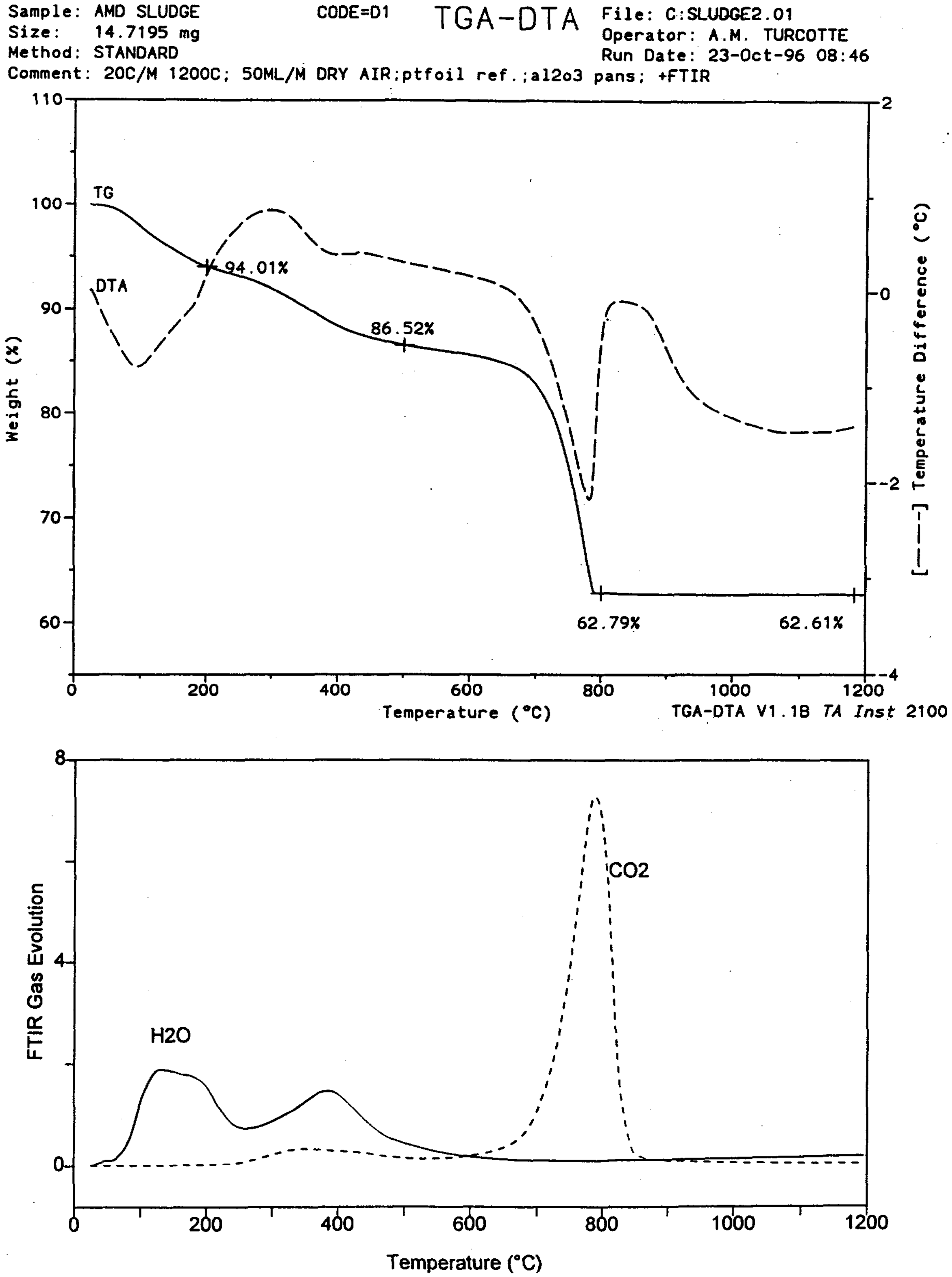


Figure H-2: TG/DTA/FTIR diagram for D-1 aged sludge in air.

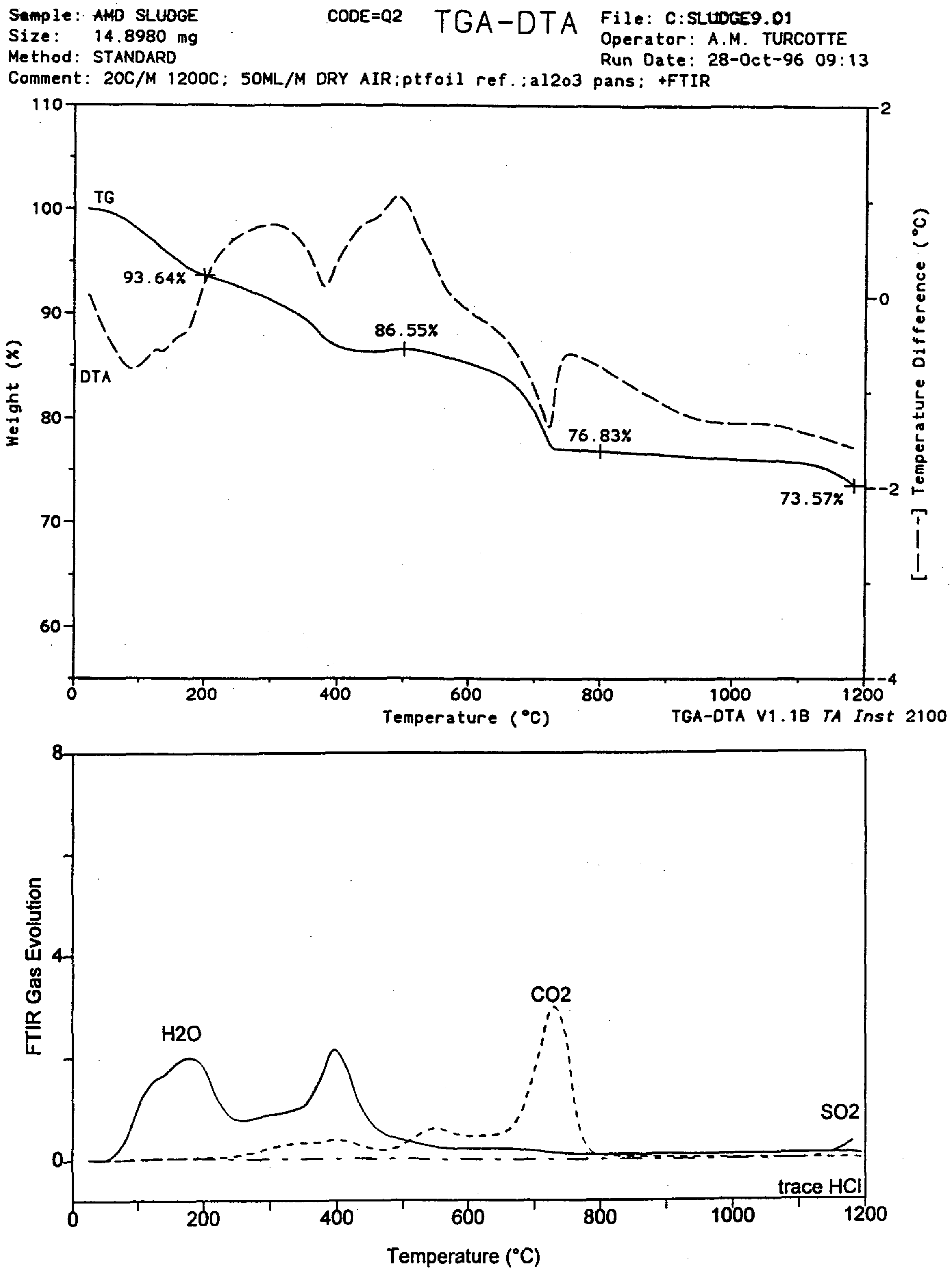
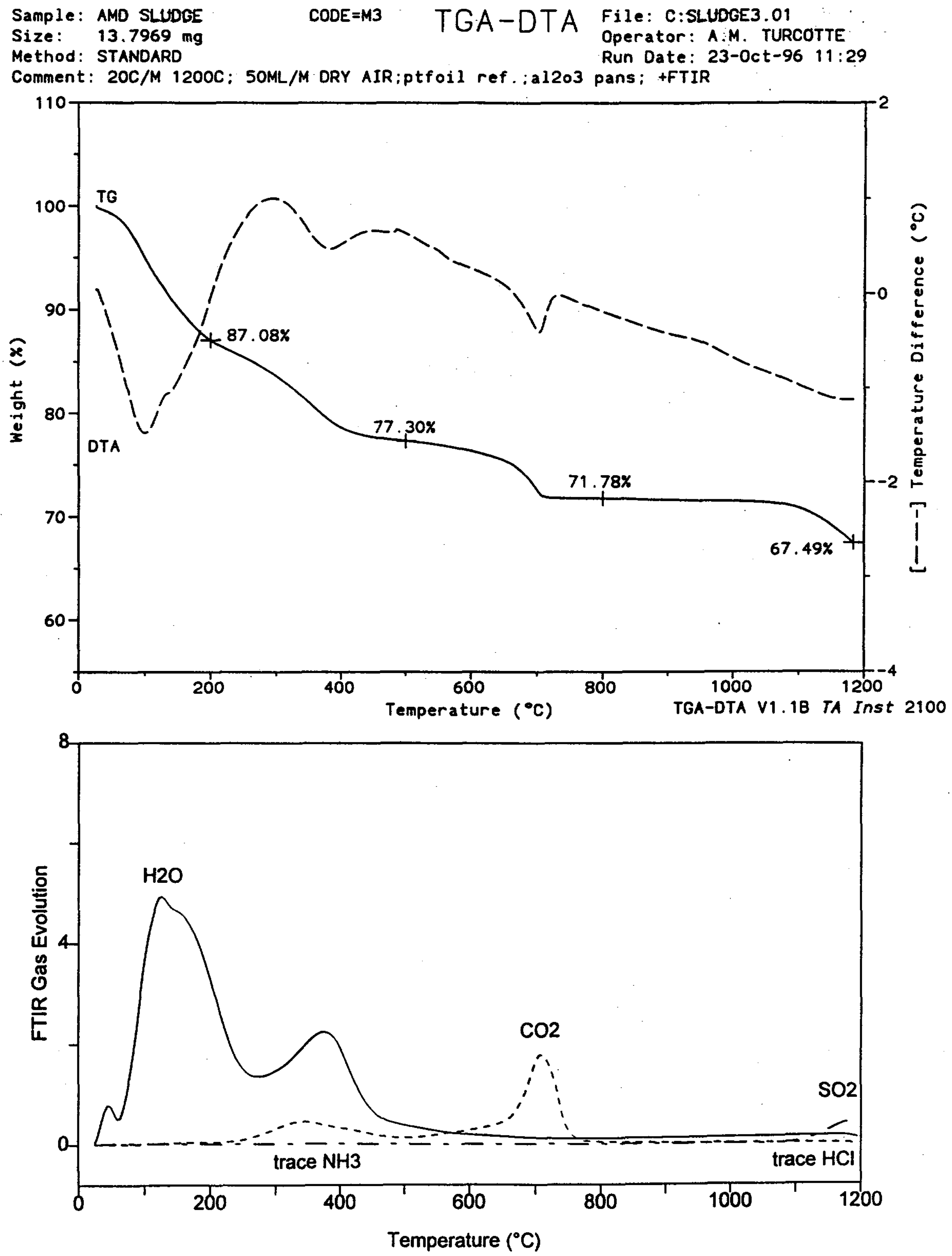


Figure H-3: TG/DTA/FTIR diagram for Q-2 aged sludge in air.



Sample: AMD SLUDGE CODE=M3 TGA-DTA File: C:SLUDGE3.01
Size: 13.7969 mg Operator: A.M. TURCOTTE
Method: STANDARD Run Date: 23-Oct-96 11:29
Comment: 20C/M 1200C; 50ML/M DRY AIR;ptfoil ref.;al2o3 pans; +FTIR

Figure H-4: TG/DTA/FTIR diagram for M-3 fresh sludge in air.

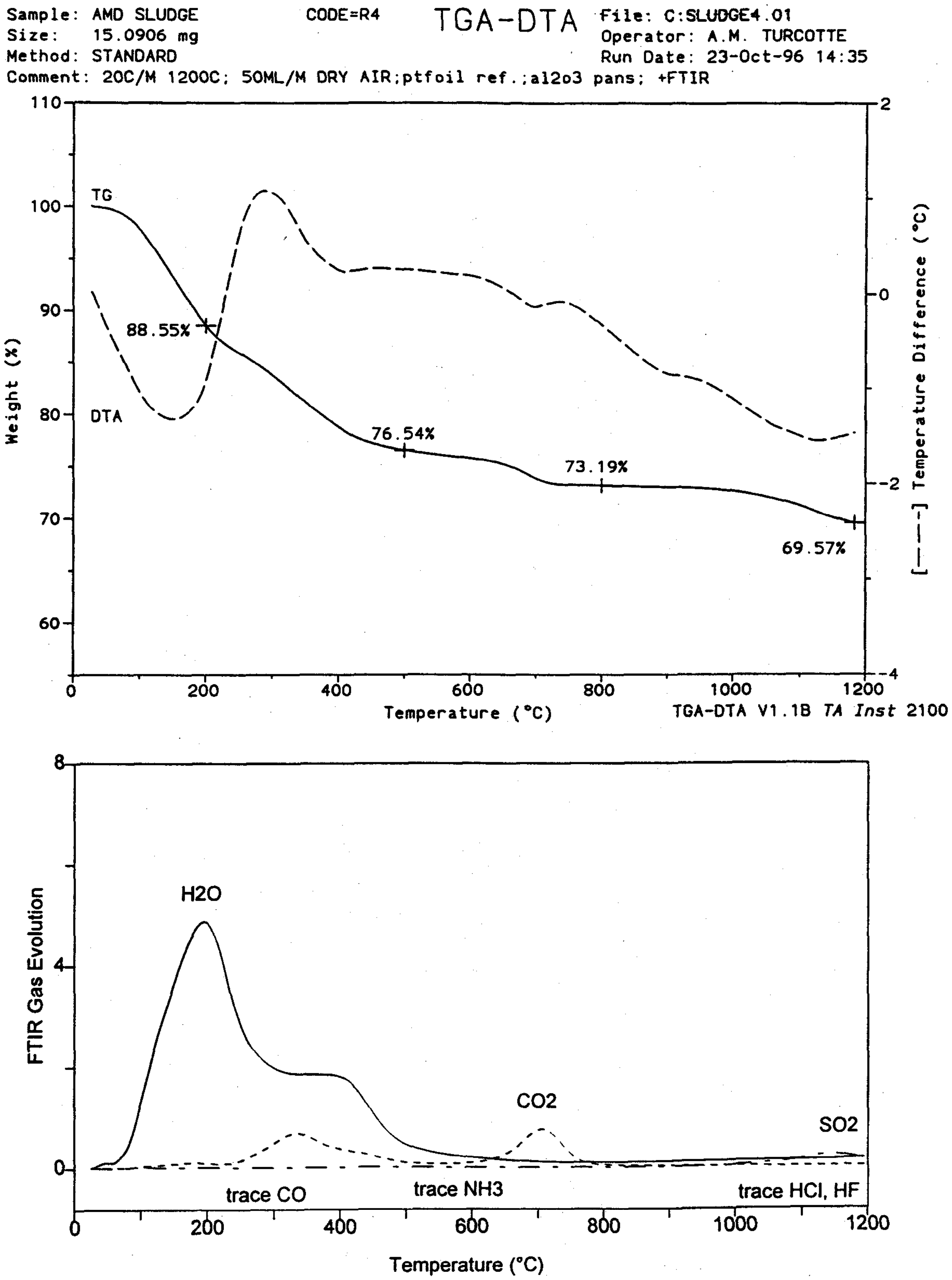


Figure H-5: TG/DTA/FTIR diagram for R-4 fresh sludge in air.

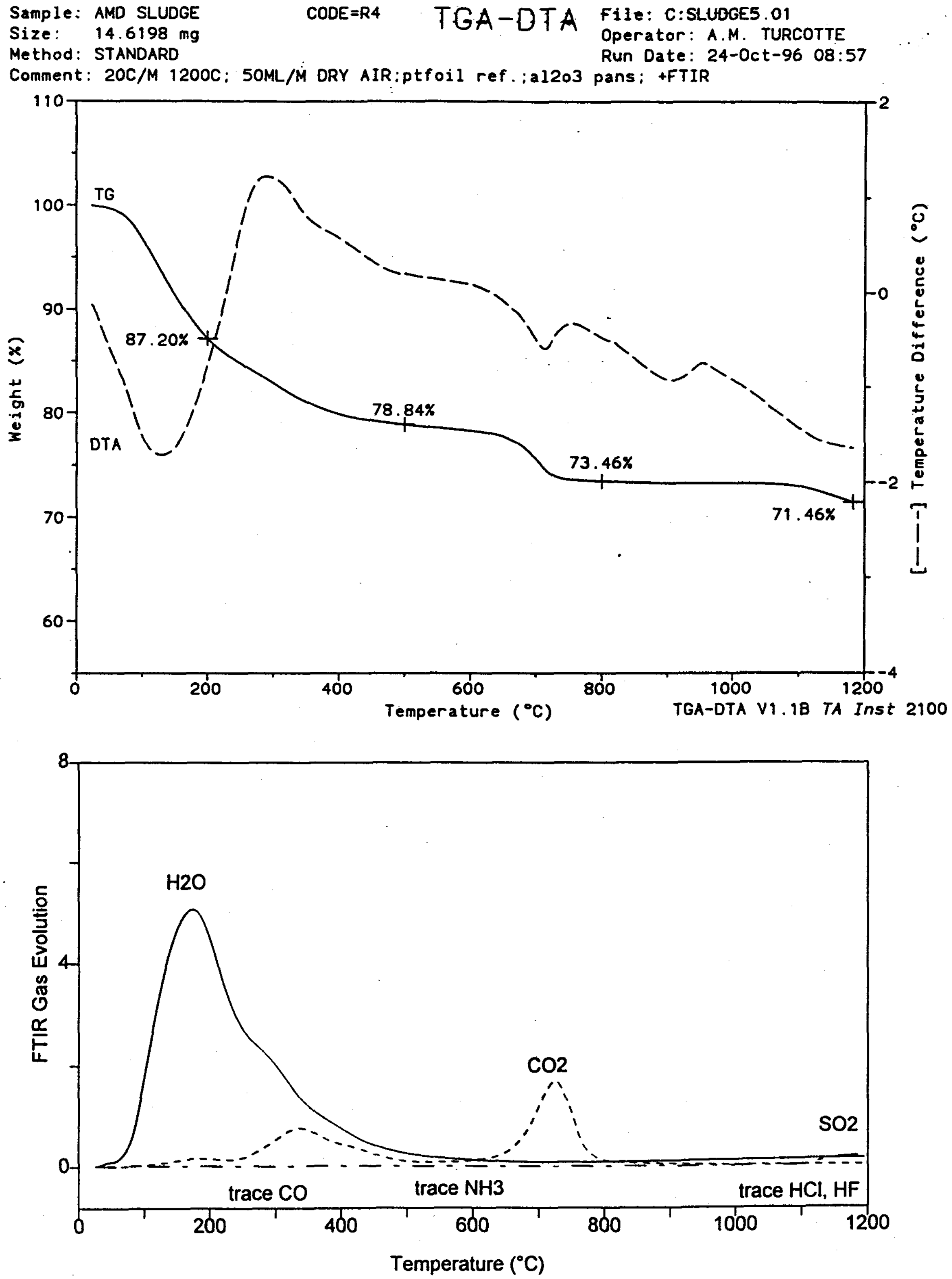


Figure H-6: TG/DTA/FTIR diagram for R-4 aged sludge in air.

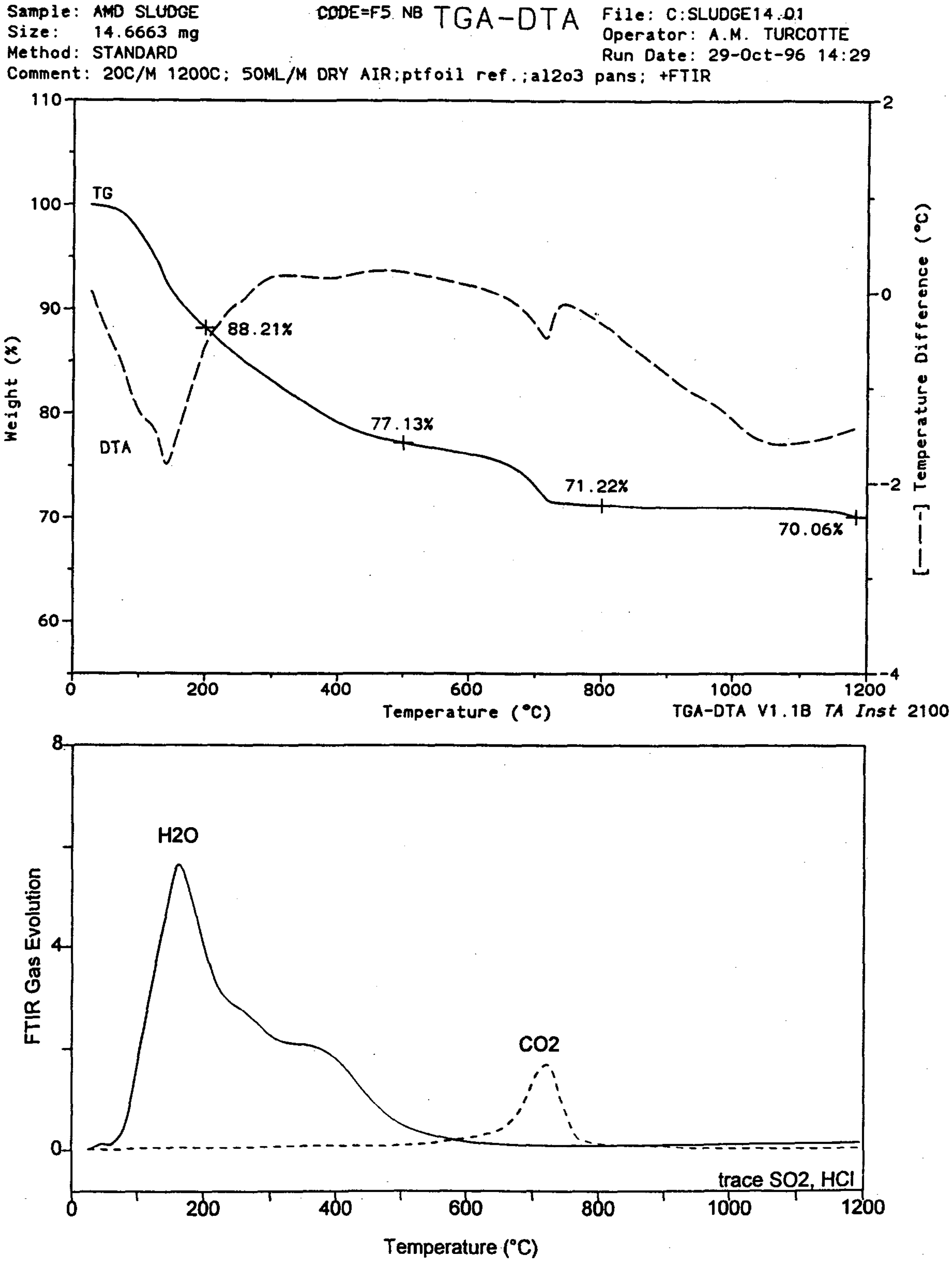


Figure H-7: TG/DTA/FTIR diagram for F-5 fresh sludge in air.

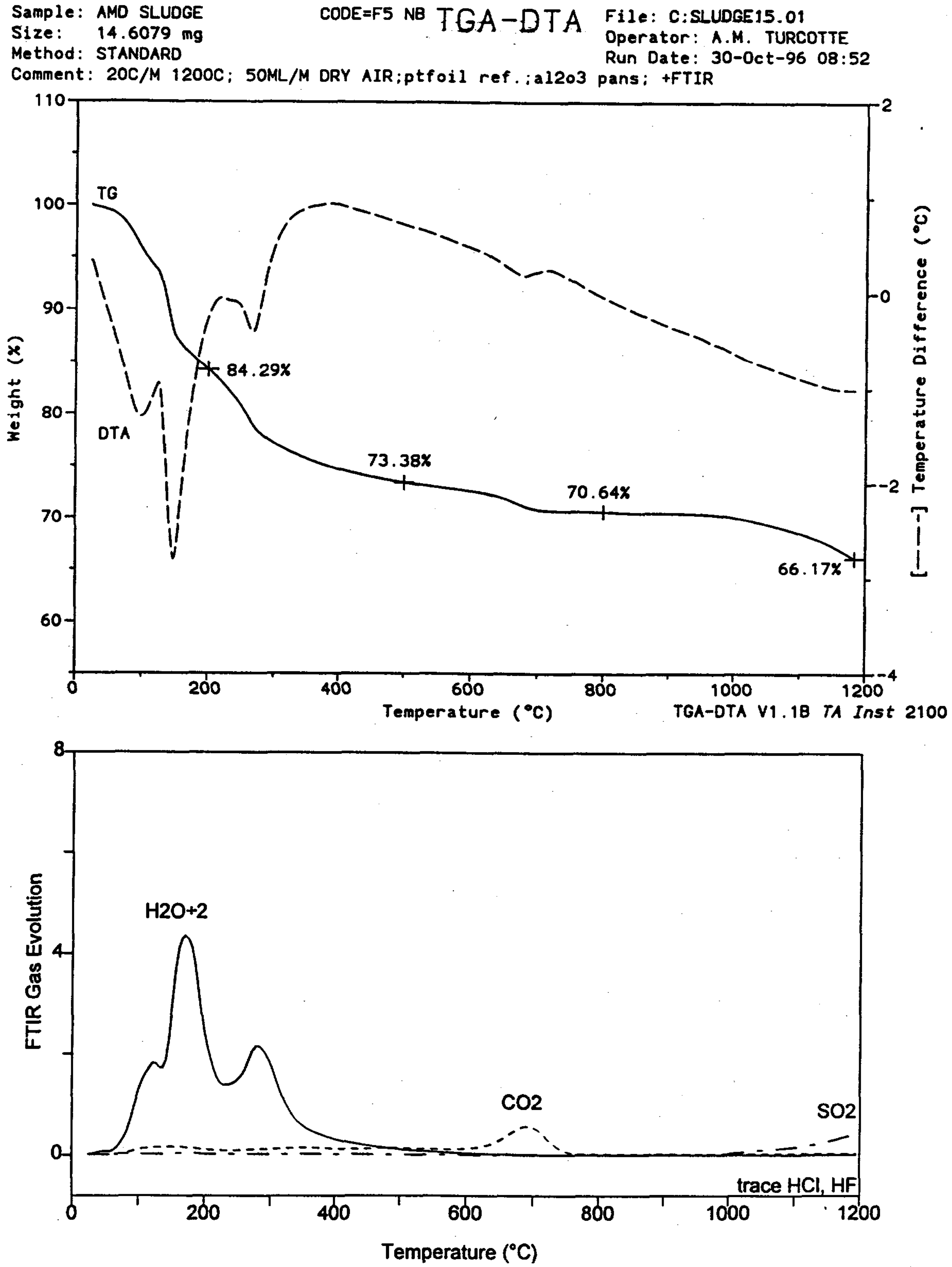
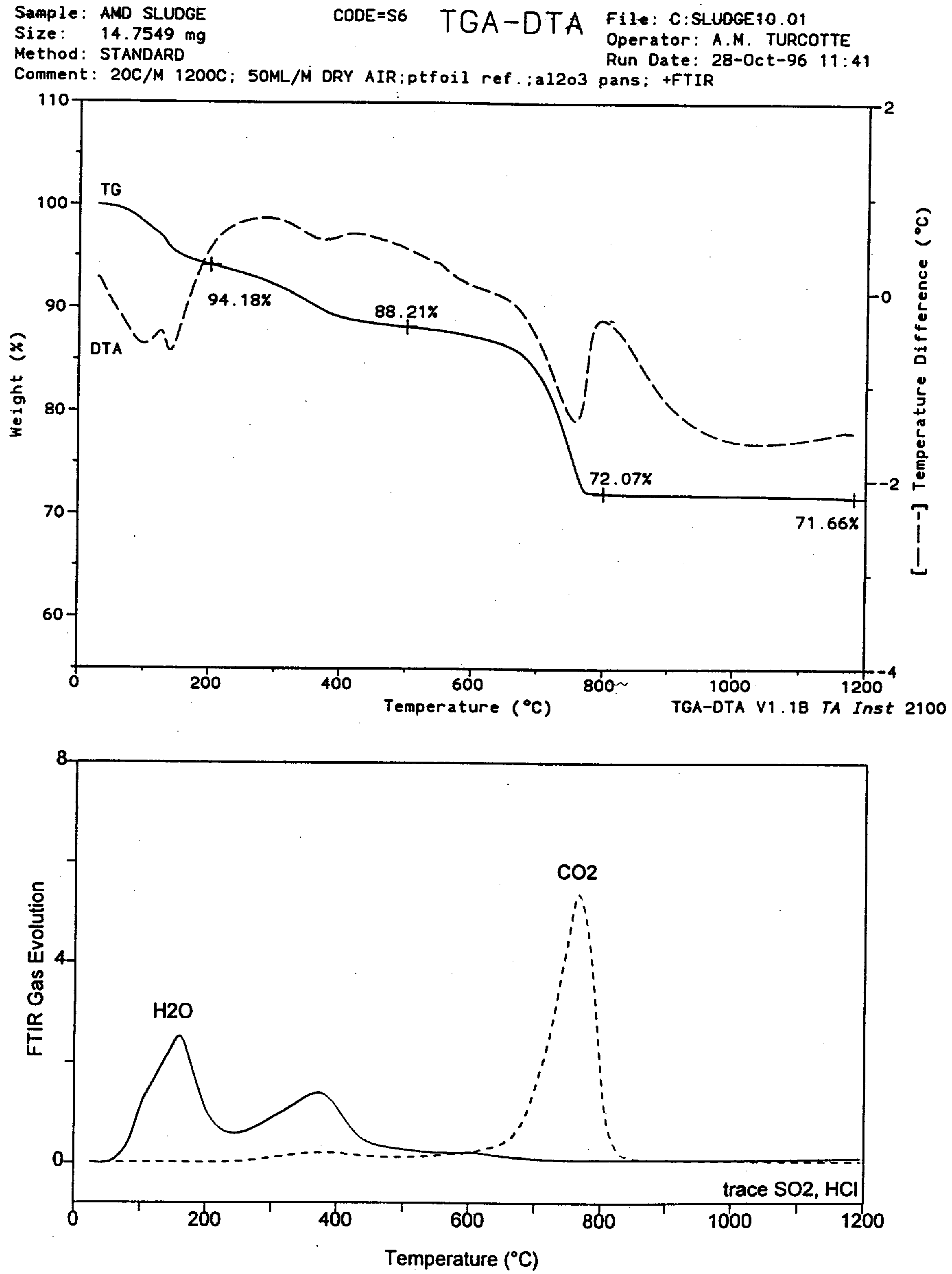


Figure H-8: TG/DTA/FTIR diagram for F-5 aged sludge in air.



Sample: AMD SLUDGE
Size: 14.7549 mg
Method: STANDARD
Comment: 20C/M 1200C; 50ML/M DRY AIR;ptfoil ref.;al2o3 pans; +FTIR
CODE=S6
TGA-DTA
File: C:SLUDGE10.01
Operator: A.M. TURCOTTE
Run Date: 28-Oct-96 11:41

Figure H-9: TG/DTA/FTIR diagram for S-6 fresh sludge in air.

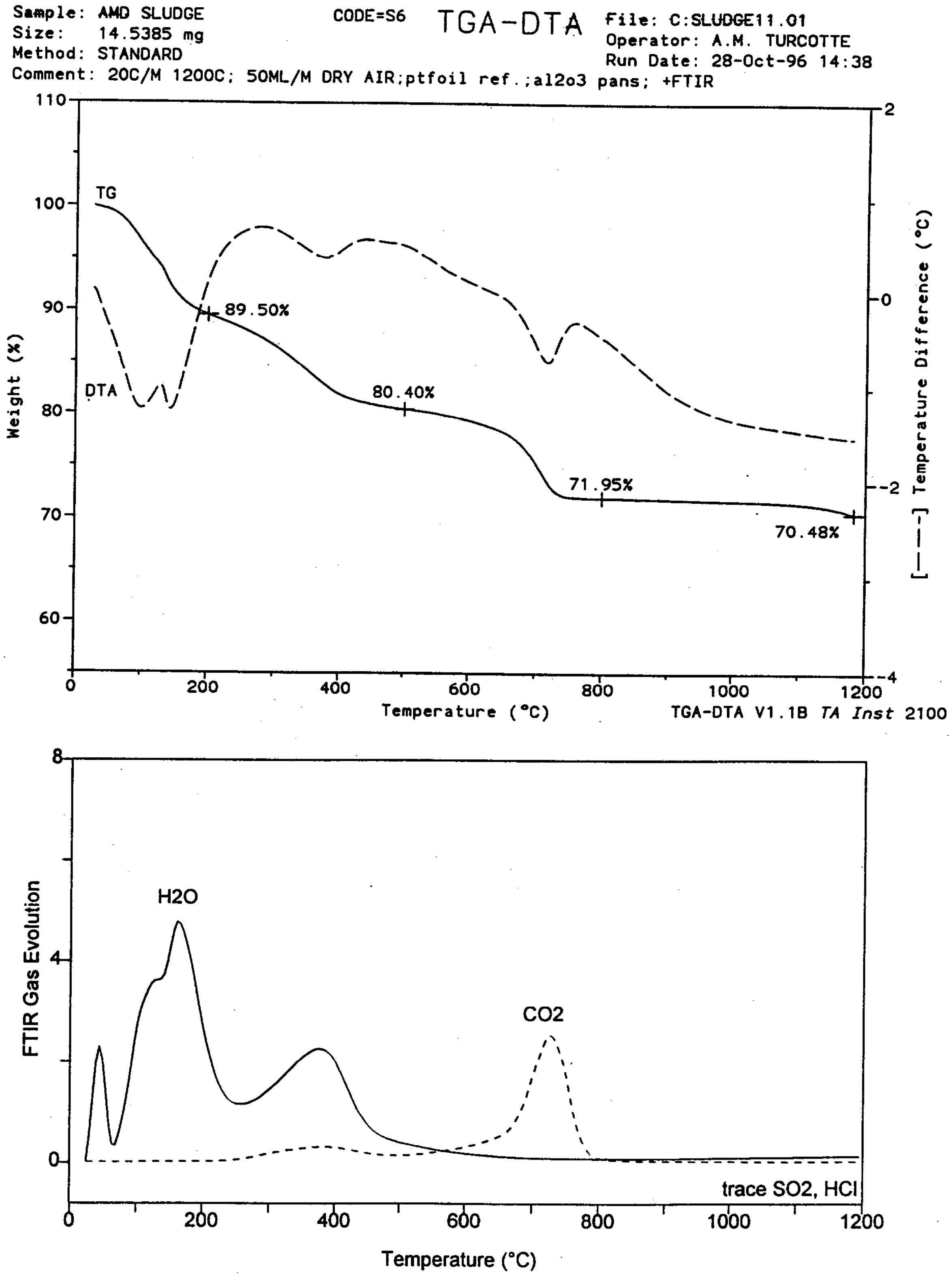


Figure H-10: TG/DTA/FTIR diagram for S-6 aged sludge in air.

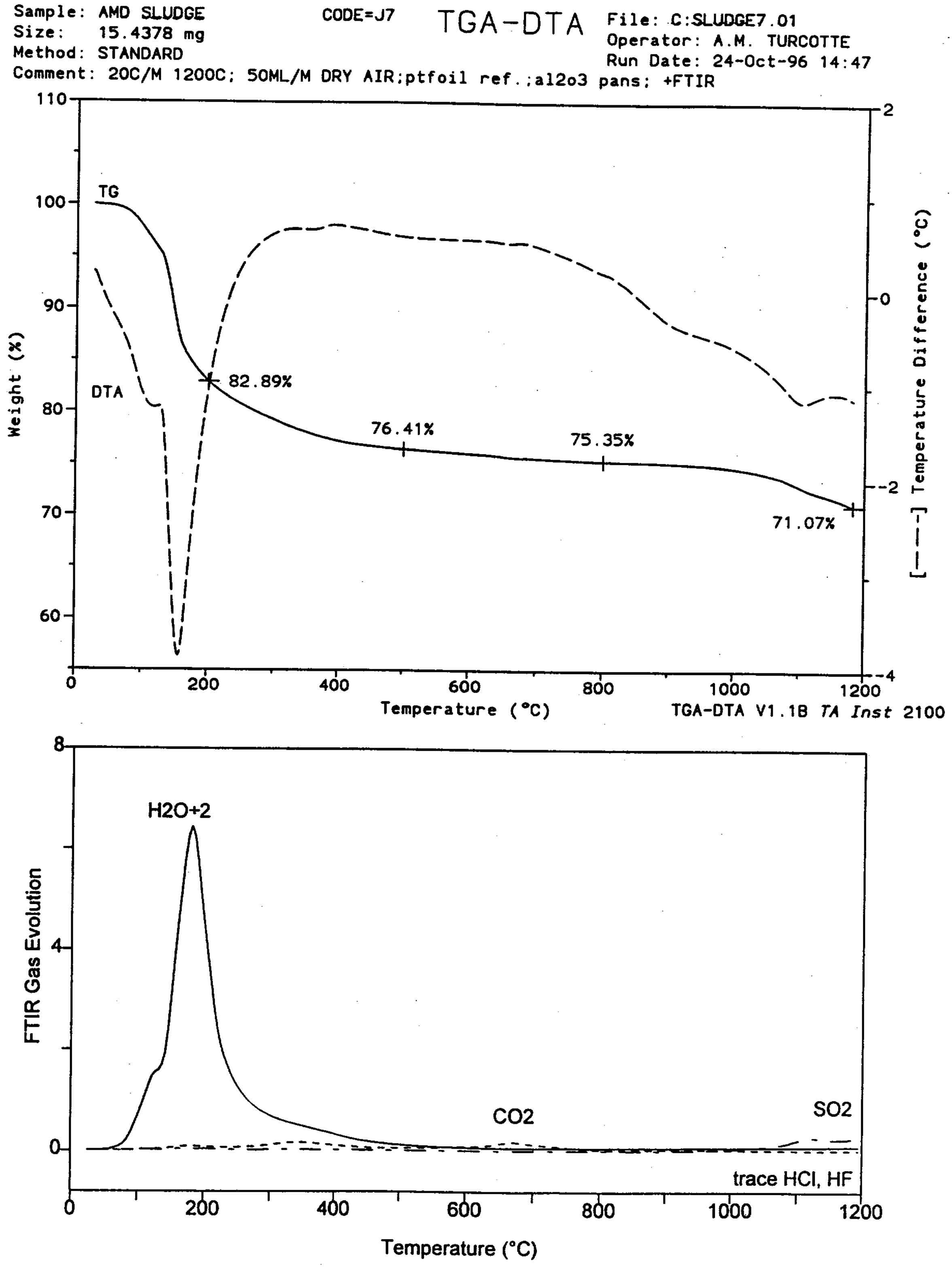


Figure H-11: TG/DTA/FTIR diagram for J-7 fresh sludge in air.

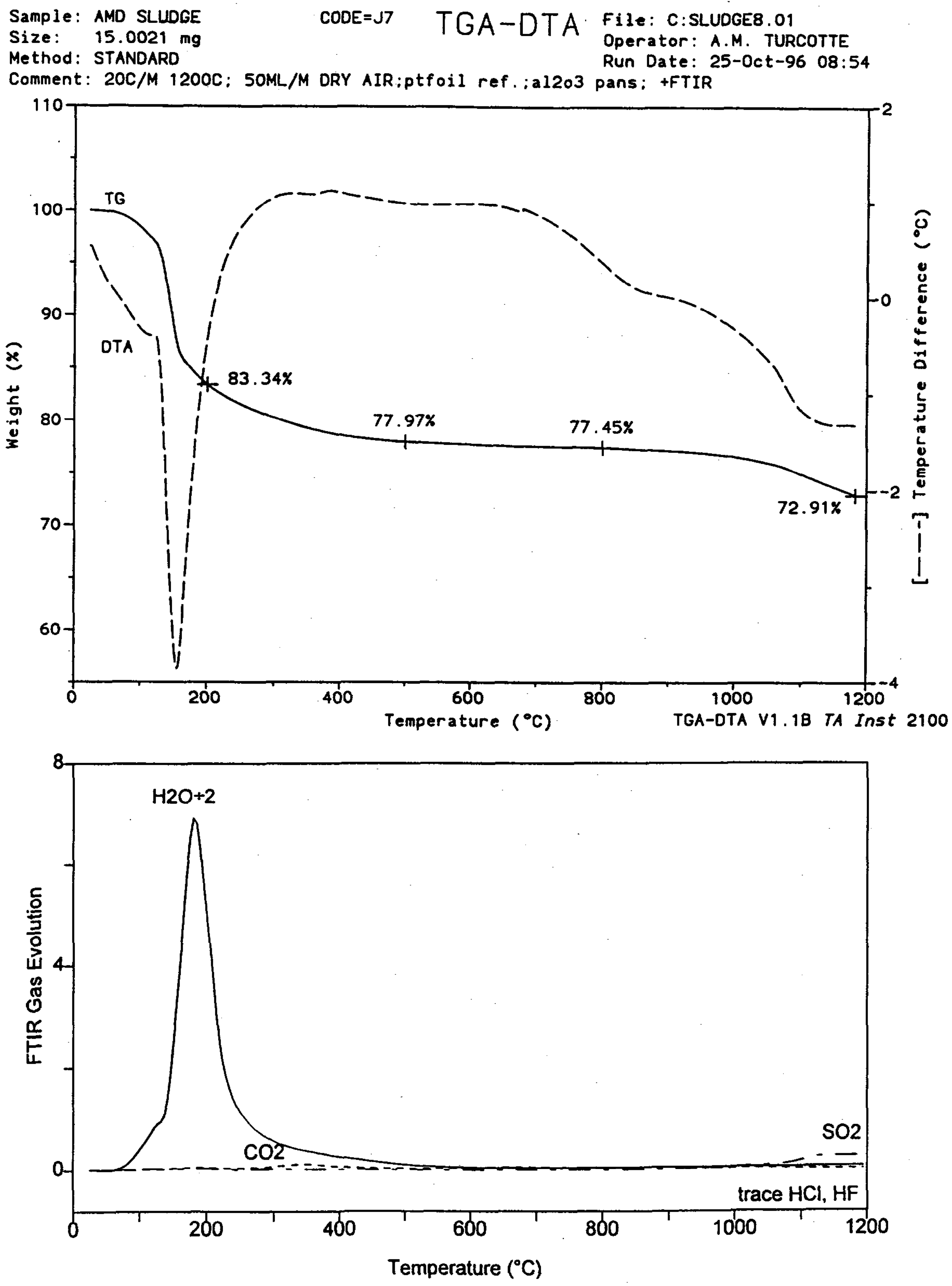


Figure H-12: TG/DTA/FTIR diagram for J-7 aged sludge in air.

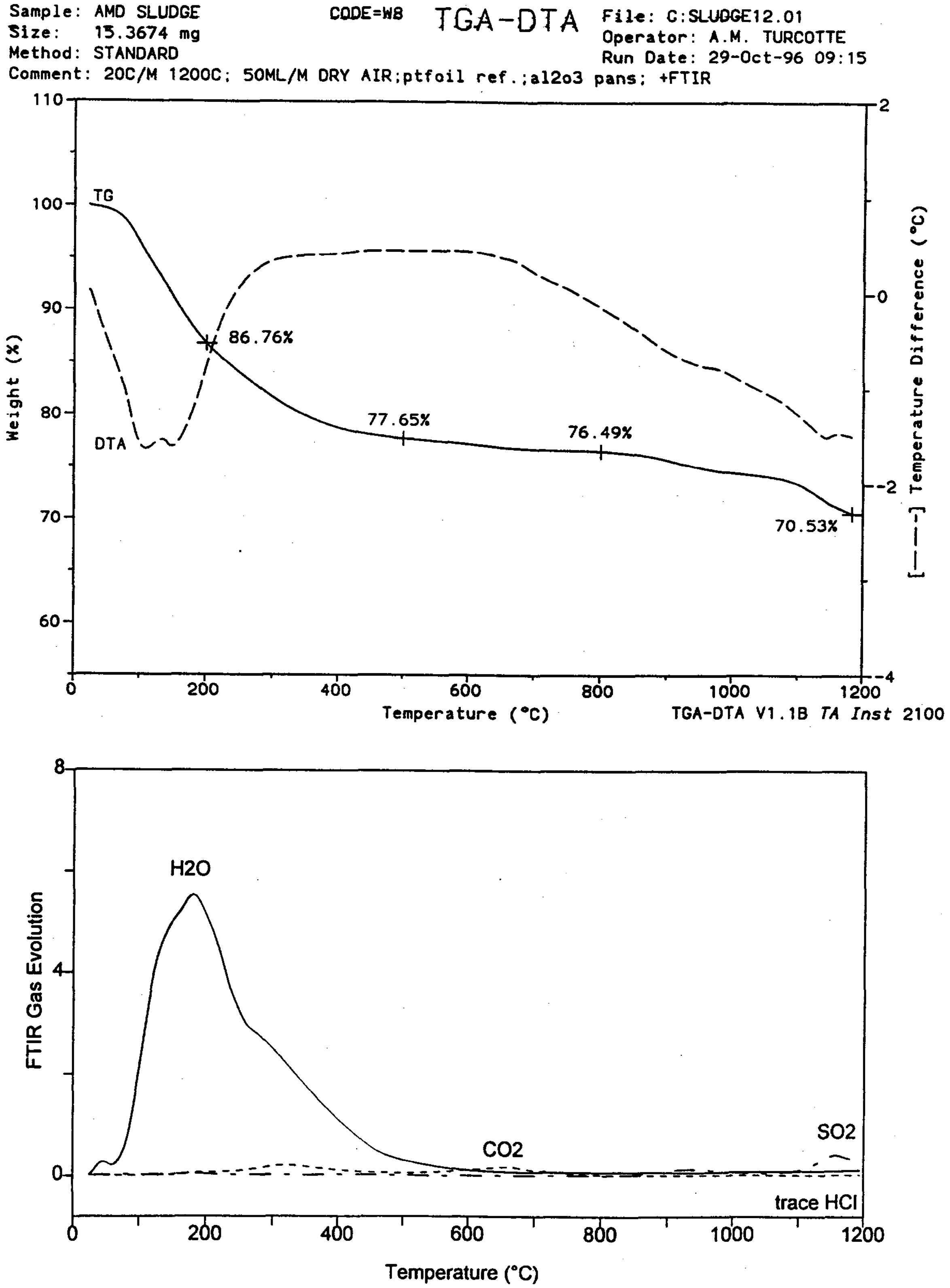


Figure H-13: TG/DTA/FTIR diagram for W-8 fresh sludge in air.

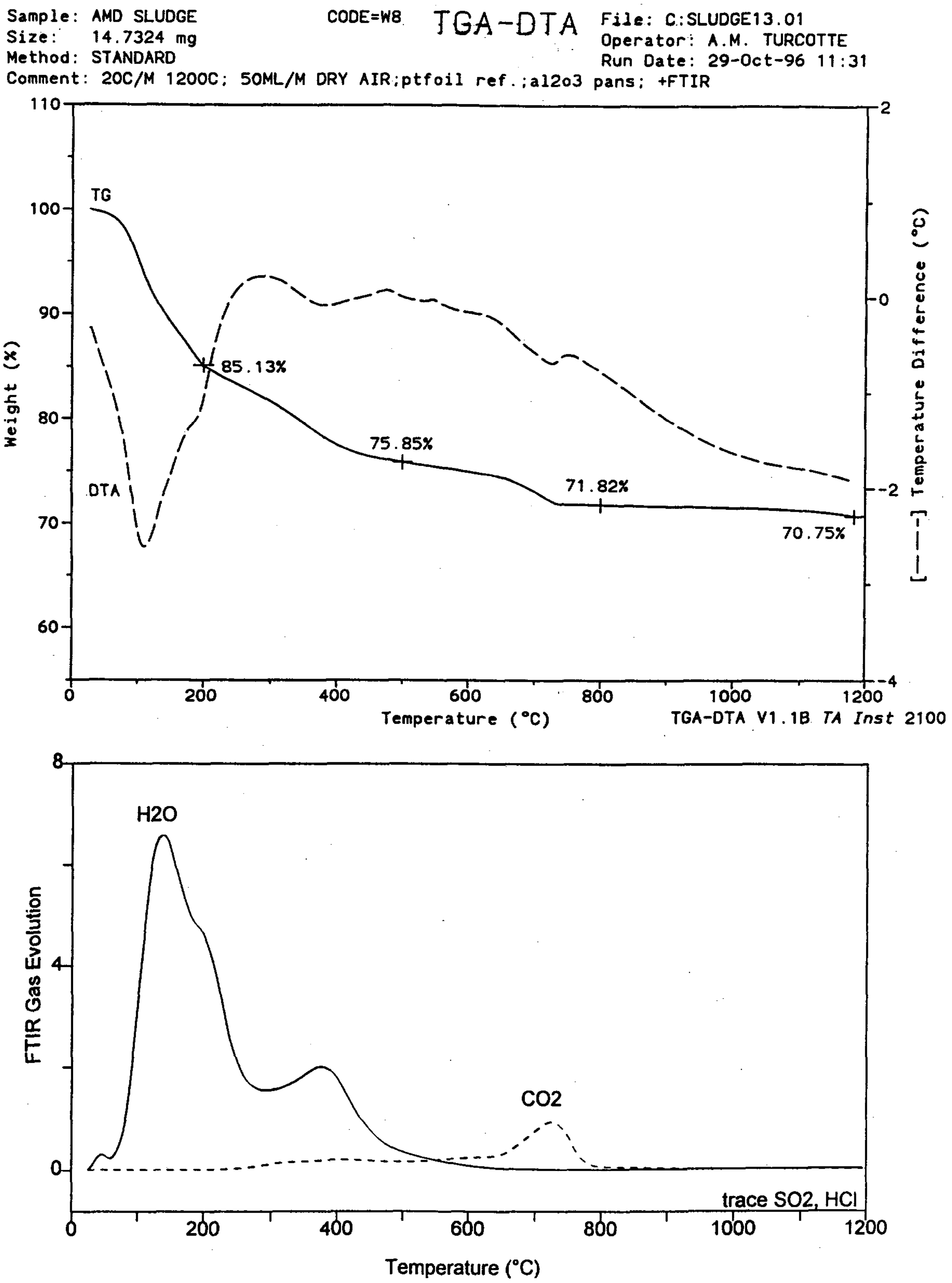


Figure H-14: TG/DTA/FTIR diagram for W-8 aged sludge in air.

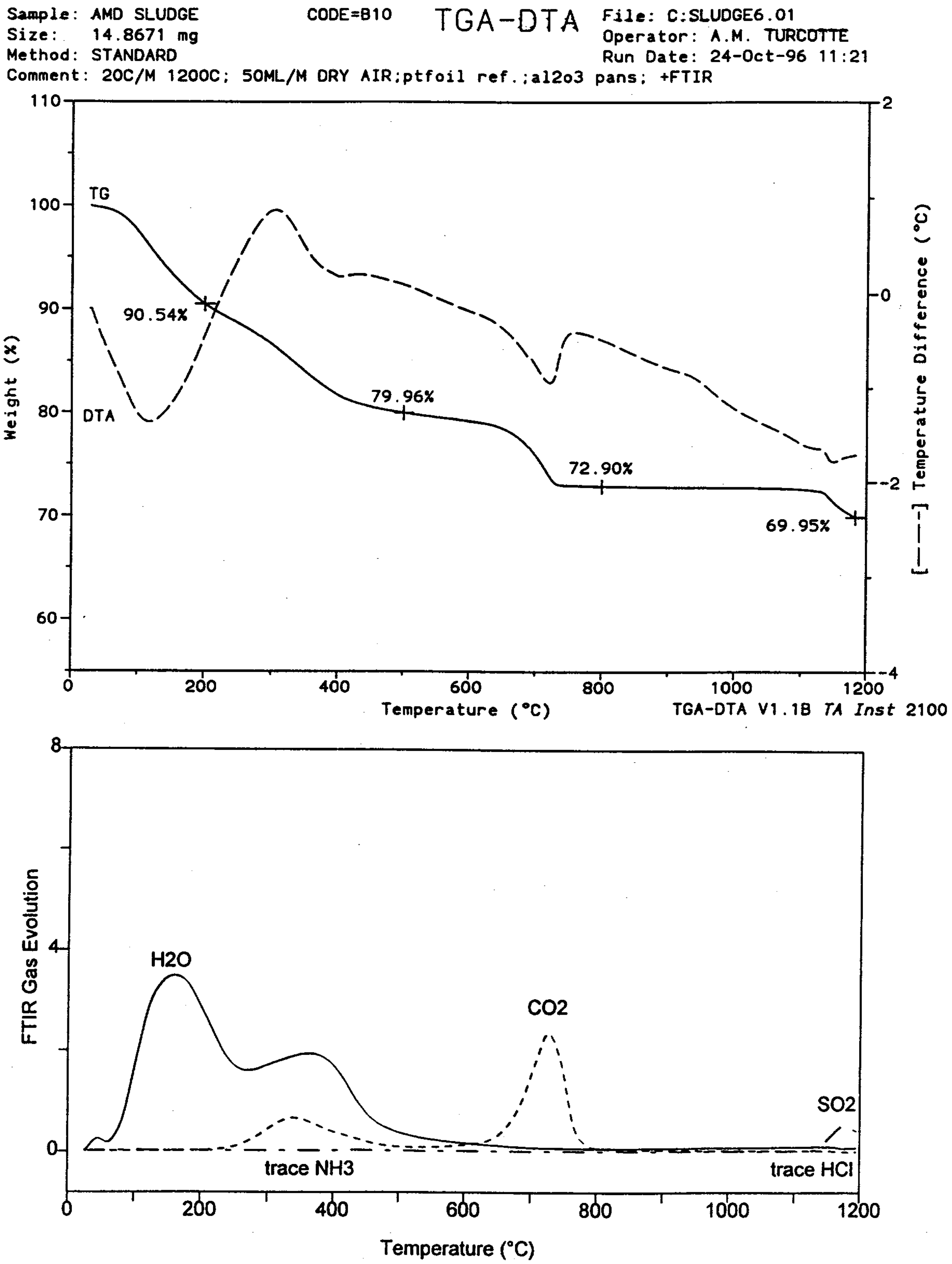


Figure H-15: TG/DTA/FTIR diagram for B-10 fresh sludge in air.

APPENDIX I: REGULATORY CONTEXT FOR LEACH TESTING OF AMD TREATMENT SLUDGES

(May 1997)

CONTENTS

LIST OF TABLES

Note: See Section 9.0 “References” in the main body of the report for details of the references used in this appendix.

REGULATORY CONTEXT FOR LEACH TESTING OF AMD TREATMENT SLUDGES

AMD treatment sludges may be subject to hazardous waste management regulations with respect to their transport or disposal. In general, legislation defines hazardous characteristics of a waste by one or both of the following systems: i) static lists of specific wastes according to chemical content, waste types or waste origins; and ii) application of hazardous characteristics test procedures (e.g., leachate extraction tests). Leachate extraction tests are generally applied when the hazardous characteristics of wastes cannot be determined from the lists of specific wastes, waste streams or processes; or from other hazardous characteristics test procedures (e.g., for corrosivity, flammability). Leachate extraction tests are undertaken to evaluate if the waste is capable of yielding a leachate which exhibits toxicity (Fenco MacLaren Inc. 1995).

I.1 Leachability Testing Methods

The leachability of solid wastes has been extensively studied and documented in the literature. For example, four summary reports (Fenco MacLaren Inc. 1995; Wilson 1994a; Environment Canada 1990; United States Environmental Protection Agency 1989) and two experimental papers (Jackson et al. 1984, Côté and Constable 1982) provide detailed discussions of solid waste leach tests. Although these documents do not deal with AMD treatment sludges, they do provide a framework of general concepts and test methods which can be applied in the examination of sludge leachability. The following paragraphs outline pertinent background information from these reports.

I.1.1 General information on leach tests

Ordinarily, a leach test involves contacting a waste material with a liquid to determine which components in the waste will dissolve in the liquid. A leach test can be used for either waste classification or leachate quality prediction. When a test is used for waste classification (e.g., as hazardous or non-hazardous), a standard methodology is preferred since it allows comparison among laboratories and ensures better reproducibility of the results. When a test is used for predicting the quality of leachate in a field disposal situation, the major concern is to make the test conditions as site specific as possible.

Extraction tests (or batch tests) refer to leach tests that involve agitation of a waste in a predefined quantity of leaching solution (leachant) for a specified time. Leaching is assumed to reach equilibrium by the end of the extraction period; therefore, extraction tests are generally assumed to determine the maximum, or saturated, leachate concentrations under a given set of test conditions. Extraction tests are short term (lasting from hours to days), have good reproducibility, are simplistic in design (can be set up and used routinely by laboratory personnel), and are often employed to simulate 'worst case' leaching conditions (because of particle size reduction and agitation). Standard regulatory leach tests are extraction tests.

In dynamic leach tests, the leachant is continuously or intermittently renewed to maintain a driving force for leaching. Dynamic tests can provide information about the kinetics of contaminant mobilization. Serial extraction tests involve the separation of leachate from solids, and replacement with fresh leachant until the desired number of leaching periods (elutions) has been completed. Column leach tests are dynamic tests in which a column is packed with a porous solid waste through which leachant is passed at a specified rate. Column tests are considered to be more representative of field leaching conditions than extraction tests since the mechanism of contacting a fixed body of waste with a transient liquid resembles the leaching mechanism imparted by gravity flow of liquid through a waste disposal site. However, channelling effects, non-uniform packing of the wastes, biological growth, and clogging of the column all contribute to problems with the reproducibility of test results. As well, column tests often require longer time frames, from weeks to months, to obtain results.

There are eight experimental variables which define leach tests. A short explanation of each follows:

i) sample preparation - this may include subsampling, surface washing and particle size reduction; the latter is performed in order to reduce the time required to reach steady-state conditions by increasing the surface area of contact between the waste and the leachant.

ii) leachant composition - commonly used leachants include water, site liquid and chemical solution (e.g., acetic acid).

iii) method of contact - in extraction tests, agitation methods include shaking, stirring, tumbling and gas bubbling.

iv) liquid-to-solid ratio - the ratio should be low enough to avoid dilution of contaminants to less than analytical detection limits, and high enough to prevent solubility constraints from limiting the amount of contaminants that can be leached from the waste (usually between 1:1 and 100:1 as mL:g depending upon the purpose of the test).

v) contact time - in extraction tests, this is the duration of the test; in dynamic tests, it is a function of the flow rate, or the number of elutions, in addition to the test duration.

vi) number of elutions - typically only one elution is performed; however, successive elutions can reveal the release pattern of a contaminant over time.

vii) temperature - for convenience, most leach tests are performed at room temperature.

viii) leachate separation - leachates are commonly separated from agitated non-monolithic wastes by filtration using a 0.45 μm membrane filter (a convention used to define soluble species).

I.1.2 Leach test protocols

Numerous leach protocols have been developed to test solid wastes. Comparisons of an extensive number of waste leach tests can be found in the literature (Fenco MacLaren Inc. 1995; Wilson 1994a; Environment Canada 1990; United States Environmental Protection Agency 1989). The protocols selected for review in this report (see Tables I-1A and I-1B) consist of Canadian and American regulatory procedures, as well as some of the standard research and test methods sponsored by recognized organizations. None of these leach tests has been specifically designed for evaluating AMD treatment sludge leachability. However, some of the research and test methods simulate an actual sludge environment more closely than do the regulatory leach protocols (e.g., through use of simulated acid rain rather than acetic acid as a leachant). Note that column tests have not been considered because the percolation rate of the leachant through finely divided solids such as sludges would likely be prohibitively slow. A comparative summary of the leach protocols in relation to testing a wet sludge for inorganic contaminants is presented in Tables I-2A and I-2B. Further details of the leach protocols (including the intended purpose, test variables, and noteworthy features) are provided in Appendix J.

Table I-1A: Regulatory Leach Protocols Selected for Review

Protocol Name and Proponent	Reference
Extraction Procedure Toxicity Test (EP Tox) - Method 1310 U.S. Environmental Protection Agency (U.S. EPA)	U.S. EPA (1986a)
Toxicity Characteristic Leaching Procedure (TCLP) - Method 1311 U.S. Environmental Protection Agency (U.S. EPA)	U.S. EPA (1990)
Leachate Extraction Procedure (LEP) Ontario Ministry of the Environment and Energy	Government of Ontario (1994b)
CGSB Leachate Extraction Procedure (CGSB LEP) Canadian Department of Transport	Canadian General Standards Board (1987)
Special Waste Extraction Procedure (SWEP) British Columbia Ministry of the Environment	Government of British Columbia (1992)
Leachate Extraction Procedure (LEP) Manitoba Ministry of the Environment	Government of Manitoba (1987)
Procedure for Assessing the Characteristics of Solid Waste and Pumpable Sludges Québec Ministère de l'Environnement et de la Faune	Government of Québec (1985)
Waste Extraction Test (WET) California Environmental Protection Agency	Government of California (1996)

Table I-1B: Research Leach Protocols Selected for Review

Protocol Name and Proponent	Reference
Synthetic Precipitation Leaching Procedure (SPLP) - Method 1312 U.S. Environmental Protection Agency (U.S. EPA)	U.S. EPA (1992)
Multiple Extraction Procedure (MEP) - Method 1320 U.S. Environmental Protection Agency (U.S. EPA)	U.S. EPA (1986b)
Monofill Waste Extraction Procedure (MWEP) U.S. Environmental Protection Agency (U.S. EPA)	Garrett et al. (1984)
Shake Extraction of Solid Waste with Water (D-3987) American Society for Testing and Materials (ASTM)	ASTM (1993a)
Sequential Batch Extraction of Waste with Water (D-4793) American Society for Testing and Materials (ASTM)	ASTM (1993b)
Single Batch Extraction Method for Wastes (D-5233) American Society for Testing and Materials (ASTM)	ASTM (1993c)
Sequential Batch Extraction of Waste with Acidic Extraction Fluid (D-5284) American Society for Testing and Materials (ASTM)	ASTM (1993d)

Table I-2A: Comparison of Regulatory Leach Protocols[1]

Protocol	Sample Preparation (multi-phase wastes)	Sample Mass	Leachant	Liquid-to-Solid Ratio	Method of Contact	Duration
U.S. EPA EP Tox 1310	Solid/liquid phase separation. Filter (0.45 µm) with vacuum or pressure (up to 75 psi).	Min. 100 g (liquid and solid)	Deionized water adjusted to pH 5.0 with periodic additions of 0.5 N acetic acid[2]	16:1 at start; 20:1 at end	Continuous agitation	24-28 h
U.S. EPA TCLP 1311	Solid/liquid phase separation. Filter (0.6-0.8 µm) with vacuum or pressure (up to 50 psi).	Min. 100 g (liquid and solid)	Choice of acetic acid solution: pH 4.93 (buffered) or pH 2.88	20:1	End-over-end agitation (30 rpm)	18 h
Ont. LEP	Liquid/solid phase separation. Filter (0.45 µm) with pressure (up to 71 psi).	50 g dry weight basis (of de-watered undried material)	Reagent water[3] adjusted to pH 5.0 with periodic additions of 0.5 N acetic acid[2]	Same as EP Tox	End-over-end agitation (10 rpm)	24 h
CGSB LEP	Same as Ont. LEP	Same as Ont. LEP	Same as Ont. LEP	Same as Ont. LEP	Same as Ont. LEP	Same as Ont.LEP
B.C. SWEP	Same as Ont. LEP	Same as Ont. LEP	Same as Ont. LEP	Same as Ont. LEP	Same as Ont. LEP	Same as Ont. LEP
Man. LEP	Same as Ont. LEP	Same as Ont. LEP	Same as Ont. LEP	Same as Ont. LEP	Same as Ont. LEP	Same as Ont. LEP
Québec Leach Protocol	No liquid/solid phase separation.	5-100 g dry weight basis (depending on % solids)	Buffered acetic acid (pH 4.5) plus distilled water	10:1	End-over-end agitation (5-30 rpm)	24 h
Cal. WET	As received if non-filterable, non-millable sludge. Phase separation if filterable (0.45 µm). Solids milled to pass 2.0-mm sieve.	50 g (or less if sample is filterable solid/ liquid mixture)	0.2 M sodium citrate at pH 5.0	10:1	Vigorous agitation	48 h

[1] Further details are provided in Appendix J.

[2] Up to a specified maximum addition of acetic acid; hence, a pH of 5.0 may not be attained for highly alkaline samples.

[3] Type IV reagent water meets ASTM Specification D-1193.

Table I-2B: Comparison of Research Leach Protocols[1]

Protocol	Sample Preparation	Sample Mass	Leachant	Liquid-to-Solid Ratio	Method of Contact	Duration
U.S. EPA SPLP 1312	Same as TCLP	Same as TCLP	Mixture of 60/40 weight percent sulphuric/nitric acids; pH 4.20 or pH 5.00	Same as TCLP	Same as TCLP	Same as TCLP
U.S. EPA MEP 1320	Same as EP Tox	Same as EP Tox	1st extraction - same as EP Tox; then a mixture of 60/40 weight percent sulphuric/nitric acids at pH 3	Same as EP Tox	Same as EP Tox	24-28 h for 1st extraction; then 24 h each per extraction (9 times)
U.S. EPA MWEP	Test designed for solid wastes; no direction provided regarding multi-phase wastes.	not specified	Distilled water or site water	10:1	End-over-end agitation	18 h per extraction (4 or more times)
ASTM D-3987	As received. Test designed for solid wastes; no direction provided regarding multi-phase wastes.	70 g	Reagent water[2]	20:1	End-over-end agitation (29 rpm)	18 h
ASTM D-4793	As received. Mix thoroughly.	100 g dry weight basis	Reagent water[2]	20:1	End-over-end agitation (30 rpm)	18 h per extraction (10 times)
ASTM D-5233	Solid/liquid phase separation. Filter (0.6-0.8 µm) with vacuum or pressure (up to 50 psi).	Same as TCLP	Same as TCLP	Same as TCLP	Same as TCLP	Same as TCLP
ASTM D-5284[3]	As received. Mix thoroughly.	100 g dry weight basis	Mixture of 60/40 weight percent sulphuric/nitric acids; pH of local acid rain	Same as SPLP	Same as SPLP	18 h per extraction (10 times)

[1] Further details are provided in Appendix J.

[2] Type IV reagent water meets ASTM Specification D-1193.

[3] ASTM is currently balloting the Draft Test Method for Shake Extraction of Mining Waste by the Synthetic Precipitation Leaching Procedure. This method is similar to the first extraction of ASTM D-5284.

Promulgation of EP Tox by the U.S. EPA has spawned the development of a whole family of similar regulatory protocols, including TCLP, Ontario LEP, CGSB LEP, B.C. SWEP, Manitoba LEP, Québec leach protocol, and California WET. While use of a single standard procedure of this nature within a jurisdiction simplifies the regulatory process, it does not take the actual disposal scenario into account. EP Tox is based upon a 95% municipal / 5% industrial co-disposal mismanagement scenario (Fenco MacLaren Inc. 1995, U.S. EPA 1995). This does not represent normal management practice for AMD treatment sludges which are not subject to organic acids, nor are they commingled with materials which have potential for organic acid formation. Consequently, the mobility of contaminants may be substantially over predicted by these protocols.

I.2 Regulatory Context

Hazardous waste regulations may apply to AMD treatment sludges in many jurisdictions. However, decisions about disposal of these materials may also be closely linked to other regulations which govern mining operations (e.g., effluent quality regulations). Mine operation permits or licences may incorporate site-specific requirements that determine how AMD treatment sludges are managed. The following paragraphs review current hazardous waste regulations and other pertinent regulations and guidelines as they apply to the leachability testing of AMD treatment sludges.

I.2.1 United States Environmental Protection Agency

The United States aroused concern in its mining community about the regulation of mine waste streams with the passage of the *Resources Conservation and Recovery Act* (RCRA) in 1976. Mining and minerals processing wastes that were ignitable, corrosive, reactive, or extraction procedure (EP) toxic (i.e., leachable) could be classified as hazardous and could, therefore, require rigorous and expensive methods of treatment and disposal. In response to concerns expressed by the mining industry, some high volume wastes with relatively low potential environmental hazard were defined as 'special wastes' and were excluded from regulation until studies on the nature of these wastes could be completed (Shea-Albin and Fitch 1991). For example, mine tailings qualify for the exclusion as a solid waste from the extraction or beneficiation of ores and minerals (United States Code of Federal Regulations - 40CFR261.4(b)7). However, AMD treatment sludges do not appear on the list of excluded wastewater treatment sludges (40CFR261.4(b)6).

The Extraction Procedure Toxicity Test (EP Tox) was promulgated in 1980 by the United States Environmental Protection Agency under the RCRA. The development of this test was based upon an assumption that the waste material is saturated with the organic acids present in a municipal landfill site and thus, the test uses acetic acid (pH 5) as a leaching medium. EP Tox is very conservative with respect to AMD treatment sludges since it does not represent the conditions under which sludges are disposed. If sludges are maintained in an acidic environment (e.g., exposure to acid rain), the acids present are inorganic which are less aggressive than organic acids with respect to metal mobility.

EP Tox defined the toxicity of a waste by attempting to measure the potential for the toxic constituents present in the waste to leach out, thereby contaminating groundwater and surface water, and by extension drinking water sources, at levels of health or environmental concern. The National Interim Primary Drinking Water Standards (NIPDWS) were used as the basis for the contaminant criteria because at that time, the NIPDWS were the only health-based regulatory standards available. To account for the likely dilution and attenuation of the toxic constituents that would occur as they travelled from the point of generation to a drinking water source, the EPA multiplied the NIPDWS by a 'dilution and attenuation factor' (DAF) of 100. The DAF of 100 was not derived from any model or empirical data, but rather was an estimated factor that the EPA believed would indicate substantial hazard (U.S. EPA 1995).

EP Tox was used to classify wastes as hazardous or non-hazardous based on maximum permissible concentrations in the leachate for eight metals, four pesticides and two herbicides from the NIPDWS of the day. The U.S. EPA criteria for metals are given in Table I-3. Many of these limits do not correlate by a factor of 100 with the current U.S. drinking water standards (see Table I-4) since drinking water quality criteria have been amended since 1980. The current drinking water standards could be used, however, as the basis for a review of the regulated limits.

Table I-3: Comparison of Toxic Leachate Constituents

Constituent	Regulated Limits (mg/L)[1]							
	TDGR[2]	Québec[3] (current)	Québec[4] (draft)	Ontario[5] and Manitoba[6]	B.C.[7]	Alberta[8]	Cal. EPA[9] (STLC)	U.S. EPA[10]
Arsenic (As)	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0
Barium (Ba)	100.0		100	100.0	100.0	100.0	100	100.0
Boron (B)	500.0		500	500.0	500.0	500.0		
Cadmium (Cd)	0.5	2.0	0.5	0.5	0.5	1.0	1.0	1.0
Chromium (Cr)	5.0	5.0	5.0	5.0	5.0	5.0	5	5.0
Cobalt (Co)						100.0	80	
Copper (Cu)		10			100.0	100.0	25	
Iron (Fe)						1000.0		
Lead (Pb)	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0
Mercury (Hg)	0.1	0.2	0.1	0.1	0.1	0.2	0.2	0.2
Nickel (Ni)		10				5.0	20	
Selenium (Se)	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
Silver (Ag)			5.0	5.0	5.0	5.0	5	5.0
Uranium (U)	2.0		2.0	2.0	10.0	2.0		
Zinc (Zn)		10			500.0	500.0	250	

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[1] Numerical values are recorded as given in the source.

[2] Transportation of Dangerous Goods Regulations, Part III, ∮ 3.27(3) (Transport Canada 1997).

[3] Hazardous Waste Regulation [Q-2, r.3.01] Schedule III (Government of Québec 1992).

[4] Draft Hazardous Materials Regulation (Government of Québec 1995); comments from the public in response to this draft have included a recommendation that silver be removed from the list of contaminants in order to harmonize with the TDGR (St-Laurent 1996).

[5] General - Waste Management Regulation (Ont. Reg. 347) Schedule 4 (Government of Ontario 1994b).

[6] Classification Criteria for Products, Substances and Organisms Regulation (Man. Reg. 282/87) Schedule B, Table 1 (Government of Manitoba 1987).

[7] Special Waste Regulation (B.C. Reg. 63/88) Schedule 4, Table 1 (Government of British Columbia 1992). Draft amendments to this regulation, which will be released for public consultation in 1997, will likely include recommendations for changes to the Leachate Quality Standards (Bindra 1996).

[8] Schedule to the Alberta User Guide for Waste Managers, Table 2 (Government of Alberta 1995).

[9] California Code of Regulations, Title 22, Chapter 11, ∮ 66261.24(a)(2)(A) (Goverment of California 1996).

[10] United States Code of Federal Regulations, Title 40, Chapter 1, ∮ 261.24(a), Table 1.

Table I-4: Drinking Water Quality Criteria (concentrations in mg/L)

Constituent	Canadian Drinking Water[1]	U.S. Primary Drinking Water[2]	U.S. Secondary Drinking Water[3,4]
Arsenic (As)	0.025[5]	0.05	
Barium (Ba)	1.0	2	
Boron (B)	5.0[5]		
Cadmium (Cd)	0.005	0.005	
Chromium (Cr)	0.05	0.1	
Cobalt (Co)	-[6]		
Copper (Cu)	≤1.0[7]	1.3[8]	1.0
Iron (Fe)	≤0.3[7]		0.3
Lead (Pb)	0.01	0[8]	
Mercury (Hg)	0.001	0.002	
Nickel (Ni)	-[6]		
Selenium (Se)	0.01	0.05	
Silver (Ag)	-[9]		0.1
Uranium (U)	0.1[6]		
Zinc (Zn)	≤5.0[7]		5
pH	6.5-8.5		6.5-8.5

[1] "Canadian Water Guidelines - Summary of Guidelines for Water Quality in Canada 1995". Health Canada and Environment Canada, Catalogue No. H49-95/1995E.

[2] United States Code of Federal Regulations, Title 40, SubChapter D - Water Programs, Part 141 National Primary Drinking Water Regulations, SubPart F, Section 141.51 and SubPart G, Section 141.62.

[3] United States Code of Federal Regulations, Title 40, SubChapter D - Water Programs, Part 143 National Secondary Drinking Water Regulations, Section 143.3.

[4] Secondary Drinking Water Standards are designed to protect the aesthetic qualities of the water and are not federally enforceable. They are issued for states to use as guidelines.

[5] Interim water guideline.

[6] Under review for addition to the Guidelines for Canadian Drinking Water Quality (GCDWQ) or possible changes to the current value.

[7] Aesthetic objective.

[8] Maximum contaminant level goal (MCLG) - non-enforceable health goal.

[9] Parameter identified as not requiring a numerical guideline.

The Toxicity Characteristic Leaching Procedure (TCLP) was developed in 1984 under the *Hazardous and Solid Waste Amendments* to RCRA. It was promulgated in 1986 under the *Hazardous Waste Management System Land Disposal Restrictions* to be used as the standard criterion for evaluating hazardous waste treatment technologies; and in 1990 for use in hazard determination to replace EP Tox. TCLP incorporates a number of procedural and equipment modifications compared to EP Tox. The leaching medium is acetic acid at either pH 4.93 (buffered) or 2.88 depending on the neutralizing capacity of the waste as determined by a preliminary evaluation. The metal concentrations in TCLP leachates generally tend to be greater than those in EP Tox leachates (U.S. EPA 1989).

The applicability of the TCLP to mining wastes is currently being debated in the United States. The National Mining Association contends that the Synthetic Precipitation Leaching Procedure (SPLP) is a more appropriate test than the TCLP for identifying hazardous mining wastes (U.S. EPA 1995). The SPLP is a variation of the TCLP which uses synthetic acid rain as a leaching medium. SPLP more closely mimics actual sludge disposal scenarios. The EPA has used the SPLP to screen wastes that are 'clearly not low hazard' for the purpose of determining which mineral processing wastes would qualify for temporary exclusion from regulation as a 'special waste' (U.S. EPA 1995). The Agency has also used the SPLP in remedial investigations of Superfund sites (inactive and abandoned hazardous substances sites) containing mining wastes (Wilson 1994a). However, the EPA maintains that the use of one leach test, TCLP, is the appropriate approach for hazard classification (U.S. EPA 1995). Federal regulations in the United States currently require that AMD treatment sludges be tested using the TCLP. If a sludge fails the TCLP, the sludge must be treated and disposed of as a hazardous waste, at considerable cost.

I.2.2 California

State environmental programs which regulate the mining industry may simply enact federal legislation or they may design their own programs to be more stringent, but not less so, than the federal minimum requirements. In California, hazardous wastes are those wastes that are listed or characteristically hazardous either under the federal RCRA regulations (RCRA hazardous wastes) or under California law and regulations (non-RCRA hazardous wastes). California defines some industrial waste as hazardous under state law that is considered non-hazardous under federal definitions, including some wastes that are exempt or excluded under RCRA, such as special wastes (e.g., mining wastes). California hazardous waste regulations are published in the *California Code of Regulations*, Title 22, Division 4.5, entitled "Standards for the Management of Hazardous Waste".

The California Waste Extraction Test (WET) is based on EP Tox. However, the use of a chelating agent, sodium citrate, rather than acetic acid as the leachant may make this test more aggressive towards certain waste components. WET was promulgated in 1985 to identify hazardous non-RCRA wastes according to the Persistent and Bioaccumulative Toxic Substances criteria which include the Soluble Threshold Limit Concentration (STLC) and the Total Threshold Limit Concentration (TTLC) for each substance. A waste is considered to exhibit the characteristic of toxicity if any of the following conditions are met: i) it fails the TCLP; ii) the WET leachate contains a substance listed in the Persistent and Bioaccumulative Toxic Substances criteria at a concentration greater than or equal to its STLC (see Table I-3 for some of the inorganic substances); or iii) its chemical composition includes a substance listed in the Persistent and Bioaccumulative Toxic Substances criteria at a concentration greater than or equal to its TTLC.

Disposal of waste on land in California is regulated relative to protection of water quality by the *California Code of Regulations*, Title 23, Subchapter 15. California distinguishes among three subcategories of non-hazardous waste; these subcategories determine the type of waste management unit appropriate for the waste. A 'designated waste' is a non-hazardous waste that contains constituents which could be released at concentrations in excess of applicable water quality objectives, or that could cause degradation of state waters, but is not a threat to human health. Because of their high metal loadings, AMD treatment sludges may be classified as designated wastes and consequently, they may require treatment and disposal in a highly engineered waste management unit. Treatment would be required for many sludges because the state currently does not allow materials containing higher than 50% moisture content to be landfilled (Environmental Resource Centre 1996).

I.2.3 Canada - Transportation of Dangerous Goods

In 1987, the Canadian General Standards Board (CGSB) accepted the Leachate Extraction Procedure (LEP) 164-GP-1MP as a provisional standard to identify materials that could pose a risk to the environment by producing a toxic leachate. The CGSB LEP is based on EP Tox. This test was incorporated into the *Transportation of Dangerous Goods Regulations* (TDGR) in 1989. The regulated concentration limits for a waste extract are provided in the TDGR (a partial list is given in Table I-3). The applicability of these criteria will likely be assessed in relation to current drinking water standards (see Table I-4) during the review process described below. Unlike the other hazard class criteria under TDGR, the classification of a material that is leachate toxic is meant to represent a risk to human health and the environment based on a long term period. The inclusion of the concepts of leachability and storage in the TDGR definitions permits provinces to utilize the provisions of TDGR for waste management control. Environment Canada, in conjunction with industry, provincial governments and other federal government departments, is currently undertaking a review of the applicability and suitability of the CGSB leachate extraction procedure for wastes destined for recycling operations. A study conducted for Environment Canada of various tests currently used to determine the toxicity of mobile constituents of wastes concluded that a test similar to the U.S. EPA TCLP is the most efficient, cost effective and presents a realistic approach for the determination of the inherent toxic leachability hazard of a waste (Fenco MacLaren 1995). The adoption of a TCLP-type leach test to replace the provisional CGSB LEP is under consideration by the Canadian Council of Ministers of the Environment (CCME) Hazardous Waste Task Group to provide for national harmonization, as well as to allow for closer conformity with the existing U.S. EPA leach test (Wittwer 1996).

I.2.4 Ontario

In the province of Ontario, *Regulation 347* under the *Ontario Environmental Protection Act* governs general waste management. The Ontario Leachate Extraction procedure (LEP), which was promulgated in 1985, is the regulatory extraction test used by the province. Only minor changes were made to EP Tox in developing this test. The Ontario LEP differs slightly from the CGSB LEP in the pH/volume adjustment procedure. Pertinent inorganic leachate quality criteria are given in Table I-3.

It is common practice in Ontario for AMD treatment sludges to be stored/disposed in the mill tailings area. Although mill tailings from a mine are exempted from *Regulation 347*, they are regulated under the *Ontario Water Resources Act* through Certificates of Approval which include site-specific enforceable parameters. The *Clean Water Regulation for the Metal Mining Industry* (Government of Ontario 1994a) enumerates the minimum requirements for effluent criteria at the mine site (see Table I-5). These criteria must be at least as stringent as the federal *Metal Mining Liquid Effluent Regulations* (MMLER) (Environment Canada 1977) which are also shown in Table I-5. Recent recommendations by the AQUAMIN initiative (AQUAMIN 1996) to revise the MMLER are awaiting approval. It was recommended that all current parameters along with cyanide be regulated in the revised MMLER, that a review of treatment technology be completed prior to revising the MMLER, and that appropriate concentration limits for all regulated parameters be established on the basis of this review.

I.2.5 Québec

Currently, in the province of Québec, hazardous wastes are controlled through the use of the *Hazardous Waste Regulation* [*Q-2, r.3.01*] (Government of Québec 1992) under the *Environment Quality Act* (*R.S.Q., chapter Q-2*). Mine tailings is the only exclusion related to mining which is listed in the regulation. The extraction test required by the regulation is outlined in the document "Procedure for Assessing the Characteristics of Solid Waste and Pumpable Sludges". It was developed in 1980 based on EP Tox, and promulgated in 1985. Part of the schedule of maximum allowable contaminant concentrations is given in Table I-3. Québec is one of only two jurisdictions in Canada which have criteria for all of copper, nickel and zinc - three metals which are commonly found in AMD treatment sludges.

A new *Hazardous Materials Regulation* is scheduled to be implemented in the summer of 1997; it will replace the existing *Hazardous Waste Regulation*. The Draft Regulation (Government of Québec 1995) indicates that the current extraction test will be replaced by the federal CGSB 164-GP-1MP. However, the Government received many comments from the

Table I-5: Effluent Quality Criteria (concentrations in mg/L)

Constituent	Ontario Monthly Mean[1]	Québec Monthly Mean[2]	B.C. Range[3]	Sask. Monthly Mean[4]	Nfld.	MMLER[5]		
						I[6]	II[7]	III[8]
Arsenic (As)	0.5	0.50	0.10-1.0	0.5	0.5	0.5	0.75	1.0
Barium (Ba)					5.0			
Boron (B)					5.0			
Cadmium (Cd)	0.001[9]		0.01-0.1		0.05			
Chromium (Cr)			0.05-0.3					
Cobalt (Co)			0.5-1.0					
Copper (Cu)	0.3	0.30	0.05-0.3	0.3	0.3	0.3	0.45	0.6
Iron (Fe)		3.00	0.3-1.0					
Lead (Pb)	0.2	0.20	0.05-0.2	0.2	0.2	0.2	0.3	0.4
Mercury (Hg)	0.001[9]		nil-0.005		0.005			
Nickel (Ni)	0.5	0.50	0.2-1.0	0.5	0.5	0.5	0.75	1.0
Selenium (Se)			0.05-0.5					
Silver (Ag)			0.05-0.5					
Uranium (U)				2.5				
Zinc (Zn)	0.5	0.50	0.2-1.0	0.5		0.5	0.75	1.0
pH	6.0-9.5	6.5-9.5	6.5-8.5 6.5-10	6.0-9.5	5.5-9.0	6.0	5.5	5.0

[1] Daily concentration limit is double the monthly mean value.
[2] The sum of individual concentrations measured for copper, nickel, lead and zinc cannot exceed 1.0 mg/L.
[3] Lower limits are for new mills, higher limits are for older mills. Limits are guidelines as opposed to regulations.
[4] Maximum grab sample concentration limit is double the monthly mean value.
[5] Metal Mining Liquid Effluent Regulations are used by Manitoba, Nova Scotia and New Brunswick. MMLER are a starting point for effluent requirements in the Yukon and the Northwest Territories.
[6] Maximum authorized monthly arithmetic mean concentration.
[7] Maximum authorized concentration in a composite sample.
[8] Maximum authorized concentration in a grab sample.
[9] Although this parameter is not listed in the metal mining effluent limits regulation, it has recently been applied with this typical value on Certificates of Approval under the Ontario Water Resources Act, Section 24 (AQUAMIN 1996).

public in response to the publication of the Draft Regulation. One of the recommendations was to replace the current extraction test with the TCLP (St-Laurent 1996). The Draft Regulation also includes a new table of contaminants (inorganic only) and their maximum concentrations. As shown in Table I-3, the listed criteria are identical to those used in Ontario. Hence, the proposed new criteria do not include the metals copper, nickel and zinc. One final item of note from the Draft Regulation is the inclusion of "mine tailings and sludge resulting from treatment of the effluent from a tailings storage yard, where such sludge is deposited in the yard" in a list of materials which are specifically not defined as hazardous materials. If this portion of the Draft Regulation is adopted, AMD treatment sludge will be exempted from leach testing.

I.2.6 British Columbia

In the province of British Columbia, hazardous wastes are managed through the use of the *Special Waste Regulation* under the *Waste Management Act.* The leachate extraction procedure described in Schedule 4 of the regulation was promulgated in 1988. It is identical to Ontario's LEP, although it has come to be known as the Special Waste Extraction Procedure (SWEP). The leachate quality standards for inorganics include values for copper and zinc (see Table I-3). *Special Waste Regulation* amendments which are currently being drafted include recommendations for the adoption of a modified U.S. EPA TCLP (using the Bottle Extraction Vessel for both volatile and non-volatile contaminants) and for changes to the Leachate Quality Standards (Bindra 1996).

Since AMD treatment sludges are not exempted from the *Special Waste Regulation,* they must be tested for toxicity characteristic. In British Columbia each mine site has site-specific parameters specified in their waste management permit. This permit governs the on- or off-site disposal of the sludge. Most mines dispose of sludge on-site in facilities such as tailings ponds or open pits.

I.2.7 Other provinces and territories

In the province of Alberta, hazardous wastes are managed through the *Waste Control Regulation* (Government of Alberta 1996) under the *Environmental Protection and Enhancement*

Act. Schedule 1 of the *Waste Control Regulation* refers to the "Alberta User Guide for Waste Managers" (Government of Alberta 1995). This document recommends the U.S. EPA TCLP as a test method for toxic leachate wastes. The results of the leachate analysis are compared to the standards set out in Table 2 of the Schedule to the Alberta Users Guide for Waste Managers. This schedule provides an extensive list of criteria as shown by the sample provided in Table I-3.

In the province of Manitoba, hazardous wastes are governed by the *Classification Criteria for Products, Substances and Organisms Regulation* under the *Dangerous Goods Handling and Transportation Act*. Schedule B of this regulation includes the Leachate Extraction Procedure which was promulgated in 1987. It is identical to the Ontario LEP. The Classification criteria are also identical to those used in Ontario (see Table I-3). AMD treatment sludge is dealt with on a site-specific basis in Manitoba.

The remaining provinces and territories (New Brunswick, Newfoundland, Northwest Territories, Nova Scotia, Saskatchewan and Yukon) refer to the provisions of the federal TDGR which references the CGSB Leachate Extraction Procedure. The testing of AMD treatment sludge and its storage/disposal are controlled via site-specific licences or permits based on the various provincial legislation for site operation.

APPENDIX J: SUMMARY OF LEACH TEST PROTOCOLS

As used to determine non-volatile, inorganic contaminants in
samples with >0.5 % solids content.

LEACH TEST PROTOCOLS SELECTED FOR REVIEW

Extraction Procedure Toxicity Test (EP Tox), U.S. EPA Method 1310

Purpose
Standard regulatory method (1980) used to classify wastes as hazardous or non-hazardous.

Sample preparation
Multi-phase wastes (liquid/solid) allowed to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration. Sample filtered through 0.45 µm membrane filter with vacuum or pressure (up to 75 psi).
Monolithic wastes subjected to the Structural Integrity Procedure for particle size reduction.
Non-monolithic wastes cut, crushed or ground to pass through a 9.5-mm sieve.

Sample mass
100 g minimum of original sample (liquid and solid).

Leachant
Deionized water adjusted during the course of the extraction (if the pH >5.2) to pH 5.0 ± 0.2 with periodic additions of 0.5 N acetic acid (maximum addition of 4 mL of acid per gram of solid).

Liquid-to-solid ratio
16:1 at the beginning and 20:1 at the end.

Leaching vessel
Unspecified.

Method of contact
Continuous agitation (details unspecified).

Duration
24 hours; 28 hours if the final pH > 5.2 and the maximum amount of acid has not been added.

Leachate separation
Filtration with 0.45 µm membrane filter at up to 75 psi pressure or vacuum, with standing or centrifugation as above if necessary.

Comments
Test based upon a 95% municipal / 5% industrial co-disposal mismanagement scenario.

Toxicity Characteristic Leaching Procedure (TCLP), U.S. EPA Method 1311

Purpose
Standard method (1986) for evaluating hazardous waste treatment technologies.
Standard regulatory method (1990) used to classify wastes as hazardous or non-hazardous (replacement for EP Tox).

Sample preparation
Multi-phase wastes (liquid/solid) allowed to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration. Sample filtered through 0.6-0.8 µm borosilicate glass filter with vacuum or pressure (up to 50 psi).
Wastes cut, crushed or ground to pass through a 9.5-mm sieve.
No allowance for structural integrity testing of monolithic samples.

Sample mass
100 g minimum of original sample (liquid and solid).

Leachant
Two choices of acetic acid solution, pH 4.93 ± 0.05 (buffered) or pH 2.88 ± 0.05, depending on the alkalinity of the solid phase of the waste as determined by a preliminary evaluation.

Liquid-to-solid ratio
20:1

Leaching vessel
Bottle Extraction Vessel: 2-liter plastic or borosilicate glass bottles for inorganics.

Method of contact
End-over-end agitation at 30 ± 2 rpm.

Duration
18 ± 2 hours

Leachate separation
Filtration with 0.6 to 0.8 µm borosilicate glass fibre filter at up to 50 psi pressure or vacuum, with standing or centrifugation as above if necessary.

Comments
Based on the same assumptions as EP Tox (Method 1310). Equipment changes and specifications were made to improve reproducibility and reduce contamination.

Leachate Extraction Procedure (LEP), Ontario

Purpose
Standard regulatory method (1985) used to classify wastes as hazardous or non-hazardous.

Sample preparation
Liquid and solid phases separated by first agitating the sample and then pouring a representative aliquot into the filtration unit. Sample filtered through 0.45 µm membrane filter with pressure (up to 71 psi). For coarse grained solids, vacuum filtration may be used.
Monolithic wastes subjected to the Structural Integrity Procedure for particle size reduction.
Non-monolithic wastes cut, crushed or ground to pass through a 9.5-mm sieve.

Sample mass
Equivalent of 50 g dry weight of de-watered undried material.

Leachant
Reagent water (Type IV, ASTM Specification D-1193) adjusted during the course of the extraction (if the pH >5.2) to pH 5.0 ± 0.2 with periodic additions of 0.5 N acetic acid (maximum addition of 4 mL of acid per gram of solid).

Liquid-to-solid ratio
16:1 at the beginning and 20:1 at the end.

Leaching vessel
1250-mL wide mouth, plastic or glass cylindrical bottle.

Method of contact
End-over-end agitation at 10 rpm.

Duration
24 hours

Leachate separation
Filtration with 0.45 µm filter at up to 71 psi (5 kg/cm^2) pressure. For leachates containing very fine grained particles, it may be necessary to centrifuge the suspension at high speed before filtration.

Comments
Based on the same assumptions as EP Tox (Method 1310). Only minor changes made to EP Tox in developing test.

CGSB Leachate Extraction Procedure (CGSB LEP), Canadian General Standards Board

Purpose
Provisional standard regulatory method (1987) used to classify wastes as hazardous or non-hazardous.

Sample preparation
Liquid and solid phases separated by first agitating the sample and then pouring a representative aliquot into the filtration unit. Sample filtered through 0.45 µm membrane filter with pressure (up to 71 psi). For coarse grained solids, vacuum filtration may be used.
Monolithic wastes subjected to the Structural Integrity Procedure for particle size reduction. Non-monolithic wastes cut, crushed or ground to pass through a 9.5-mm sieve.

Sample mass
Equivalent of 50 g dry weight of de-watered undried material.

Leachant
Reagent water (Type IV, ASTM Specification D-1193) adjusted during the course of the extraction (if the pH >5.2) to pH 5.0 ± 0.2 with periodic additions of 0.5 N acetic acid (maximum addition of 4 mL of acid per gram of solid).

Liquid-to-solid ratio
16:1 at the beginning and 20:1 at the end.

Leaching vessel
1250-mL wide mouth, plastic or glass cylindrical bottle.

Method of contact
End-over-end agitation at 10 rpm.

Duration
24 hours

Leachate separation
Filtration with 0.45 µm filter at up to 71 psi (500 kPa) pressure. For leachates containing very fine grained particles, it may be necessary to centrifuge the suspension at high speed before filtration.

Comments
Differs slightly from the Ontario LEP (one decision point regarding volume adjustment occurs at pH 4.8 in the CGSB LEP and at pH 5 in the Ontario LEP).

Special Waste Extraction Procedure (SWEP), British Columbia

Purpose
Standard regulatory method (1988) used to classify wastes as hazardous or non-hazardous.

Sample preparation
Liquid and solid phases separated by first agitating the sample and then pouring a representative aliquot into the filtration unit. Sample filtered through 0.45 µm membrane filter with pressure (up to 71 psi). For coarse grained solids, vacuum filtration may be used.
Monolithic wastes subjected to the Structural Integrity Procedure for particle size reduction.
Non-monolithic wastes cut, crushed or ground to pass through a 9.5-mm sieve.

Sample mass
Equivalent of 50 g dry weight of de-watered undried material.

Leachant
Reagent water (Type IV, ASTM Specification D-1193) adjusted during the course of the extraction (if the pH >5.2) to pH 5.0 ± 0.2 with periodic additions of 0.5 N acetic acid (maximum addition of 4 mL of acid per gram of solid).

Liquid-to-solid ratio
16:1 at the beginning and 20:1 at the end.

Leaching vessel
1250-mL wide mouth, plastic or glass cylindrical bottle.

Method of contact
End-over-end agitation at 10 rpm.

Duration
24 hours

Leachate separation
Filtration with 0.45 µm filter at up to 71 psi (5 kg/cm^2) pressure. For leachates containing very fine grained particles, it may be necessary to centrifuge the suspension at high speed before filtration.

Comments
Identical to Ontario LEP.

Leachate Extraction Procedure (LEP), Manitoba

Purpose
Standard regulatory method (1987) used to classify wastes as hazardous or non-hazardous.

Sample preparation
Liquid and solid phases separated by first agitating the sample and then pouring a representative aliquot into the filtration unit. Sample filtered through 0.45 μm membrane filter with pressure (up to 71 psi). For coarse grained solids, vacuum filtration may be used.
Monolithic wastes subjected to the Structural Integrity Procedure for particle size reduction.
Non-monolithic wastes cut, crushed or ground to pass through a 9.5-mm sieve.

Sample mass
Equivalent of 50 g dry weight of de-watered undried material.

Leachant
Reagent water (Type IV, ASTM Specification D-1193) adjusted during the course of the extraction (if the pH >5.2) to pH 5.0 ± 0.2 with periodic additions of 0.5 N acetic acid (maximum addition of 4 mL of acid per gram of solid).

Liquid-to-solid ratio
16:1 at the beginning and 20:1 at the end.

Leaching vessel
1250-mL wide mouth, plastic or glass cylindrical bottle.

Method of contact
End-over-end agitation at 10 rpm.

Duration
24 hours

Leachate separation
Filtration with 0.45 μm filter at up to 71 psi (5 kg/cm^2) pressure. For leachates containing very fine grained particles, it may be necessary to centrifuge the suspension at high speed before filtration.

Comments
Identical to Ontario LEP.

Procedure for Assessing the Characteristics of Solid Waste and Pumpable Sludges, Québec

Purpose
Standard regulatory method (1985) used to classify wastes as hazardous or non-hazardous.

Sample preparation
No phase separation for multi-phase samples.
Dry samples pulverized, using a mortar and pestle, to pass through a 9.5-mm sieve. If all or part of sample is too hard to be pulverized in this manner, it is tested as is or combined with the portion that has been crushed and sieved, respectively.

Sample mass
Equivalent of 5-100 g dry weight for sample with 0.5-10% solids (1 L of sample is leached).
Equivalent of 100 g dry weight for sample with >10% solids.

Leachant
For 0.5-10% suspension, 1 mL buffered acetic acid (pH 4.5 ± 0.1) per gram suspended solids added to 1 L of sample; the liquid to solid ratio may be slightly different from 10:1.
For >10% suspension, 100 mL buffered acetic acid (pH 4.5 ± 0.1) added to a 1-L mixture of sample plus distilled water (the volume of sample required to provide 100 g dry weight equivalent is made up to 1 L with distilled water).
For dry samples, 100 mL buffered acetic acid (pH 4.5 ± 0.1) added to 900 mL distilled water.

Liquid-to-solid ratio
10:1

Leaching vessel
Wide mouth, glass container with a minimal capacity of 1 L.

Method of contact
End-over-end agitation at 5 to 30 rpm.

Duration
24 hours

Leachate separation
Decantation for 30 minutes. Filtration with 0.45 µm filter under pressure or in a vacuum if required. Centrifuge or prefilter if needed to facilitate the final filtration.

Comments
Development based on EP Tox (Method 1310).

Waste Extraction Test (WET), California

Purpose
Standard regulatory method (1985) used to classify wastes as hazardous or non-hazardous.

Sample preparation
If waste is non-filterable and non-millable sludge, sample is used as received.
If a filterable mixture of liquid and solids (>0.5% solids), phase separation by filtration through 0.45 µm membrane filter. Once separated solids milled to pass through a 2.0-mm sieve.
If a millable solid, waste milled to pass through a 2.0-mm sieve.

Sample mass
50 g (may be <50 g if solids separated by filtration).

Leachant
0.2 M sodium citrate at pH 5.0 ± 0.1

Liquid-to-solid ratio
10:1

Leaching vessel
Polyethylene or glass container.

Method of contact
Vigorous agitation with a table shaker, overhead stirrer or rotary extractor.

Duration
48 hours

Leachate separation
Centrifugation if necessary. Filtration with 0.45 µm membrane filter under pressure or vacuum. Finer solids may require a prefiltration using a medium porosity filter.

Comments
Similar to EP Tox (Method 1310). Use of sodium citrate (different metal chelating ability) in the leachant may make test more aggressive towards some metals. Takes into account that some sludges are difficult to filter.

Synthetic Precipitation Leaching Procedure (SPLP), U.S. EPA Method 1312

Purpose

Standard test method (1988) used to assess the impact that contaminated soils and wastes may have on groundwater.

Sample preparation

Multi-phase wastes (liquid/solid) allowed to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration. Sample filtered through 0.6-0.8 µm borosilicate glass filter with vacuum or pressure (up to 50 psi).
Wastes cut, crushed or ground to pass through a 9.5-mm sieve.
No allowance for structural integrity testing of monolithic samples.

Sample mass

100 g minimum of original sample (liquid and solid)

Leachant

Two choices of sulphuric acid/nitric acid (60/40 weight percent) mixture (pH 4.20 ± 0.05 or pH 5.00 ± 0.05) depending on the geographic source of the soil and hence, on the acidity of the local precipitation; the more severe extraction fluid is used for wastes.

Liquid-to-solid ratio

20:1

Leaching vessel

Bottle Extraction Vessel: 2-L plastic or borosilicate glass bottles for inorganics.

Method of contact

End-over-end agitation at 30 ± 2 rpm.

Duration

18 ± 2 hours

Leachate separation

Filtration with 0.6 to 0.8 µm borosilicate glass fibre filter at up to 50 psi pressure or vacuum.

Comments

A variation of TCLP (Method 1311) using simulated acid rain.

Multiple Extraction Procedure (MEP), U.S. EPA Method 1320

Purpose
Standard test method (1986) involves multiple (sequential) extractions of the waste with a synthetic acid rain solution after an initial acetic acid extraction. Determines the maximum leachate concentrations under acidic conditions.

Sample preparation
Multi-phase wastes (liquid/solid) allowed to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration. Sample filtered through 0.45 μm membrane filter with vacuum or pressure (up to 75 psi).
Monolithic wastes subjected to the Structural Integrity Procedure for particle size reduction. Non-monolithic wastes cut, crushed or ground to pass through a 9.5-mm sieve.

Sample mass
100 g minimum of original sample (liquid and solid).

Leachant
First extraction: deionized water adjusted during the couse of the extraction (if the pH >5.2) to pH of 5.0 ± 0.2 with periodic additions of 0.5 N acetic acid (maximum addition of 4 mL of acid per gram of solid). Subsequent extractions: synthetic acid rain solution (concentrated sulphuric acid/nitric acid, 60/40 weight percent, diluted to pH 3).

Liquid-to-solid ratio
16:1 at the beginning and 20:1 at the end.

Leaching vessel
Unspecified.

Method of contact
Continuous agitation (details unspecified).

Duration
First extraction: 24 hours; 28 hours if the final pH >5.2 and the maximum amount of acid has not been added. Subsequent extractions (9 or more): 24 hours per extraction.

Leachate separation
Filtration with 0.45 μm membrane filter at up to 75 psi pressure or vacuum, with standing or centrifugation as above if necessary.

Comments
Based on EP Tox (Method 1310), except that successive elutions are performed on the same sample with a synthetic acid rain after initially conducting an acetic acid elution. Designed to simulate the leaching that a waste would undergo if it were exposed to repeated events of acid precipitation in an improperly designed landfill.

Monofill Waste Extraction Procedure (MWEP), U.S. EPA

Purpose

Standard test method (1986) used to derive reasonable leachate compositions for industrial wastes subjected to monofilling in properly engineered facilities; used to determine delays in the release of hazardous constituents; or used to assess the maximum leachate concentrations achieved under mildly acidic conditions.

Sample preparation

Monolithic wastes subjected to the Structural Integrity Procedure for particle size reduction. Non-monolithic wastes crushed to pass through a 9.5-mm sieve.

Sample mass

Unspecified.

Leachant

Distilled water or site water.

Liquid-to-solid ratio

10:1

Leaching vessel

Wide mouth sample bottle.

Method of contact

End-over-end agitation.

Duration

18 hours per extraction (4 or more extractions).

Leachate separation

Settling and filtration.

Comments

Previously called the Solid Waste Leaching Procedure (SWLP). Distilled water used as the leaching medium to simulate precipitation, which would be the predominant liquid entering the fill. Successive extractions are used to infer a time dependent leaching response. However, it does not attempt to simulate field leaching. Test is designed for solid wastes and does not provide direction regarding sample preparation of multi-phase wastes.

Shake Extraction of Solid Waste with Water (D-3987), ASTM

Purpose
Standard research method (1985) used as a rapid means for obtaining an extract of a solid waste. It does not simulate site-specific leaching conditions.

Sample preparation
As received.

Sample mass
70 g

Leachant
Reagent water (Type IV, ASTM Specification D-1193).

Liquid-to-solid ratio
20:1

Leaching vessel
Round, wide mouth bottle; sample plus liquid occupies 80-90% of container.

Method of contact
End-over-end agitation at 29 rpm.

Duration
18 ± 0.25 hours

Leachate separation
Initial separation by decantation, centrifugation or filtration through coarse paper after 5 minutes of settling. Then filtration with 0.45 µm membrane filter under pressure or vacuum.

Comments
Water extraction simulates conditions where the solid waste is the dominant factor in determining the pH of the extract. Test is designed for free-flowing particulate solid wastes and does not provide direction regarding sample preparation of multi-phase wastes.

Sequential Batch Extraction of Waste with Water (D-4793), ASTM

Purpose
Standard research method (1993) used for obtaining sequential extracts of a waste. It does not simulate site-specific leaching conditions.

Sample preparation
As received. For multi-phase wastes (>5% solids) mix thoroughly to ensure that a representative sample is withdrawn.

Sample mass
100 g on a dry weight basis (for each extraction).

Leachant
Reagent water (Type IV, ASTM Specification D-1193).

Liquid-to-solid ratio
20:1

Leaching vessel
Cylindrical, wide mouth bottle; sample plus liquid occupies 95% of container.

Method of contact
End-over-end agitation at 30 ± 2 rpm.

Duration
18 ± 0.25 hours per extraction (10 extractions).

Leachate separation
Filtration with 0.45 or 0.8 µm filter under pressure.

Comments
The final pH of each of the extracts reflects the interaction of the leachant with the neutralizing capacity of the waste. Water extractions reflect conditions where the waste is the dominant factor in determining the pH of the extracts.

Single Batch Extraction Method for Wastes (D-5233), ASTM

Purpose

Standard research method (1992) used to provide an indication of leaching potential. Contaminant concentrations in the extract may be compared against set or chosen acceptance levels in some applications.

Sample preparation

Multi-phase wastes mixed thoroughly to ensure that a representative sample is withdrawn. Multi-phase sample (liquid/solid) then allowed to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration. Sample filtered through 0.6-0.8 µm borosilicate glass filter with vacuum or pressure (up to 50 psi).

For monolithic materials such as rock or slag, a coring that will fit into the extraction apparatus may be produced.

Sample mass

100 g minimum of original sample (liquid and solid).

Leachant

Two choices of acetic acid solution, pH 4.93 ± 0.05 (buffered) or pH 2.88 ± 0.05, depending on the alkalinity of the solid phase of the waste as determined by a preliminary evaluation.

Liquid-to-solid ratio

20:1

Leaching vessel

Cylindrical bottle, minimum 2-L size.

Method of contact

End-over-end agitation at 30 ± 2 rpm.

Duration

18 ± 2 hours

Leachate separation

Filtration with 0.6 to 0.8 µm glass fibre filter at up to 50 psi (345 kPa) pressure or vacuum, with standing or centrifugation as above if necessary.

Comments

Similar to TCLP (Method 1311). Recognizes that interpretation and use of the results of this test are limited by the assumptions of a single co-disposal scenario. The sample/extractor interaction is expected to correlate with the environmental conditions to which a waste may be exposed.

Sequential Batch Extraction of Waste with Acidic Extraction Fluid (D-5284), ASTM

Purpose
Standard research method (1992) using an extraction fluid based on acidic precipitation to obtain sequential extracts of a waste. It does not simulate site-specific leaching conditions.

Sample preparation
As received. For multi-phase wastes (>5% solids) mix thoroughly to ensure that a representative sample is withdrawn.

Sample mass
100 g on a dry weight basis (for each extraction).

Leachant
Sulphuric acid/nitric acid (60/40 weight percent) mixture (pH ± 0.05) depending on the pH of the acid precipitation in the geographic region in which the waste is to be disposed.

Liquid-to-solid ratio
20:1

Leaching vessel
Cylindrical, wide mouth bottle; sample plus liquid occupies 95% of container.

Method of contact
End-over-end agitation at 30 ± 2 rpm.

Duration
18 ± 0.25 hours per extraction (10 extractions).

Leachate separation
Filtration with 0.45 or 0.8 µm filter under pressure.

Comments
Similar to U.S. EPA Method 1312. The final pH of each of the extracts reflects the interaction of the leachant with the neutralizing capacity of the waste.