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WASTEWATER TECHNOLOGY CENTRE
REP - 0002

SEDIMENT WASHING TREATABILITY STUDY ON WELLAND RIVER SEDIMENT

GREAT LAKES CLEAN-UP FUND

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EXECUTIVE SUMMARY

ALTECH Environmental Consulting Ltd. conducted a treatability study for metal, oil and grease, and total organic carbon (TOC) removal from Welland River Sediment, collected from the river adjacent to the Atlas-Mansfield outfall. The main effort of the project was aimed at the reduction of the metal content within the sediment. Further, the sediment employed during the treatability study was, nominally, the sub-75μm sediment fraction from the Acres process. The work was completed for the Contaminated Sediments Treatment Technology Program, (Great Lakes Cleanup Fund) under contract to the Wastewater Technology Centre (WTC). Funding for the provided by Environment Canada's Great Lakes Environment Office.

It was found that, for the **ALTECH** treatability studies, the most efficient method of metal removal was through the combined use of an attrition scrubber and a flotation cell. The sediment was subjected to a number of differing trials which included:

- sediment dilution with water
- chemical additions for metal removal from soil and collection
- pH adjustment
- chemical additions for metal flotation

Analysis of all results for metals was performed on the Atomic Absorption Spectrometer at Brock University. Analysis was performed by Dr. U. Brand, professor and geochemist. Analysis for TOC and Oil and Grease was performed by Technitrol Expertise Inc. of Toronto.

Through characterization, the sediment was found to contain highly insoluble metal particles entrained in a tight clay host. This is a difficult sediment package to treat, and physical separation enhanced by chemical additions is considered to be one of the few options available.

Lead and zinc concentrations in the sediment were brought down under the Severe Effects Level for the Provincial Sediment Quality Guidelines (PSQG). Zinc was removed from the sediment from a pre-treatment level of 731.3 ppm to 461.2 ppm remaining after the washing process; the Severe Effects Level for Zinc is 820 ppm. Lead was removed from the sediment from a pre-treatment level of 267.3 ppm to 169.8 ppm remaining after the washing process; the Severe Effects Level for Lead is 250 ppm. The PSQG's were implemented because the most recent information from the MOE (June 1992), indicates that these levels are the starting blocks for all sediment, including open water disposal of dredged sediments.

Significantly, analytical results indicate that metals above atomic number 30 were removed from the sediment in larger quantities than those metals with below atomic number 30. The reasons for this pattern are not known at the time of publication of this report.

Metals below atomic number 30 showed significantly less removal than those above atomic number 30. Because some clay sediment was removed along with the metals from the flotation cell, the end result was a concentration and higher value in ppm for the metals below atomic number 30 left in the sediment which did not leave the flotation cell.

Removal efficiencies were noted for both TOC and oil and grease. The TOC limit for the Provincial Sediment Quality Guidelines appears to be attainable, however, the oil and grease limit of 0.15%, carried over from the Open Water Disposal Guidelines, would require further system optimization.

1.0 INTRODUCTION

ALTECH Environmental Consulting Ltd. submitted a proposal for bench scale demonstration of soil washing technology. The proposal was entitled "RFP - 0002, Bench Scale Studies, Welland River Sediment", and was dated July 8, 1991. The proposal was submitted to the Contaminated Sediments Treatment Technology Program, under the Great Lakes Cleanup Fund for work performed under contract to the Wastewater Technology Centre (WTC), in Burlington, Ontario. Funding for the treatability study were provided by Environment Canada's Great Lakes Environment Office.

The purpose of the RFP was two-fold:

- to obtain a broad information base upon which the WTC could base rehabilitation decisions; and,
- to assist technology developers and vendors in the optimization of promising technology.

The technology chosen for bench scale demonstration was to be site specific; the **ALTECH** proposal was determined to be best suited to "extraction -- organic and inorganic", and was geared towards the clean-up of the Welland River sediment. The study area is outlined in Figure #1.

The Welland River sediment in question had been heavily contaminated with oily and metal discharges from nearby Atlas-Mansfield Ltd. (Atlas), and other industrial discharges. Previous studies have been completed by M.D. Dickman, J.R. Yang and I.D. Brindle of Brock University. These studies compared the upstream concentrations of metals and the associated impact on the river's aquatic plant life and benthic invertebrate zonation pattern. Analytical data from Dickman *et al.* pertaining to the concentrations of metals upstream from Atlas are implemented as background concentrations of metal in the river sediment. This represents metal concentrations found in Welland River sediment which has been unaffected by the industrial discharges at this outfall. This data is included as Table #1.

Promising technologies for clean-up of the metal and oil contaminated sediment were awarded bench scale trial contracts. The **ALTECH** soil washing system design was chosen as one such promising technology and was granted a contract from the WTC for research into it's possibilities. The contract date was September 26, 1991, and work was authorized to proceed on September 30, 1991. The contract was amended on November 25, 1991. The contract reference number is **1-6015**.

The WTC contract is site specific. The sediment which was to be tested in the **ALTECH** soil washing process was provided by the WTC, and comes from the Welland River. Although this bench scale process is directly aimed at the removal of metals and oil

Figure #1

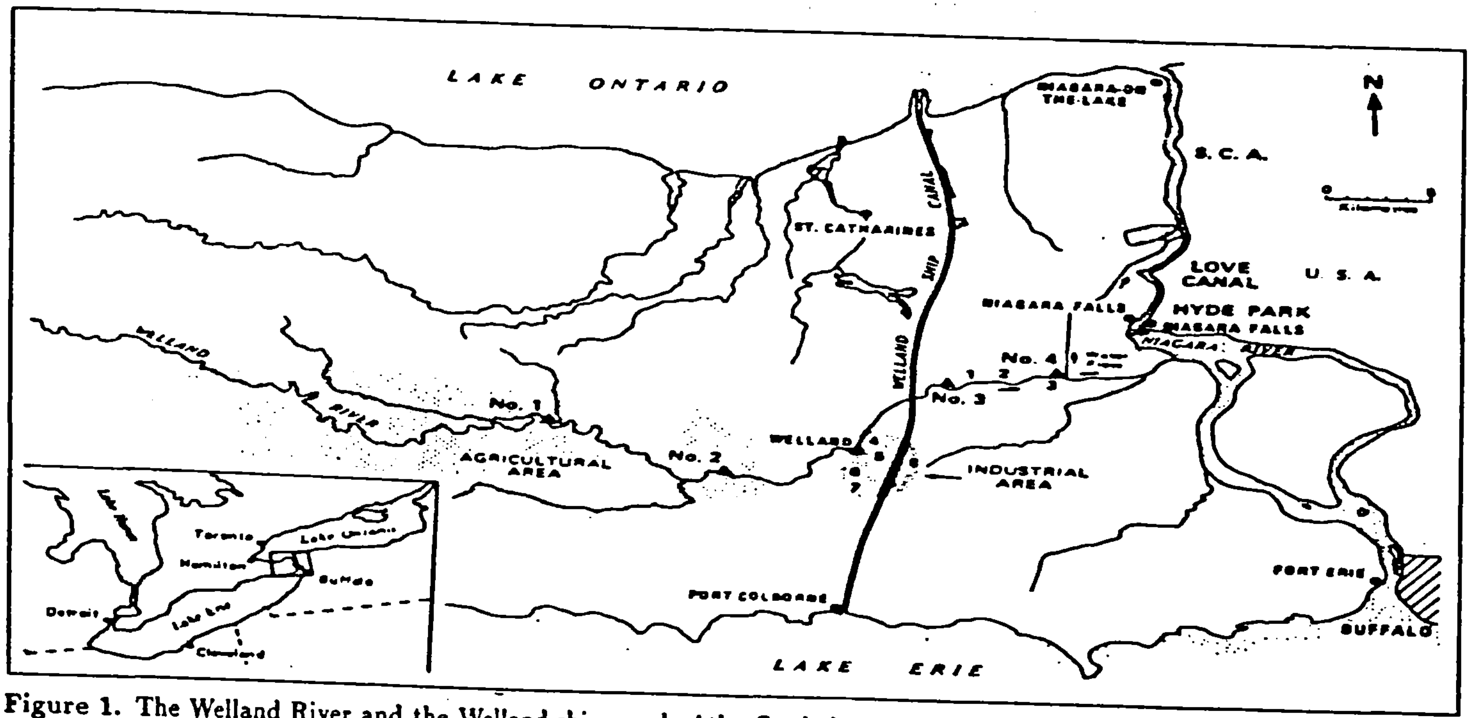


Figure 1. The Welland River and the Welland ship canal. Atlas Steels is represented by the solid circle at the "D" in Welland along the river. Numbers represent sites studied by Lan *et al.* (1990). The inset in the left hand corner represents the location of the study area in the context of the lower Great Lakes. The lower river flow direction arrows are confusing unless one has read Chapman and Putman "Actually the Welland River no longer runs into the Niagara River, but out of it, as the last four miles serves as the intake of the Chippawa-Queenston power canal, and as a final indignity (the river) has been forced to crawl beneath the Welland Canal through a siphon" (Chapman and Putnam 1966).

TABLE #1

Background Sediment Values
-- from Dickman et. al.

Parameter Tested	Upper Welland	Lyons Creek	Thomton (1983)
Nickel	29.5	44.9	2-100
Chromium	30.0	33.7	n/a
Lead	69	72	10-150
Zinc	127	111	25-200
Copper	27.6	28.6	2-60
Cobalt	n/a	n/a	n/a

Note: all values in ppm

Dickman, M.D., et al.
Impacts of Heavy Metals on Higher Aquatic Plant,
Diatom and Benthic Invertebrate Communities
in the Niagara River;
WATER POLLUTION RESEARCH JOURNAL OF CANADA
Volume 25, no. 2, 1990

from the Welland River sediment, the soil washing process is applicable to many different contaminants and many different sediment and soil types.

Of particular concern were contaminants which were present in the Welland River sediment and exceeded the Provincial Sediment Quality Guidelines'(P.S.Q.G) Severe Effect Level (S.E.L). These contaminants were found in 'reef-like' deposits in the Welland River. The reef deposits totalled 5100 m^3 of sediment. Nonetheless, a more extensive area of 30,000 m^3 was delineated as having been contaminated and will need removal and remediation. The contaminants which exceed the P.S.Q.G are metals in particular and include copper, chromium, iron, lead, manganese, nickel and zinc. Total organic carbon and oil and grease were also of concern. Sediment characterization reveals both parameters to be in excess of the SEL for the PSQG for the sediment fraction ($<75\mu m$) being used in the **ALTECH** bench scale trials. The sediment is non-hazardous waste as outlined by Regulation 309 leachate testing.

The bench scale testing was assembled and performed in the Environmental Geochemistry Laboratory and Brock University. Dr. U. Brand, Professor of Geochemistry at Brock University, was a member of the **ALTECH** team and acted as analytical specialist and supervisor of laboratory processes. Equipment used for the soil washing bench scale tests included an attrition scrubber, and a flotation cell. Chemicals were added to enhance different components of the process, as well as alterations in the pH of the system. Results were analyzed using the Scanning Electron Microscope (SEM), Energy Dispersive X-Ray Analyzer (EDAX), and Flame and Furnace Atomic Absorption Spectrometer (AA) for metals, solvent extraction for oil and grease, TOC analyzer for liquid TOC, and TOC solids was completed by the removal of the inorganic carbon through acid digestion with organic carbon calculated by furnace combustion.

All processes, methodologies and results are discussed and documented in detail in the body of the report.

2.0 TECHNOLOGY DESCRIPTION

The bench scale treatability study addressed the contamination problem in several innovative ways; these were outlined in the original proposal. As the bench scale testing progressed, priority areas were identified and optimization was carried through on these areas.

A flow diagram for the bench scale system design implemented in the WTC study is included as Figure #2. This outlines, in box and line format, the steps which were taken during the bench scale treatability study. It includes only the implementation of the equipment, the chemicals, and the analytical procedure. It does not describe in detail the theoretical work or calculation stages for optimization. Nor does it describe the data compilation and review. As a whole, these latter two steps were a major portion of the bench scale work.

Further, the treatability study was run in a 'batch' format, and was not continuous.

2.1 Sediment Collection and Handling

Sediment samples were collected from the Welland River adjacent to the Atlas-Mansfield outfall. These samples of sediment were given to **ALTECH** in 30 litre white pails, and consisted of a mixture of sediment, contaminants entrained in the sediment, a large component of water, and a variety of wood and uprooted aquatic plant life. The samples had a noticeable organic odour although it was not easily identifiable. As well, there was a slight oily film on the water layer in the white pails.

A second group of 30 litre white pails containing sediment was delivered to **ALTECH** at a later date. This sediment had been subjected to the Acres separation and classification technique, which included a series of filters and mesh screens, as well as centrifuging at one point. The Acres process was studied and is outlined in a diagram received from the WTC, here noted as Figure #3. The majority of the soil washing process was carried out on the Acres processed soil which was all considered nominally to be <75μ, and had been dewatered previously by centrifuge. Flocculants and dewatering aids had been previously added to the sediment.

The samples were stored at room temperature at **ALTECH** as well as in the geochemistry laboratory at Brock University. The base samples were not removed from the white pails in which they were delivered, and nothing was added or subtracted during storage.

The samples were handled in the white pails, and when being transferred to any component of the soil washing process, clean glass or plastic was implemented.

FIGURE #2

ALTECH SOIL WASHING SYSTEM DESIGN

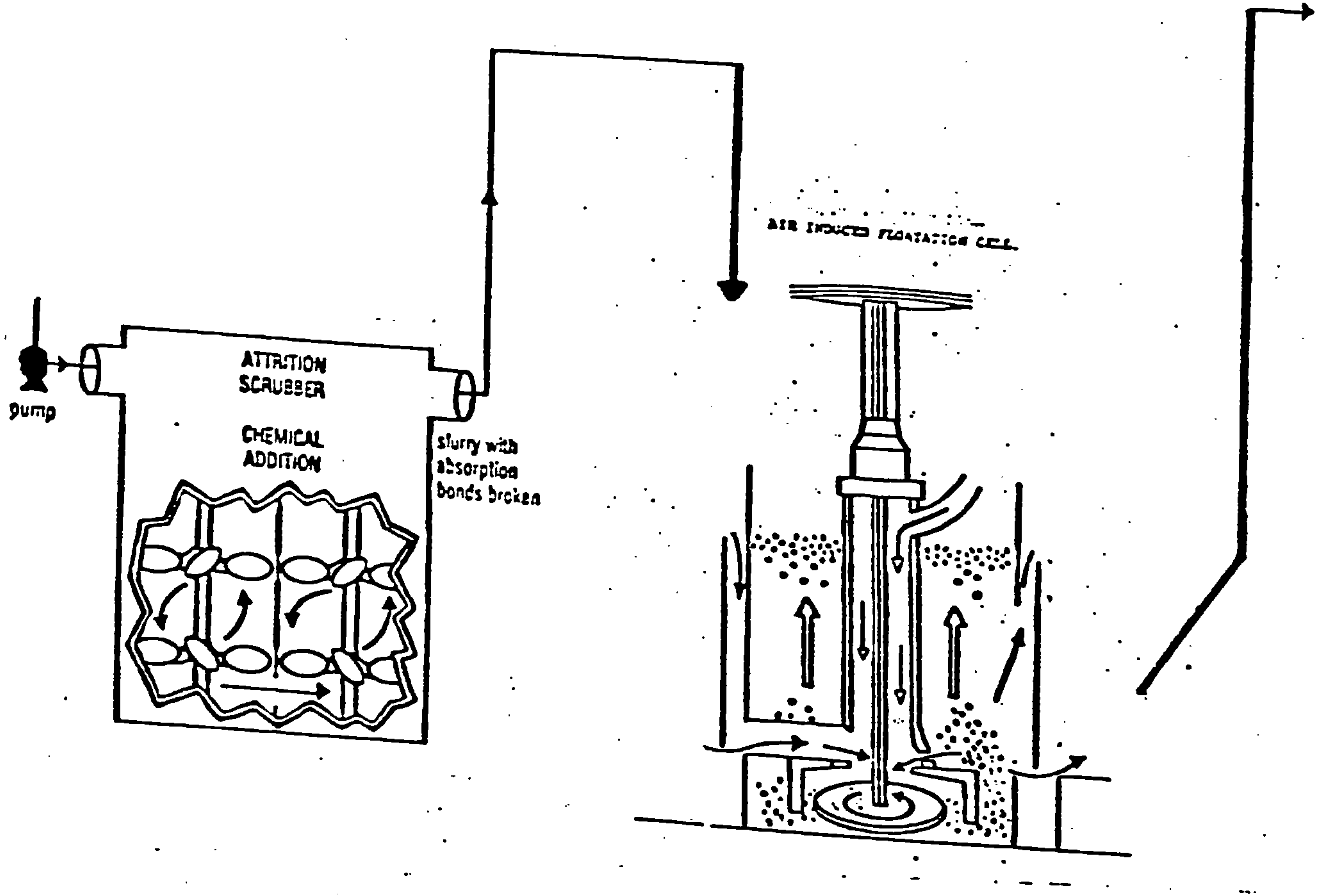


FIGURE #3

Pilot-Scale Sediment Pretreatment Facility

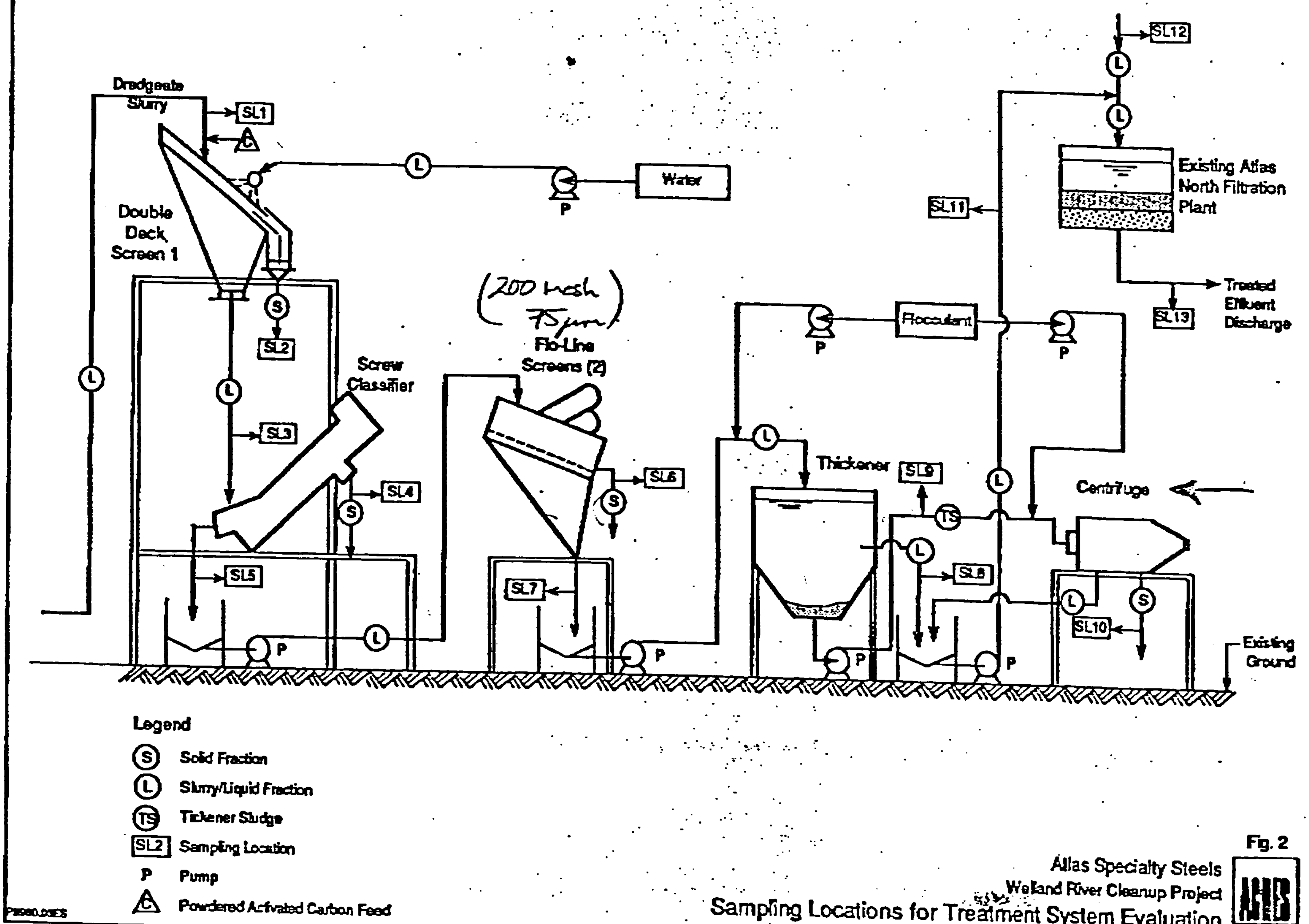


Fig. 2

Atlas Specialty Steels
Welland River Cleanup Project
Sampling Locations for Treatment System Evaluation

2.2 Attrition Scrubber

The attrition scrubber was the primary contaminant liberation mechanism employed. The purpose of the attrition scrubber was to impart shear energy into the sediment package, thereby liberating the contaminants bound to the sediment particles.

Contamination is bound to individual soil or sediment particles by adsorption. Adsorption is a weak attractive force, and is usually on the order of Van der Waals bonding, or if water is present, it may be hydrogen bonding. Either way, the metal contamination (for example) has adhered to the surface of the soil particle, and has not been incorporated into the matrix of the mineral. Energy may be used, in favourable circumstances, to release the contamination from the sediment particle.

Chemicals were added to impart a favourable environment for metal/clay separation in the sediment. These chemicals included surfactants, acids, and frothers and assisted in maintaining the separation which may be achieved through the shearing of the soil.

The shearing of the soil is carried out by very active high energy mixing inside the attrition scrubber. This is accomplished by three mixing propellers which are attached to a central axis and then lowered into the attrition scrubber bucket and rotated at very high speeds on the order of 500 r.p.m. The intensity of the shearing action can be altered by tightening or loosening the drive belt on the attrition scrubber. Percent solids in the attrition scrubber can also be altered to give more, or less, shearing action to the sediment package. The attrition scrubber works best at high percent solids, because the shearing action is the force of particles and agglomerations abrading one another, as opposed to the a fluid shear over particles and/or agglomeration surfaces. Further, chemical additions for pH adjustments can be made at the attrition scrubbing stage, although experience has shown that alterations of pH are most efficient and useful in the subsequent stage of froth flotation. The fourth variable for attrition scrubbing is residence time; this is altered by leaving the soil sample in the attrition scrubber for longer or shorter periods of time.

2.3 Flotation Cell

The flotation cell is the primary separation mechanism. The flotation cell will separate the free contamination from the sediment package. The flotation cell uses a number of influencing factors and agents to assist the efficient separation of contamination, in this case metals, from the sediment. These may include the following:

- *Air*; air was introduced at the base of the flotation cell through an Aer-o-flo DP 75 diffuser. The diffuser is entirely plastic in construction, and the air source entered through a plastic pipe in the side of the cell. The air intensity could be varied up to 12 CFM.

- *Water*; water will reduce or increase the percent solids of the sediment slurry being treated with the optimum range between 2-15% solids. As well, additional water assists in the intermixing of the sediment, the released metals, and the chemical promoter.

- *Collector*; the collector is a chemical, which when added to the flotation cell, will attach to the metal of choice and allow that metal to be floated by air bubbles. The collectors are specific to types of sediment and contaminants, and there is much trial involved in this stage. Collector molecules generally have one end which is attracted to the metal, and another free end which will attach to air bubbles.

- *pH*; collector additions made to the flotation cell will work better at certain pH levels. pH may be altered with common acids and bases to maximize the flotation of the desired contaminants.

- *Frother*; a frother may be used to float the metal particulate within the sediment slurry. The frother is an aliphatic polyether which will enhance the flotation properties of the collector. A frother will enhance the air bubble content within a float cell, giving any released contamination (associated with collector) a vehicle of transport to the surface of the liquid layer.

The flotation cell was implemented as the main separation device in the bench scale treatability tests. The flotation cell operates on the basis of specific gravity differences. Air is introduced into the flotation cell, lighter released contamination will rise to be removed from the overflow at the top of the flotation cell. Chemical additions are used to enhance the susceptibility of the contaminant to attach to an air bubble, and, consequently rise.

All sample was transferred directly from the attrition scrubber into the flotation cell. The flotation cell works best at considerably lower per cent solids than the attrition scrubber, and several trials were necessary to determine the optimum concentration of water. Concentrations generally went from 60% solids in the attrition scrubber to 2-15% solids in the flotation cell.

The flotation cell was designed so that froth which was produced inside the cell could be collected from different heights. Exit points for the froth could be opened along the vertical extent of the flotation cell, where the froth was withdrawn into channels along the exterior of the cell. The channels were sloped so that collection of the froth from one end was possible.

2.4 Chemical Additions

The investigative direction followed in the development of a chemical strategy was the concept of collecting and removing a desired metal from the surrounding "undesired" sediment. This technology has been applied successfully in the mining industry. The field of endeavour for developing the correct chemical additive to remove a desired mineral from the surrounding gangue (undesirable rock content, in this case sediment) is known as ore dressing metallurgy.

While certain general rules apply, this area is as much an art as it is a science. The general classes of chemicals used are known as collectors, modifiers, and dispersants. Collectors are generally heteropolar, aliphatic organic molecules that have one charged "polar", "hydrophillic" (water loving) end, and one uncharged "non-polar", hydrophobic (water hating) end. A charged particle whose bond with the soil particle has been broken in the attrition scrubber, will be chemically attacked by the collector in the foregoing manner. The particle will be coated with a spherical shell of collector molecules that attach to the particle. The polar ends of the collector molecules bond with the contaminant. The non-polar ends stick out into the water. The result is that the sphere becomes a hydrophobic particle, which rises to the surface of the water layer in contact with air in a float cell. Furthermore, these hydrophobic spheres will attach to any air bubbles in the matrix, and will rise to the surface.

A dispersant is a surface active agent added to a suspending medium to promote uniform and maximum separation of extremely fine solid particles, often of colloidal size. True dispersing agents are polymeric electrolytes that neutralize weak electrostatic surface forces. This serves to free contaminant particles from the soil matrix materials.

A list of generic chemical facilities that are candidates for use in the soils washing experiments were developed based on the contaminant parameters and their concentrations in the sediments as well as through interviews with professionals in the field of ore dressing metallurgy. This list is outlined in the table below. The materials that were selected for use included items B, C, D, E, and F; although listed in the Table, category A was not used. These were selected based on a professional chemical review and matching of the contaminant with the chemical additive to delineate those chemical additives that are thought to work the best.

TABLE 2

SELECTION OF CHEMICAL ADDITIVES TO BE EXPERIMENTED IN SOILS WASHING SYSTEM

A. Non Sulphide Collectors
eg. Fatty Amines
Aliphatic Carboxylic Acids

B. Aliphatic Sulphonated Compounds

C. Anionic Collectors
-For Metal Carbonate and Sulphate Salts

D. Aliphatic Polyethers

E. Detergent

F. pH Modifiers
eg. Lime
Acid

3.0 SEDIMENT CHARACTERIZATION

The following characterization describes the analysis of the sediment by AA at Brock University. Following, separate sections will describe the physical characteristics as observed and documented with SEM and EDAX analysis.

The sediment was delivered to **ALTECH** in 2 separate and distinct groups. They were defined as:

- Original Sediment: sediment which was taken directly from the Welland River to **ALTECH**, and;
- Processed Sediment: sediment which was given to **ALTECH** after processing through the Acres procedure.

This Acres procedure was meant to separate out all >75μm particles and leave **ALTECH** the <75μm material.

The work performed by **ALTECH** was primarily on the processed sediment.

3.1 Original Sediment Characterization

The sediment was delivered to **ALTECH** directly, and as a result, contained some portion of wood and aquatic plants, as well as the sediment. The river sediment was treated through the Acres process, which yielded a number of different size fractions. One of the size fractions was selected by the **WTC** and given to **ALTECH** for treatability studies.

Characterization of the original sediment was performed on the Flame Atomic Absorption Spectrometer (A.A) at Brock University. The analytical work was completed by Dr. U. Brand, Professor of Geochemistry. The results were also tabulated and presented to **ALTECH** by Dr. U. Brand. The results are summarized in Table #3, and are included in original form in Appendix #1. All values are in parts per million.

The elemental analysis for metals on the A.A included:

- Copper (Cu)
- Nickel (Ni)
- Cobalt (Co)
- Chromium (Cr)
- Silver (Ag)
- Aluminium (Al)
- Zinc (Zn)

- Manganese (Mn)
- Iron (Fe)
- Magnesium (Mg)
- Selenium (Se)
- Arsenic (As)
- Cadmium (Cd)
- Lead (Pb)

The original sediment characterization was compared to the draft report of May 1991, from the Water Resources Branch, MOE, entitled *The Provincial Sediment Quality Guidelines*; the severe effect level (SEL) Guidelines for Metals and Nutrients was implemented. The guidelines outline several metals which are of concern if they are present in the sediment. These metals are:

- Arsenic (As)
- Cadmium (Cd)
- Chromium (Cr)
- Copper (Cu)
- Iron (Fe)
- Lead (Pb)
- Manganese (Mn)
- Mercury (Hg)
- Nickel (Ni)
- Zinc (Zn)

The original RFP outlined several metals which had been previously analyzed to be above the SEL of the guidelines, and included Cr, Cu, Fe, Pb, Mn, Ni, and Zn. From the Brock University AA analysis on the original sediment, Cu, Ni, Cr, Zn, and Fe were confirmed to exceed the SEL for the sediment guidelines. Pb and Mn were not above the SEL, although quantities were, in both cases, significant and approached the SEL. For this reason, normal variations from sample to sample would indicate that all metals of concern may be at, approaching, or above the SEL.

From the original sediment characterization in Table #3, it can be seen that Al and Fe concentrations are relatively high when compared to other metals. The original characterization shows the highest concentration of any metal in the soil is Al at 69,670.0 ppm. Second only to Al in concentration in the soil is Fe at 60,152.2 ppm. A possible explanation for the high Al concentration is the composition of the clay which acts as the host to the contamination. Clay, by its very nature, is composed of building blocks which are metal centred silicates. The primary metal for clay mineral centres, by far, is Al. This clay building block is named Gibbsite. Fe and Mg substitution in clay is common, as well as inherent Fe and Mg, depending on the weathering and diagenesis patterns of the sediment. For this reason, large quantities of Fe, Mg and Al can be considered a natural component

TABLE #3

Original Sediment Characterization		PSQG SEL
Cu	263.081	110
Ni	837.466	75
Co	57.107	
Cr	780.945	110
Ag	3.612	
Al	69670.05	
Zn	829.754	820
Mn	846.349	1100
Fe	60152.284	4%
Mg	13666.537	
Se	.291	
As	21.56	33
Cd	1.68	10
Pb	158.3	250

Note: All values in ppm

of sediment where there is a component of clay. There is, however, no way within the scope of the project to ascertain the exact clay mineralogy.

Ni was analyzed at 837.4 ppm, with the SEL from the PSQG set at 75 ppm. Stainless steel contains Ni. The same holds true of Cr, analyzed at 780.9 ppm and the SEL at 110 ppm. Using common stainless steel compositions of 304 and 316, the Ni percent would be 9-11% of total metal. Therefore, with stainless steel 304, Ni equals 9% and Fe as the major component, or 72%. This puts the Fe content at 6,696 ppm in direct relation to Ni for stainless steel 304. In the sediment, Fe results were 60,152.28 ppm. For this reason, a considerable portion of the Fe may be attributable to either other alloys, or the clay itself.

In summary, then, the 'original' sediment had metal values which exceeded the SEL for the PSQG.

3.2 Processed Sediment Characterization

The processed sediment is considered to be that which has been subjected to the size fractionation of the Acres process. This size fractionation was aimed at separating all material from the sediment which was of a size greater than 75 μm. SEM analysis of the Acres soil revealed a particle of approximately 300μ in length by almost 80-90μ in width; therefore, the Acres process may not have separated exactly at 75μ.

The processed sediment was used in the bench scale treatability tests. This presented the soil washing treatability study with a difficult sediment package for contaminant removal. Prior to the removal testing with differing chemical additives, the processed sediment was analyzed to be directly comparable to any removal efficiencies which were detected from the treatability study. For this reason, the processed sediment characterization was carried out a number of different times, with a specimen of the sediment which was to be used in a specific process.

By and large, however, some trends were observed. With the exception of Mg and Zn, all metals of concern to the PSQG were of a significantly higher concentration in the characterized processed sediment than in the characterized original sediment. It must be noted that the study was not aimed at characterizing the sediment, but rather at treating it, and therefore the trends are only observed and characterization was not carried out enough times to be of statistical significance. Nonetheless, the difference between Fe in the processed sediment and the original sediment, for example, was 170,272 ppm and 60,152 ppm respectively. This represents a large, if not statistically significant, difference in the Fe concentrations between the two sediment packages.

The processed sediment characterization for the final audited treatability study can be found in Table #4, (and in Appendix #5 and #6). **ALTECH** did discover the form of the metal, and through visual SEM and EDAX work, characterized the full morphology of

TABLE #4

Sediment Characterization Final Audit Trial		PSQG SEL
Fe	170272.44	4%
Mn	2022.84	1100
Zn	731.32	820
Cu	685.347	110
Ni	1884.065	75
Cr	4967.452	110
Mg	10259.84	
Ag	6.210	
Pb	267.328	250
Cd	3.305	10
As	14.976	33
Se	0.911	
Be	1.402	
Sb	6.76	
Al	48027.60	

NOTE: all values in ppm

the contamination within the sediment. Fe, Mn, Cu, Ni, Cr, and Pb are all higher than the SEL for the PSQG. Zn, Cd and As values were not in excess of the SEL values for the PSQG, although they were close in some cases. Pb was almost exactly at equivalent values for the processed sediment characterization and the SEL value for the PSQG.

4.0 SCOPE OF WORK AND RESULTS

4.1 Scope of Work

The processed sediment was subject to a number of different separation trials. These included:

- attrition scrubbing
- attrition scrubbing with change in pH and chemical collector addition
- attrition scrubbing with drastic change in pH to highly acidic, for susceptibility of metal contamination to leaching
- attrition scrubbing with added dispersant chemicals, followed by addition of the sediment to the flotation cell where variables included:
  - further alteration in pH
  - per cent solids variation
  - chemical collector addition
  - chemical frother addition
  - air addition

Sediment samples were prepared differently for different trials. The different preparations may include:

- Water and chemical addition for sediment introduction to the attrition scrubber. Sediment slurry was added to the attrition scrubber and any chemicals were added subsequently in measured amounts. The measured amounts were calculated dependent upon the volume of slurry contained within the attrition scrubber. Water may be added to the slurry/chemical makeup in the attrition scrubber.

- Water, pH alteration, chemical addition to alter specific gravity of certain metal components and chemical addition for frothing enhancement were all employed for sediment introduction to the flotation cell. Water was added to the float cell in measured amounts dependent upon volume of slurry from the attrition scrubber, and was varied for optimization from one trial run to another. The water was added to the float cell prior to the introduction of air or the sediment slurry. Air was then introduced to the float cell which contained only water through the diffuser, thereby agitating the water and causing it to become 'active'. The slurry (subsequent to the attrition scrubbing) was then brought to the float cell in the attrition scrubber bucket. The slurry was added slowly to the 'active' water. Subsequent to the addition of the slurry to the 'active' water in the float cell, any chemicals may be added. It was in the flotation cell where the most optimization of processes for separation of metals from the clay sediment was undertaken.

An experimental matrix outlining treatability trials is as follows:

TRIAL	pH	FROTHER	COLLECTR.	SURFCTNT.	SAMPLE	ANALY.	NOTE
Alt-1rs	7	—	—	—	under and over	14 metals	elutriator trials
Alt-2rs	7	yes	yes	yes	liquid, solid and reference	11 metals	attrition scrubber only
Alt-3rs							acid leach trials; sediment chartrz.
short	1.52	—	—	—	liq./solid	11 metals	
long	3.72	—	—	—	liq./solid	11 metals	
x-long	3.83	—	—	—	liq./solid	11 metals	
Alt-4rs					-froth at beg. -base sed. -froth at end -agitation zone		-attrition scrubber and float cell -sed. chartrz.
trial 1	2.81	yes	yes	—		14 metals	
trial 2	3.0	yes	yes	—		14 metals	
trial 3	2.0	yes	yes	yes		14 metals	
trial 4	10.0	yes	yes	yes		14 metals	
Alt-6rs	5.0	yes	yes	yes	-attrition scrubber -liquid slurry -base sed. -timed samples -froth -sed. chartrz.	15 metals	final audited treatability run

All components of the bench scale soil washing treatability study were designed so that they would not contaminate the sediment under review. Because the study was primarily concentrating on metal treatability rates, the attrition scrubber bucket is coated in rubber, the attrition scrubber impeller is a plastic, and the flotation cell is constructed of wood, and coated with wax.

Attrition Scrubbing

Attrition scrubbing on its own was the first treatability test employed. Different chemicals were added to the processed sediment to examine the resulting difference in separation of metal contaminants and sediment. The first set of trials involved only the agitation of the sediment package in the attrition scrubber and chemical additions. This series of treatability tests was undertaken to determine if the attrition scrubber and chemical addition could free the metal contamination from the Welland River sediment into the liquid phase. Trials under the conditions shown in Table A were performed with samples collected and analyzed. A total of 9 trials was performed with the attrition scrubber only, and a total of 686 individual metal analysis were completed. Results were documented and are contained in analysis section of the report.

Attrition Scrubber Leachate Trials

A trial testing the leachability of the metals was undertaken. Equal amounts of soil and water by volume were added to the attrition scrubber, and mixed for 10 seconds. Subsequent to the short 10 second mixing, a 90 ml solution of 65% Nitric Acid was added to the attrition scrubber. The pH in the attrition bucket reached 1.52. After attrition scrubbing was complete for the set period of 90 minutes, the final pH was 3.72.

The test was broken down into three time periods:

- short; 10 minutes and sample retrieved
- long; 50 minutes and sample retrieved
- x-long; 90 minutes (1.5 hours) and sample retrieved

Flotation Cell Trials

Four separate trials were conducted prior to the final audit run which included a representative from the WTC as an observer. The 4 trial runs incorporated a total of 672 individual metal analysis. Conditions are shown in Table B.

The chemical parameters inside the cell were optimized so that a considerable amount of froth was always present. Once collected, the froth quickly settled into sediment and water components inside the sample containers. Analysis was performed on both the sediment and liquid portion in the sample jars.

Samples were also retrieved from the inside of the flotation cell subsequent to the flotation process. These included the liquid slurry and the bottom solids, if present. Therefore, the flotation process would yield 4 samples to be compared at the conclusion of each flotation trial run. These were compared to each other, and to the soil characterization prior to each run.

Chemicals which were added during the different trials were done so by measuring either volume or weight of the selected chemical, and adding it to the mixture within the flotation cell. Generally, the air was already entering the flotation cell so that the contents were mixed thoroughly during addition of any chemicals. The chemical additions, again, consisted of the following uses:

- For increasing the susceptibility of the contaminant to flotation;
- To increase the frothing ability of the mixture, and;
- To adjust the pH inside the cell to desired levels.

When the flotation cell was in operation, there were three distinct internal levels; 1) settled base sediment, 2) middle slurry, or agitation zone, and 3) the froth produced and rising towards the sampling ports. The three distinct layers did form inside the flotation cell, and the froth was removed for analysis during operation. Also, samples of middle slurry would be pumped out, and/or, samples of the base sediment would be retrieved. Through the use of photographs, Plate #1 shows the appearance of the flotation cell during operation. In general, there would be between 3 and 5 samples collected per trial run, which may include the sediment characterization for comparison purposes following the trial run.

Separation on a vertical gradient was used to remove the froth and any floated particulate and contamination. The sample ports were holes in the side of the flotation cell, at differing levels, which could be opened or closed as desired. The froth which was a mixture of air, water, sediment, and added chemicals, would exit the cell through these ports, and be channelled through troughs to be collected in sample containers. The sample containers consisted of new glassware and were sealed subsequent to sample collection.

Samples were stored at standard temperature and pressure in the Geochemistry Laboratory at Brock University, with selected samples being submitted for immediate analysis. Conversely, the attrition scrubber trial samples with no flotation cell were allowed to settle prior to analysis.

The 672 individual metal analysis were compared to each other in detail to determine the removal efficiencies associated with the particular tests performed in the bench scale trials. The bench scale trials were, naturally, an evolving process to determine which methodology, combination of chemicals and water, and length of time best suited the removal of the contamination from the sediment.

4.2 Analysis Methodology

Analysis was carried out in a number of ways, namely;

- Atomic Absorption Spectrometer (AA) for metals in both the solid and liquid portions of any trial sample container.
- Scanning Electron Microscope (SEM) analysis of the soil itself. This was completed to gain a full characterization of the soil type and the association

of the soil types to each other, and to the contamination. Reflected light SEM using gold and platinum group plating was used primarily for visual photomicrograph quality, as well as backscatter SEM and carbon coating for hot spot determination. These hot spots are areas which deflect the electron beam further from the normal axis and thus show on the monitor as brighter patches. The brighter patches are usually associated with metal pieces.

- Energy Dispersive X-Ray (EDAX) analysis of the carbon coated samples. The hot spots were targeted and they were evaluated for their elemental make-up. The elemental make-up was displayed in semi-quantitative graph format showing appropriately labelled peaks and troughs.
- Total Organic Carbon (TOC) was performed on a TOC analyzer for liquids; inorganic carbon removed with acid and organic carbon calculated by furnace combustion for solids.
- Oil and Grease was performed by solvent extraction.
- Photographs were taken of the sample containers which presented visual changes over time. These photographs have been preserved.

All analysis was retained in tabular format from the AA results. The SEM photomicrographs were labelled according to the sediment package and process they had undergone and are presented in 4 inch by 5 inch format. The EDAX spectral analysis are presented in graph format with the peaks and trough labelled as to the metal they represent.

Appropriate QA/QC for the laboratories is included as Appendix #2.

4.3 Work Program and Results

The following section details the treatability trials carried through with the sediment subjected to the Acres process of size fractionation, identified as 'processed sediment'.

Attrition Scrubber

Samples were retrieved from the attrition scrubber trials. Analysis was performed on the different layers in the samples; these being;

1) the floating layer where present and possible to retrieve,
2) the liquid layer,
3) a coloured sediment layer, and,
4) the base sediment.

Analysis was performed on all layers of selected samples, where possible, and is presented in Appendix #3.

Liquids were removed as an aliquot from the supernatant subsequent to the settling process. The samples were static when this was done, and had been left to settle for a period of days.

Solids were removed from both the base sediment in the sample jars, and the coloured layer if present. Analysis was performed to determine if there was any marked difference in metal concentrations between the two distinct layers. Visual observation was the means of first identification of the different layers. Results are outlined as follows:

- On Dec. 30, 1991, the logging of some samples was performed. Several different trials had been run and allowed to settle naturally to determine if there would be any separation of the metal into the liquid and/or sediment portions. What was observed was the formation of, in most cases, 4 discreet layers in each sample jar -- the base sediment, a thin layer of often different coloured sediment on the top of the base sediment, the liquid layer, and a floating layer of varying thicknesses.
- The settling of the sediment in the sample jars was not rapid, however, this was to be expected as all sediment was very fine in composition.
- The coloured layer on top of the base sediment did not appear at once, and took on the order of days to develop.
- The coloured layer was, for the most part, orange and may have been due to the oxidation of the iron in the sample.
- Some coloured layers were green, to greenish yellow.
- The liquid layers were, for the most part, only slightly coloured or clear with occasional colourings (green, yellowish green or red/orange).
- The floating layers varied greatly from sample to sample and were dependent upon the chemical additions in the attrition scrubber.

The liquid analysis presented in Appendix #3 shows that there was not an appreciable amount of the metal dissolved in the liquid in any sample, for any metal. The highest values retained were for Mg, at an average of 35 to 45 ppm. However, tap water was analyzed at 9 ppm, so this must be subtracted as a background giving an average Mg concentration of 26 to 36 ppm. Most other values for the liquid were negligible and on the order of fractions of a part per million; for example, the highest value for Zn was 0.8 ppm. From this, then, it was concluded that the metals were not highly soluble.

With reference to the liquid analysis data in Appendix #3, there was no appreciable metal entrainment in the liquid portion of the samples. The highest results were obtained with the samples 4L, 5L, and 6L. The number refers to the sample jar identification; L refers to the state of the sample, that is, liquid. The metals in highest concentration in the liquid were Fe and Al. Because both Fe and Al can be naturally found in high concentrations in clays, the origin of the minor amounts of Fe and Al in the liquid may be attributable to either dissolved metal, or colloidal clays.

The base sediment and coloured layer analysis of selected samples is presented concurrent with a characterization the sediment prior to treatment in the attrition scrubber in Appendix #3; this characterization of the sediment is outlined in this set of data as TS. The samples were allowed to settle and the liquid supernatant was analyzed separately from the solids.

Samples from the different layers were removed through a pipette. They were then prepared for analysis on the AA. The results from Appendix #3 reveal that for the most part, the top coloured layer did not hold as much metal as the base sediment. Therefore, the strong colouring of some samples may have been due to a reaction which was isolated in the top 1 to 3 cm of the sediment in the sample jar. The sediment was covered with the water layer. This suggests that although there was a coloured layer, the metals were not freed into this layer to any significant degree. The chemical added to sample #RS2 caused higher results for specific metals in the top coloured layer, denoted and analyzed as RS2TS, when compared to that left in the bottom, uncoloured sediment denoted and analyzed as C2S (and C2SD -- duplicate analysis).

Accordingly, changes in chemicals affected the concentrations to some extent, although, not significantly enough to reach conclusive determinations.

Attrition Scrubber Leachate Trials

Because the treatability testing to this point failed to show any significant amounts of metal in the liquid portion of any sample treated, a trial testing the leachability of the metals was undertaken. Data is presented in Appendix #4 and Table #5.

From the results, then, it can be observed that a measure of metal contamination has entered the liquid phase and has become soluble. When compared to the attrition scrubber treatability testing performed without the very high acidification, the values in the acidified run are, on average, 2 orders of magnitude greater. A further trend of higher solubility in the longer runs was also established. The trend between the values gained from the short acid run, and the x-long acid run, can be expressed, in general, as follows:

$$E_v = 1.375 S_v$$

TABLE #5

Test Leachability

values are contamination remaining in sediment

	Short - solid	Long - solid	X-Long - solid	PSQG SEL
Fe	236077.063	219575.763	218633.029	4%
Zn	480.866	479.803	472.839	820
Mn	2898.968	3397.758	3138.878	1100
Cu	764.563	810.398	746.630	110
Ni	1099.312	1087.167	1142.942	75
Cr	3319.175	3872.246	3899.683	110
Cd	2.731	1.739	1.190	10
Pb	274.918	264.092	271.118	250
Mg	11387.5	10446.5	11320.4	
As	23.280	24.640	20.040	33
Se	11.800	15.800	12.700	

NOTE: all values in ppm

where,

E_v = x-long acid run value

S_v = short acid run value

The significance of these results should not be overstated. Even though the acidified values were significantly greater than the non-acidified values, the absolute analytical value (in ppm) gained for metal in solution was incremental when compared to the absolute analytical value (in ppm) of metal in the sediment. In other words, although more metal became soluble with a higher acid content, even this amount of metal was small when compared with the total amount of metal contained within the sediment package.

In summary, then, it can be stated that the metal contamination within the Welland River Sediment at the Atlas-Mansfield discharge does not appear to be easily soluble. For this reason, the metal may be in the form of discreet metal particles or shavings; this assumption was confirmed through SEM and EDAX analytical performed on the sediment, as described in Section 4.4.

Flotation Cell

The optimum running of the flotation cell was determined to be at approximately 2 to 15% solids. The sample containers were filled from the sampling ports and would customarily contain 1/3 of the container volume of settled sediment. The percent solids in the sediment would, of course, be very low, nonetheless, this was a rather consistent visual determination.

Removal rate results were mixed and revealed some very interesting patterns. Because Fe, Al and Mg have a high tendency to be naturally entrained in the clay matrix when compared to the other metal contamination, an accurate removal rate for these elements is difficult to determine. They were, nevertheless, included in the full analysis along with all other metals of concern. Data is presented in Appendix #5.

Table #6 displays the important trends associated with each trial run. Patterns develop and are highlighted in the section to follow as:

- *Fe partitioning:* Under all conditions, the Fe in the sediment remaining at the base of the flotation cell was present at higher concentrations than had been found in either the sediment when the trial began, or in the sediment entrapped from the froth overflow. These values were quite consistent throughout the trial period and as a result, the data appears to point to the conclusion that the Fe is becoming concentrated in the sediment which remains in the flotation cell. This may be expected due to the massive nature of Fe when in solid particle form. Again, the Fe may come from 2 sources, either the metal contamination or the clay itself. There is no way at present within the scope of the study to determine if there is a fractionation occurring,

TABLE #6

Selected Metal Analysis

	13S Sediment Characterization	16S Base Sediment Remaining	11S Base Sediment Remaining – 2	12S Froth Overflow – 2	PSQG SEL
Fe	166252.07	213890.03	239126.1	91106.1	4%
Mn	2279.23	2778.01	5543.70	2955.01	1100
Zn	698.590	585.761	473.863	1087.495	820
Cu	742.123	830.940	985.634	960.586	110
Ni	2236.733	2731.842	2293.496	1548.700	75
Cr	6285.240	10084.120	1197.127	359.162	110
Mg	11525.7	11181.8	12779.3	12205.9	
Ag	4.871	5.027	5.088	7.738	
Pb	260.365	225.687	188.348	416.839	250
Cd	2.280	0.923	0.100	2.579	10
As	21.092	15.952	9.876	33.822	33
Se	1.085	0.769	1.647	1.984	
Be	1.451	1.539	1.197	1.651	
Sb	7.36	6.87	6.68	5.47	

Note: all values in ppm

with either the Fe in the metal or the clay becoming preferentially floated. The analytical results indicate that the Fe remaining in the sediment exceeded the Severe Effect Level (SEL) for the Provincial Sediment Quality Guidelines (PSQG) of 4%.

- *Mn, Cu, Ni, Cr, and Mg:* Generally, there were certain elements which followed the pattern outlined above for Fe; that is, higher concentrations of the element in the remaining sediment in the flotation cell than was originally present in the sediment during characterization. It must be pointed out that this was a consistent pattern, and that the sediment was characterized prior to each trial, and specific to the sediment to be used in that trial. In this way, the chance of error was greatly reduced. Elements which followed this pattern were, by and large, Mn, Cu, Ni, Cr, and Mg. The analytical results indicate that the elements remaining in the sediment which exceeded the SEL for the PSQG included Mn with SEL of 1100 ppm; Cu with SEL of 110 ppm; Ni with SEL of 75 ppm; Cr with SEL of 110 ppm. Mg does not have a stated SEL.

- *Zn and Pb:* Zn and Pb showed significant declines in the amount of each element remaining in the sediment after the bench scale treatability study. There was more Zn and Pb concentrated in the flotation overflow in ppm than was originally in the sediment. This suggests that the chemicals used may be oriented towards effectively removing either Zn or Pb, or both. If the Zn and Pb are somehow locked up in the same particle, it may be either the Zn or the Pb which is the attractive force. Either way, however, the flotation process was working as expected and desired, and brought the concentrations of both elements down significantly. The results for Zn indicated that the concentrations were brought down from near the SEL of 820, to almost half of its value; from 731 to 461 ppm. Further, the Pb values were brought from in excess of the SEL to below the SEL limits of 250; 267 to 169 ppm. Because the trials were treatability studies to infer if in fact the process was viable as a system to be optimized, these results appear encouraging. Inherent in this judgment however, is the realization that although the results for Pb and Zn are encouraging, much refinement and optimization still remains to be completed. The pattern was consistent throughout the trials.

- *Ag, Cd, As, Be, and Al:* Other elements such as Ag, Cd, As, Be, and Al were also removed from the sediment through the bench scale treatability process. Although most of these elements were not found in any significant amounts within the sediment to begin with, Al being the exception, there was an appreciable reduction if percent removal is taken into consideration. Al is the only exception to this as it is found in large quantities (40,000 to 60,000 ppm in some cases) and is likely to be part of the clay sediment itself. The reason behind this selective process phenomenon is not entirely explainable under the scope of the bench scale study, however, it becomes obvious that some elements have a greater affinity for the chemically enhanced flotation occurring within the flotation cell.

- *All Metals:* All of the above data was analyzed and only one pattern was found to exist; all elements which floated were above atomic number 30, and all those which did not float were below atomic number 30. In fact, this split was consistent throughout the sampling trials. Numerous explanations were investigated, including boiling point, melting point, electronegativity, valence arrangement, ability to form oxides, ability to form minerals, however, no other pattern became apparent. It is, however, obvious that the chemical and physical arrangement within the flotation cell is conducive to the flotation of these specific elements, and therefore, partial cleaning of the Welland River Sediment.

4.4 SEM Analysis Results

The Scanning Electron Microscope (SEM) was used to gain a visual description of the Welland River Sediment appearance in the micro scale. SEM stubs were prepared of over 20 individual soil samples. They were dried and coated using the gold and platinum sputter coater at Brock University.

SEM photomicrographs were taken of the original sediment and show masses of sediment of relatively uniform size, and on the order of several hundred micrometers in both length and width. These pictures are included in Plate #2. These agglomerations appear to be homogenous, and are estimated to consist of over 90% clay; geological charts for the analysis of thin sections under microscope were used for the estimation process. The individual particle size was found to be on the order of 4μm. This reinforces the assumption that clay seldom, if ever, exceeds this size for individual mineral grains. This is thought to be due a nucleation phenomenon associated with clays. The photomicrograph of the original sediment at 5,000 power reveals the platy structure of the minerals. The tetrahedral and octahedral layers of clay have bound individual mineral grains together, to yield an unorganized, but very tightly bound structure. It is easy, then, to see how metal contamination in the form of small insoluble particles may become entrained in the matrix of plates, and become very difficult to remove.

Metal particles were unable to be identified in the original sediment. As was shown later through EDAX analysis, the metal takes on a form which is similar to the clay through purely visual observation.

The processed sediment, that which had been subjected to the Acres classification process, was next to be analyzed under SEM. The photomicrographs of this sediment, untouched by the ALTECH process, reveal much the same form as exhibited by the raw sediment. It is presented as Plate #3. Reasonably, of course, this is to be expected because the Acres separation at 75μm would not suppress any migration of clays, being primarily on the order of 2 to 4μm in size. The photomicrograph at 200 power reveals large agglomerations of clay which had adhered together to form an amorphous mass. Relief is evident, and the agglomerations appear to be limited in size to 300 to 400μm, but are rather consistent in size.

As explained previously, subsequent to the attrition scrubbing of the sediment with different collectors, the samples were allowed to settle in the sample containers. SEM stubs were prepared of the different layers in the sample containers. These samples reveal that the attrition scrubbing appears to have released the sediment from its tightly bound state which was observed prior to agitation. Further, long cylinders and curls appeared under scanning with little or no clay now attached to them. EDAX analysis was used to identify the elemental composition of these particles. They were found to be almost pure Si, leading to the conclusion that they may be mold release agents or silicone rubber materials from Atlas Steel or from some other origin. Nevertheless, the fact remains that these appeared to be foreign objects which were released from the clay by the attrition scrubbing and chemical addition.

To be sure that the curls and cylinders observed were not due to the addition of chemicals, the chemical added was scanned under SEM by itself. This was shown to have a markedly different form than the cylinders or curls and is presented as Plate #4. The chemical agent appears to have taken on a blocky form, visually similar to naturally occurring obsidian, and appears to exhibit the same conchoidal fracture.

The SEM was used extensively subsequent to each trial run through the entire system including the attrition scrubber and flotation cell. The different layers in the flotation cell (as described previously) were sampled and a specimen from each was dried for presentation under SEM. These photomicrographs were especially revealing as they showed how the base sediment left in the flotation cell was composed of larger, more disperse particles. The froth collected out of the flotation cell sampling ports had a markedly different appearance when observed under SEM, as did the agitation zone or slurry. All of these zones are presented visually in Plate #5.

One irregularity that was noted in the SEM analysis of the different layers was the identification of a small grid-like anomaly. Under further inspection, and at 5000 power, the grid was well defined and had a mesh which appeared to be on the order of 1/4 to 1/8μm. This photomicrograph is presented as Plate #6. The size alone would lead to speculation as to its origin, however, it seemed to be associated with a longer unidentifiable plate which was covered in clay. This may be the metal contamination, however, an absolute determination was unable to be made.

SEM with EDAX Results

SEM analysis was carried out at Ortech International Inc. for the use of the energy dispersive x-ray capability (EDAX). Several samples were coated with carbon (C) at Ortech prior to analysis, and some were coated with gold (Au) at Brock University and brought to Ortech for analysis. Therefore, in some EDAX spectrum, an Au peak is present, however, this is from the coating on the SEM stub.

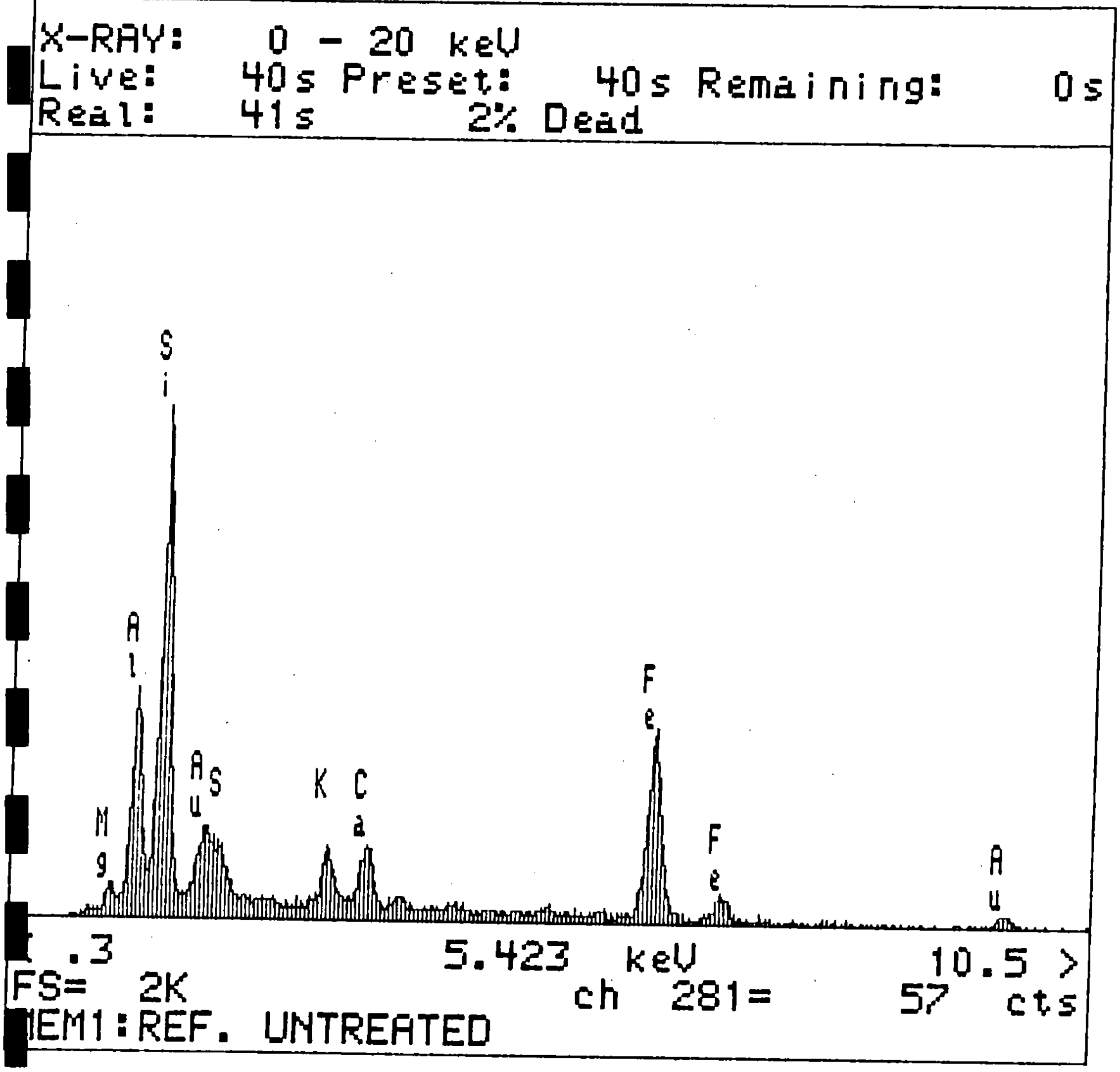
The sediment which had passed through the Acres process was analyzed as 'Reference Untreated'. The photomicrograph at Ortech essentially duplicates that initially shown at Brock; that is, large agglomerations of clay consisting of smaller mineral grains on the order of 2 to 4μm each. The photomicrograph is presented as Plate #7 with the EDAX spectrum presented as Figure #4. From this analysis, the frame reveals that the elemental make-up of the clay is 1) Si, 2) Al, and 3) Fe. This is to be expected due to the natural building block of clay, Si and Al in Gibbsite, with Fe and Mg also included in differing quantities. Mg also is shown on the EDAX spectrum, although in lower quantities than Fe. Problems arise in that the component of Fe attributable to the clay, and that attributable to the contamination, cannot be clearly defined.

With this in mind, then, samples of the sediment which had undergone attrition scrubbing were carbon coated for backscatter analysis on the SEM. This sample was from the coloured layer which was removed from the top of the base sediment in the sample jar. The backscatter radiation is deflected further relative to the densities of the material through which it passes. Accordingly, 'hot spots' are visible as they present abberations from the norm. The norm in this case would be the sediment host, clay. The photomicrograph is presented in Plate #8, with the EDAX spectrum presented as Figure #5. The 'hot spots' are highlighted in the photomicrograph as being lighter in colour than the surrounding clay. Three of these 'hot spots' are indicated with arrows and identified as particles P1, P2, and P3. The corresponding EDAX spectrums reveal that P1 is almost all Fe, with a minor amount of Mn; P2 is high in Si, Fe, and W (in this case it is likely that the high Si is a result of the interference from the surrounding clay mineralogy); and P3 is, again, almost entirely Fe.

Significantly, all of the particles show traces of Cr, Ni, Cu, and Ti. Because Atlas was a specialty steel manufacturer, these values may be due to the small amounts of each element added to the steel for purposes of strength, rust protection, etc. Nonetheless, there are some significant differences between each particle (amount of Mn and W for example) which suggests that each particle may have come from different discharges with respect to time.

It is encouraging to find that the metal particles which have undergone attrition scrubbing were visible under SEM and not completely surrounded by clay. This indicates that the metal is being liberated to some extent through the addition of appropriate chemicals and the introduction of shearing energy.

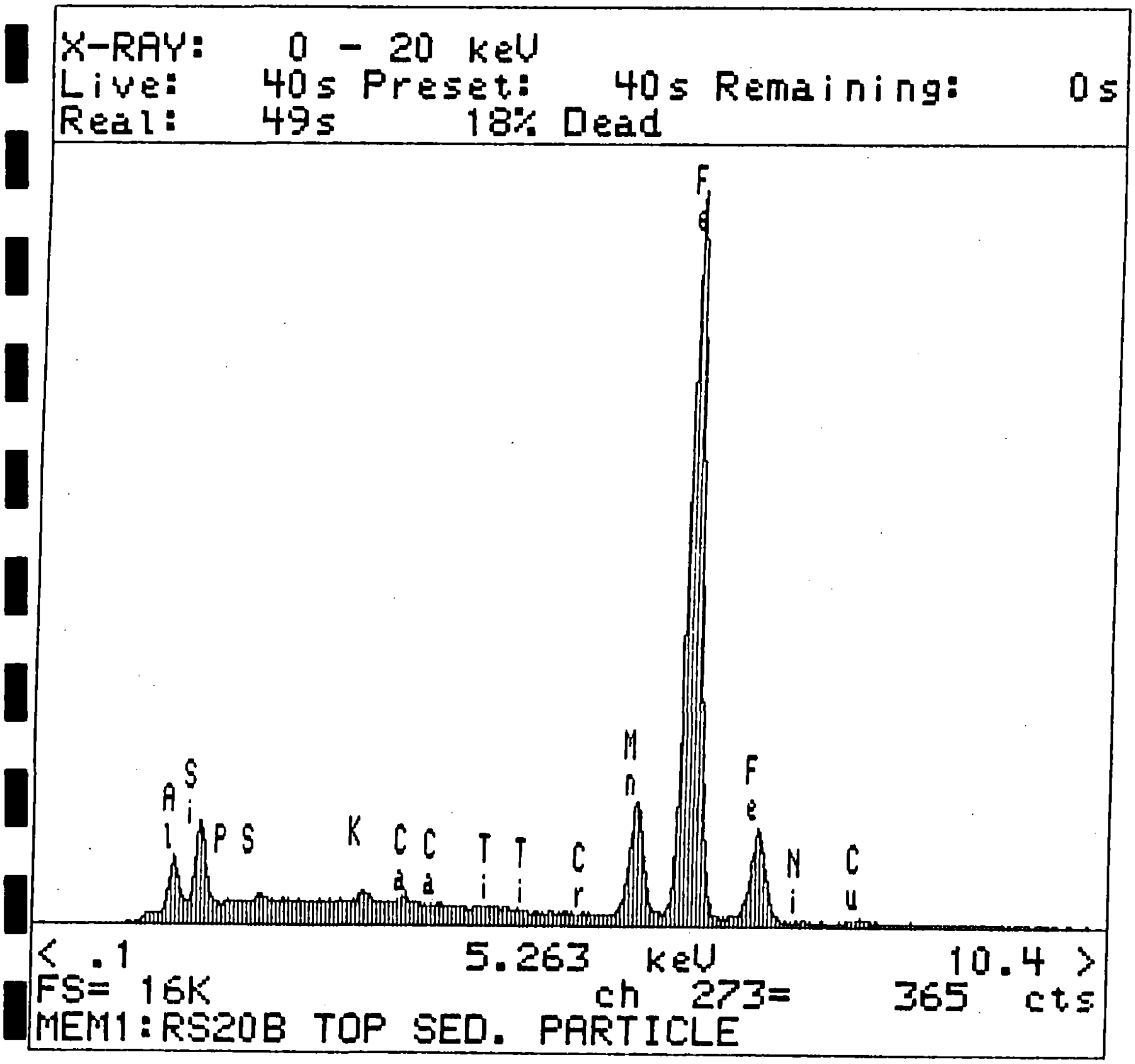
FIGURE #4



```
X-RAY:    0 - 20  keV
Live:    40s Preset:    40s Remaining:    0s
Real:    41s        2% Dead
```

```
< .3                    5.423  keV              10.5 >
FS=  2K                     ch  281=           57  cts
EM1:REF. UNTREATED
```

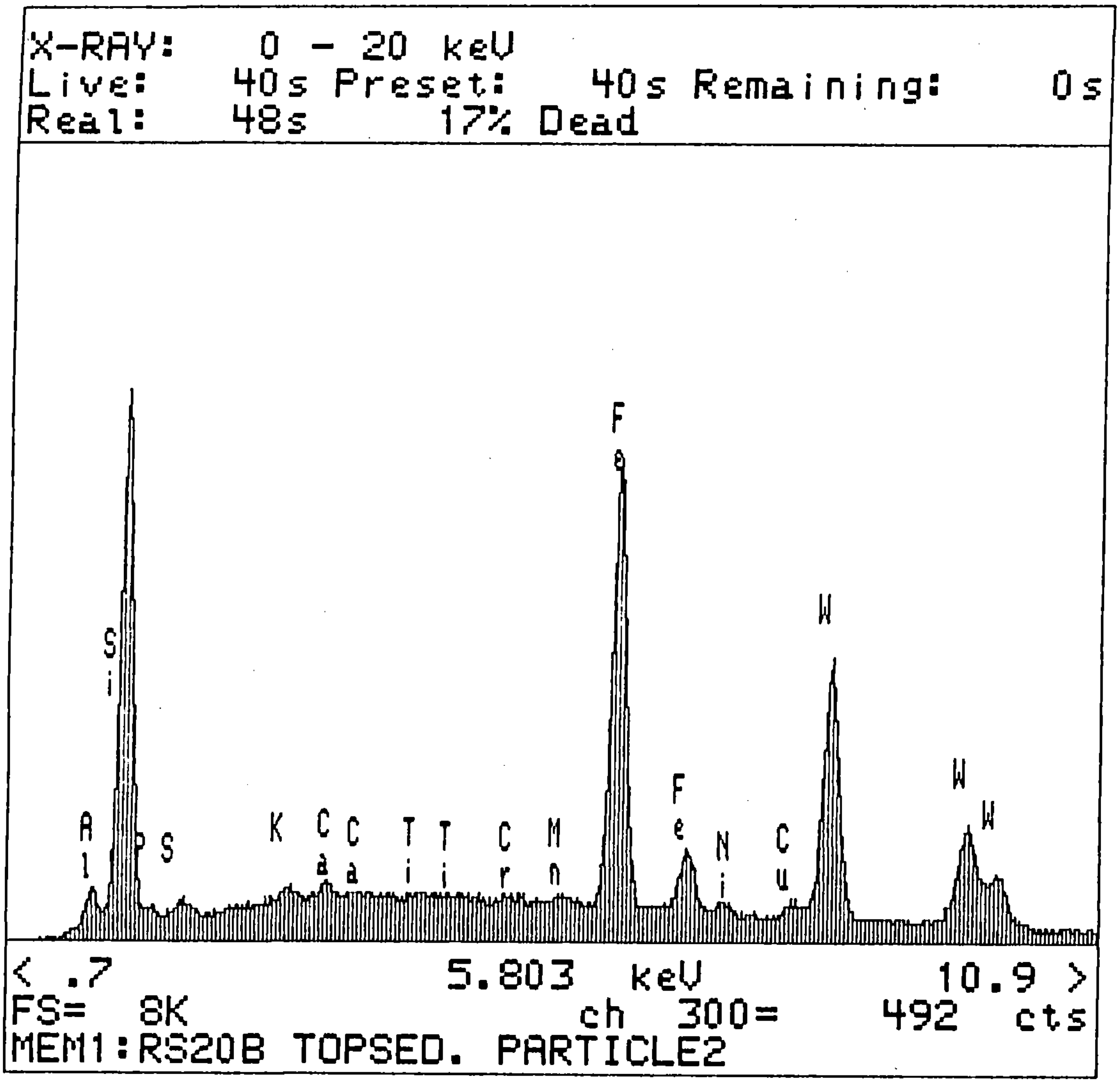
FIGURE #5



```
X-RAY:    0 - 20  keV
Live:    40s Preset:    40s Remaining:     0s
Real:    49s       18% Dead
```

```
< .1              5.263   keV           10.4 >
FS= 16K                ch  273=     365   cts
MEM1:RS20B TOP SED. PARTICLE
```

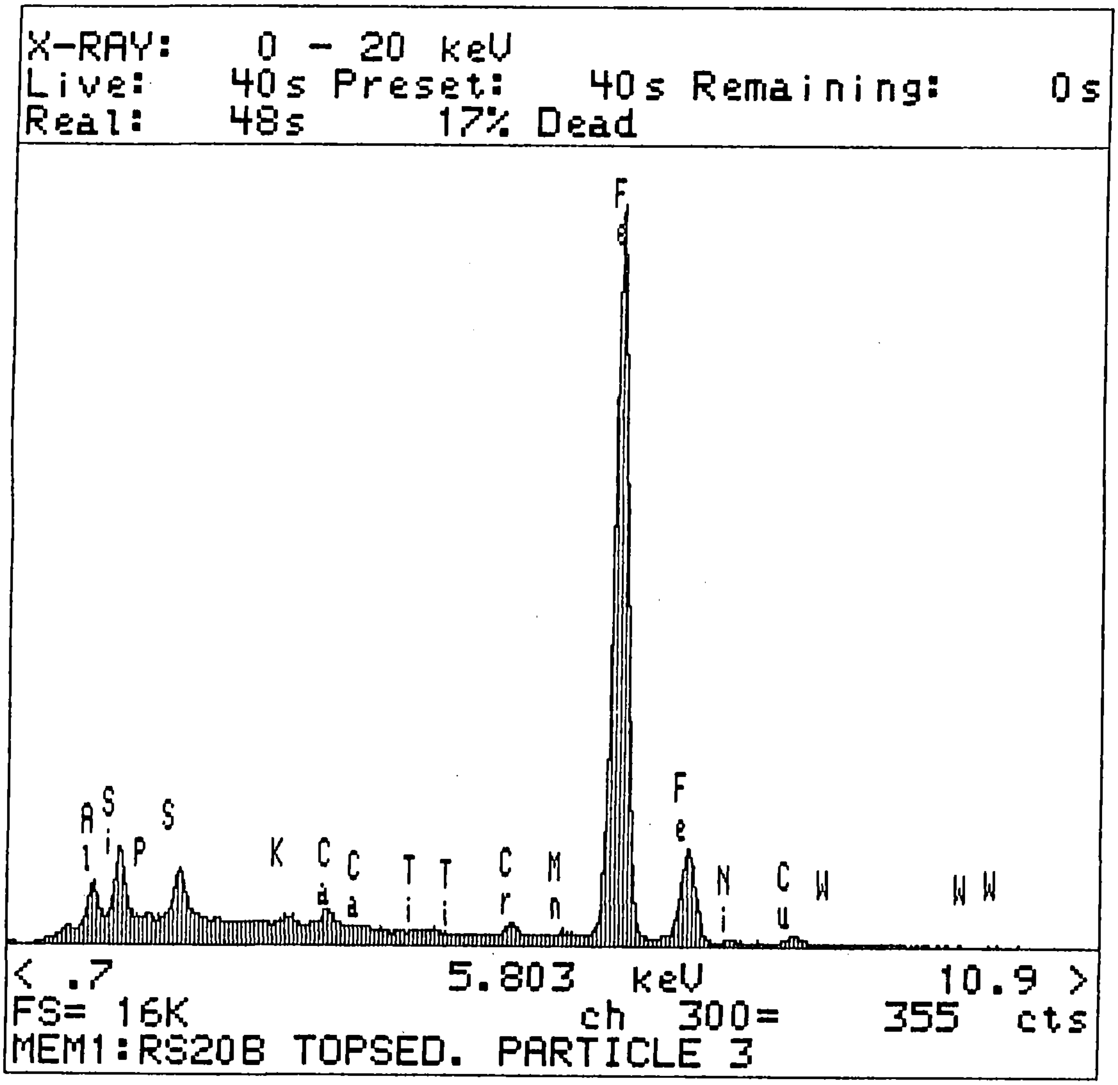
FIGURE #5 (continued)



```
X-RAY:    0 - 20  keV
Live:     40s Preset:    40s Remaining:     0s
Real:     48s       17% Dead
```

```
< .7                  5.803  keV          10.9 >
FS=  8K                    ch  300=     492  cts
MEM1:RS20B TOPSED. PARTICLE2
```

FIGURE #5 (continued)



```
X-RAY:    0 - 20  keV
Live:    40s Preset:    40s Remaining:     0s
Real:    48s       17% Dead
```

```
< .7               5.803  keV            10.9 >
FS= 16K                  ch  300=     355   cts
MEM1:RS20B TOPSED. PARTICLE 3
```

The cylinders and coils which were liberated from the attrition scrubbing were analyzed under EDAX. The photomicrograph is presented in Plate #9 with the EDAX spectrum presented as Figure #6. With this, **ALTECH** was able to determine that the cylinders were not of a metal composition, but were in fact almost entirely Si. Again, the Au peak is from the coating used to cover the sample in SEM preparation. When analysis was centred on the cylinder, Fe values were extremely low, and not of consequence. Thus, the initial hypothesis that these cylinders may represent released metal contamination was proven to be incorrect. Nevertheless, the Si may indicate a release agent which is used in metal manufacture. As well, these cylinders were not visible prior to attrition scrubbing with the chemical additions, suggesting that the action within the attrition scrubber is having a positive effect not only on some metal particles, but on other foreign material as well.

Other particles which looked neither like the Si cylinders, nor the metal particles, were identified. These are outlined in Plate #10 with the EDAX spectrum as Figure #7. The arrows point to 2 areas, one being largely covered by clays, #2, the other being exposed for the most part, #3. Both areas show large components of Ca and S, with #2 being high in Si, Al and Fe as well. Because the photomicrograph shows what appears to be a solid, surrounded by a component of clay, it is possible that this represents a mineral such as gypsum. The long, plate-like habit reaffirms this assumption. From this, then, we can infer that the Welland River Sediment holds much more than metal and clay contamination on its own.

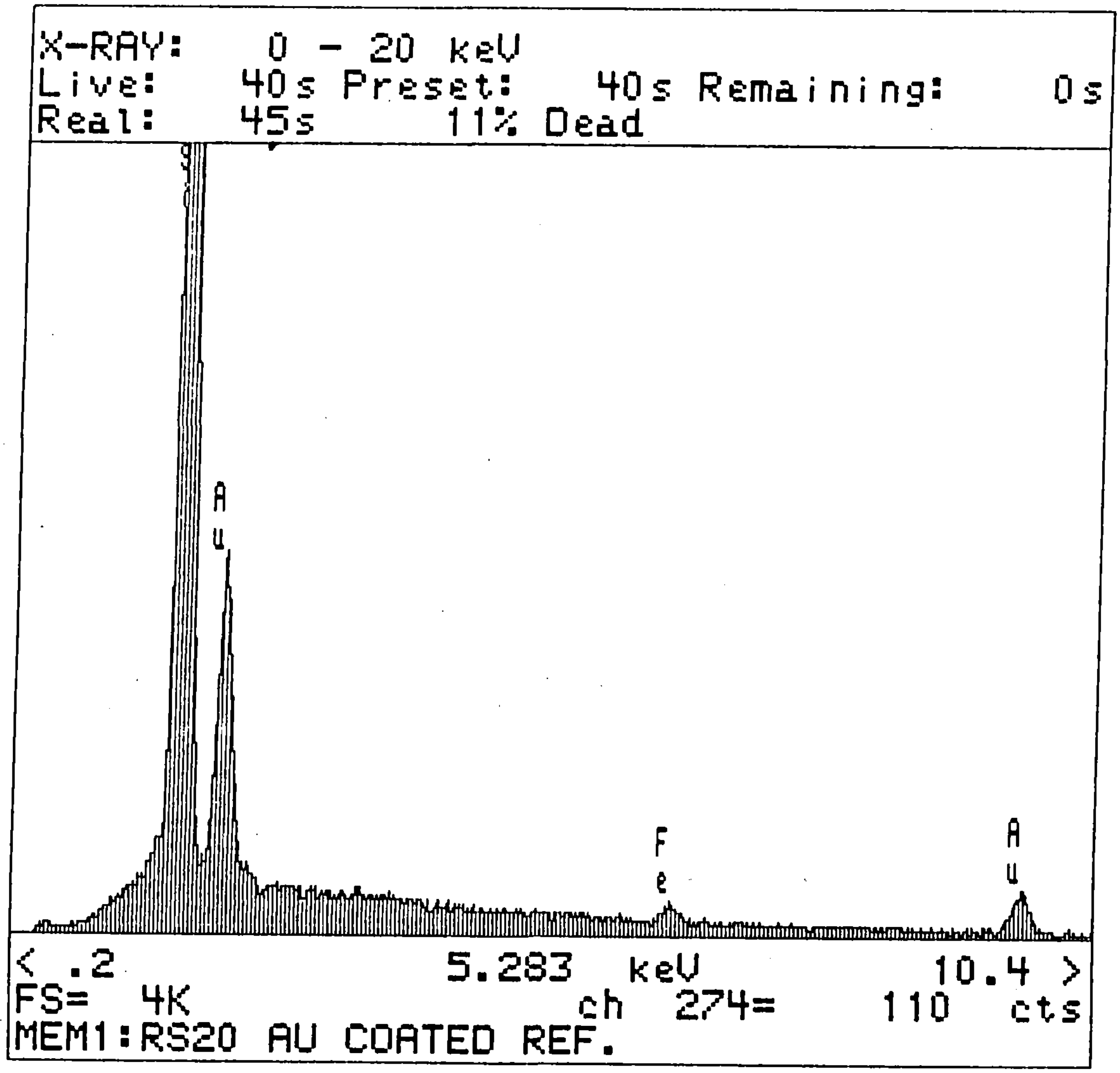
4.5 Final Audit Treatability Run

The final audited treatability run was performed on Monday, March 16, 1992, at the Brock University Geochemistry laboratory. Persons present included 2 **ALTECH** representatives, 1 **WTC** representative, and 1 **Brock University** representative. The process was both photographed and filmed for future reference. Samples were retrieved and duplicates were given to both Brock University and the WTC for analysis. Discussion and analysis of the final treatability run is outlined to follow.

As with all other complete runs, the methodology included:

- attrition scrubbing;
- chemical addition to the attrition scrubber;
- dilution of the sediment sample prior to introduction to the flotation cell;
- water in measured quantities in the flotation cell, and an alteration of the pH;
- air introduction to the flotation cell;
- sediment introduction to the flotation cell;
- chemical addition to enhance separation and flotation of the desired contamination;
- collection of the sample through sampling ports on the side of the flotation cell, and;
- analysis for all metals, TOC and oil and grease in all samples.

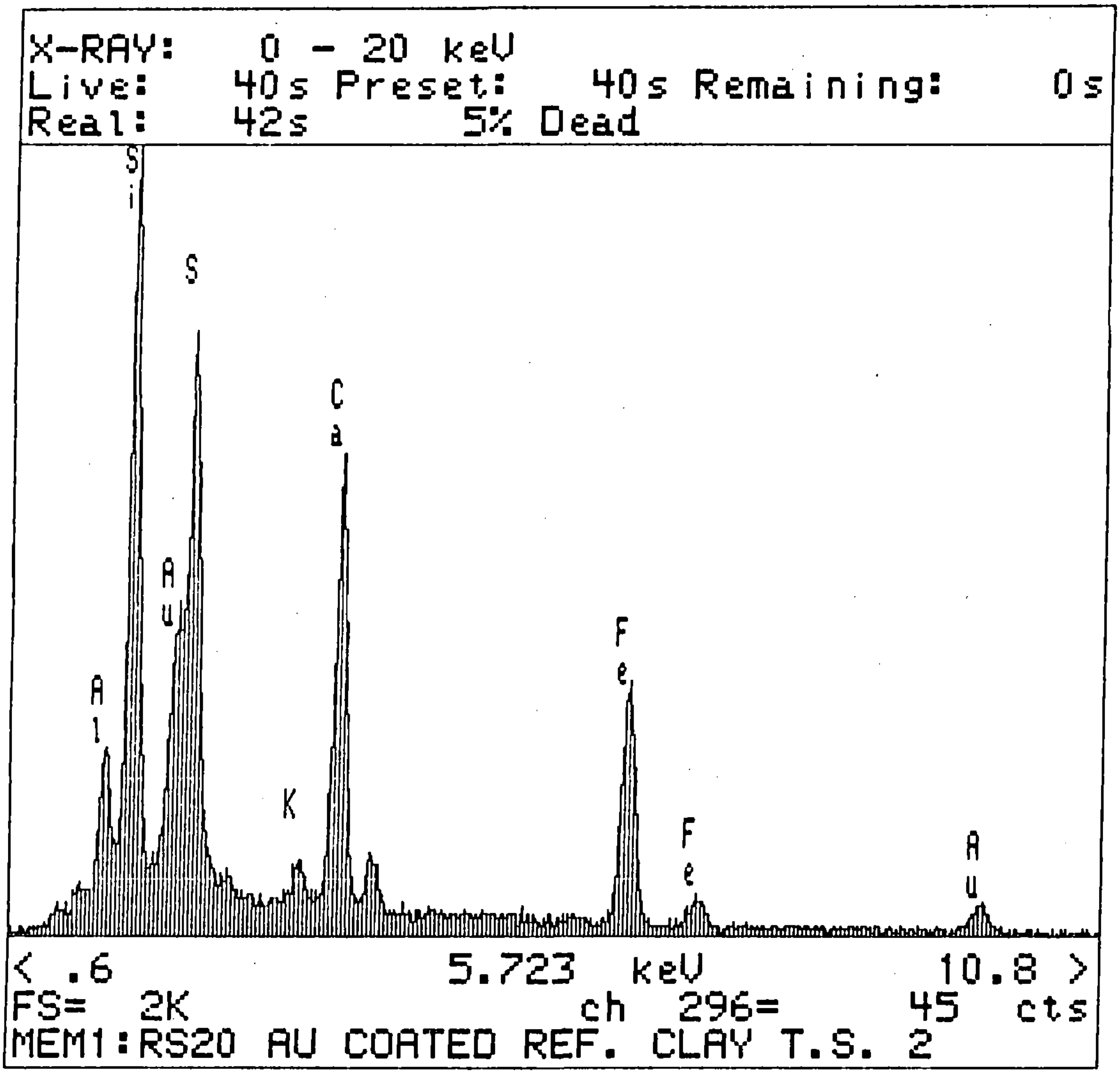
FIGURE #6



```
X-RAY:    0 - 20  keV
Live:     40s Preset:    40s Remaining:     0s
Real:     45s      11% Dead
```

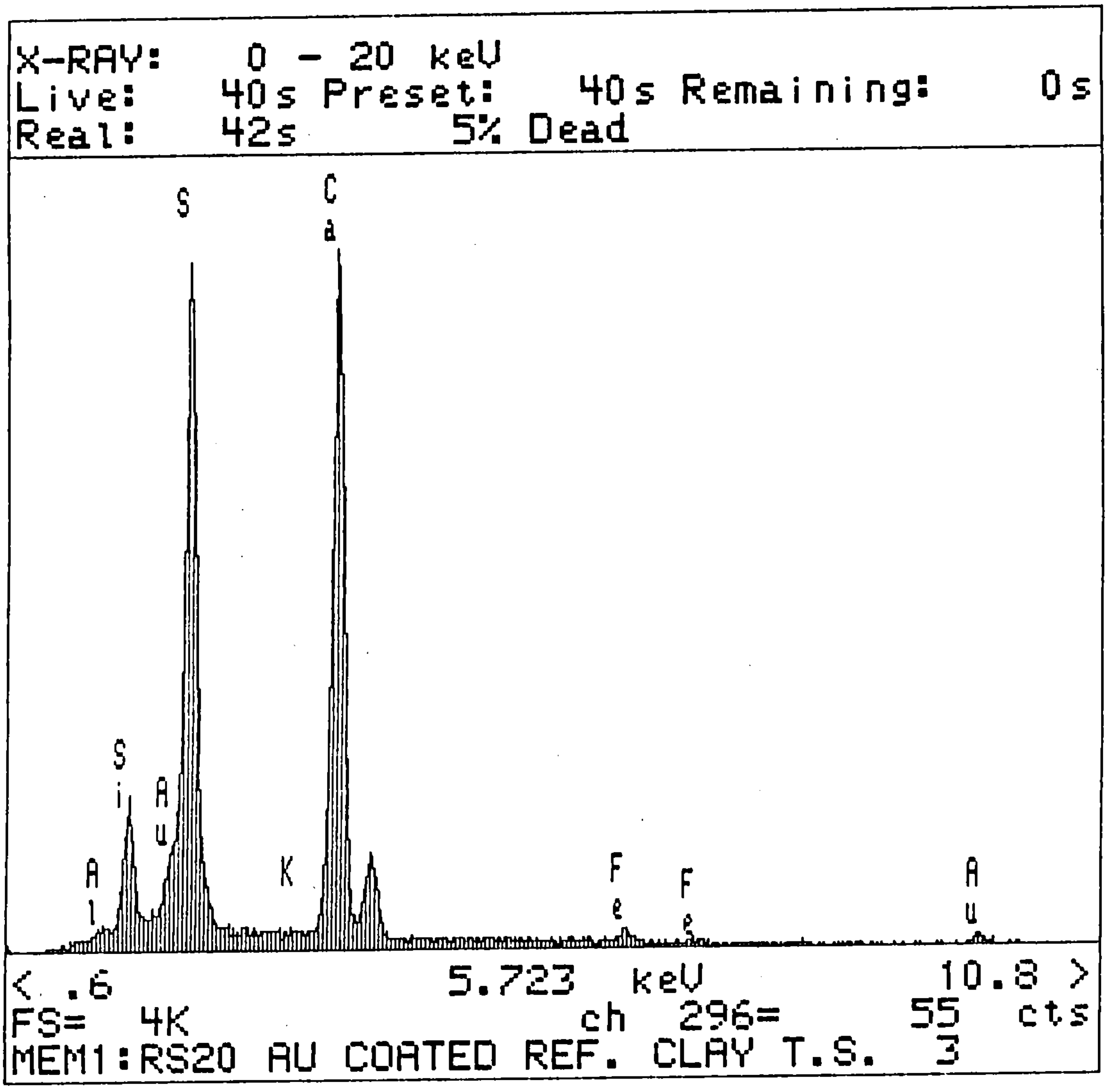
```
< .2               5.283  keV          10.4 >
FS=  4K                 ch  274=     110   cts
MEM1:RS20 AU COATED REF.
```

FIGURE #7



```
X-RAY:   0 - 20 keV
Live:   40s Preset:   40s Remaining:    0s
Real:   42s        5% Dead
< .6              5.723  keV           10.8 >
FS=  2K                ch  296=       45  cts
MEM1:RS20 AU COATED REF. CLAY T.S. 2
```

FIGURE #7 (continued)



```
X-RAY:    0 - 20  keV
Live:    40s Preset:    40s Remaining:     0s
Real:    42s          5% Dead
```

```
< .6              5.723  keV          10.8 >
FS=  4K                ch  296=      55   cts
MEM1:RS20 AU COATED REF. CLAY T.S.   3
```

Metal Removal

The laboratory results from the audited run are included in Appendix #6. The metals which were carried up through the sampling ports by the frothing agent were, by and large, those with atomic numbers greater than 30. The removal rates were greatest for Pb and Zn, with other metals greater than atomic number 30 also being reduced (Ag and As). The concentrations of these other metals were low initially, and therefore, the results may or may not be considered significant.

Be and Cd were metals which presented such small alteration in their concentrations, as well as having low concentrations to begin with, that the results cannot be considered significant.

Al was reduced in concentration in the base sediment which remained in the flotation cell. The Al could have been a component of the clay, and if this is the case, this suggests that the Gibbsite based clay is rising with the air and other selected elements, leaving some metal (Fe) and other elements to concentrate in the base sediment. In this way, the process appears to be quite discerning, with the chemicals quite selective in what metals will rise and be removed from the Welland River sediment.

The liquids analysis revealed data consistent with the conclusions reached in earlier trials; that the metal is highly insoluble and the amount of metals found in the liquid is not significant. This data has been included as well in Appendix #5.

The liquid analysis of water in the float cell prior to sediment introduction reveals that the addition of any foreign metal component as contamination was negligible. The most significant contribution from the water by way of metal was 0.17 ppm for Al; a value which in real terms is highly insignificant.

TOC and Oil and Grease Removal

The results for TOC and oil and grease are included in Appendix #5 with the metal results. The starting values were elevated for both TOC and oil and grease in the sediment characterization; 21.2% (212,000 ppm) and 24,441.2 ppm respectively. The end results for TOC and oil and grease, found in the sediment remaining at the base of the flotation cell following the audited treatability run, were 14.7% (147,000 ppm) and 21,455.8 ppm respectively. The Provincial Sediment Quality Guidelines (PSQG) are 10% for TOC (100,000 ppm), and the oil and grease values are carried over from the Open Water Disposal Guidelines at 0.15% or 1,500 ppm. Both TOC and oil and grease were reduced through the ALTECH soil washing process, however, the values were not reduced sufficiently to meet the PSQG. Nevertheless, both TOC and oil and grease were reduced in the process which was geared primarily towards metal removal. With further optimization, chemicals and trials, it is felt that satisfactory results are obtainable for TOC removal. Oil and grease removal to the PSQG of 0.15% may not be economically attainable at the present time, however, further experimentation is required.

TOC and oil and grease were removed in limited quantities, and it is not believed that their removal rates are interdependent, nor is it believed that their removal rates are associated with metal removal rates in any way.

In the froth removed from the sampling ports, the analytical values for oil and grease were higher than those values obtained for TOC. This suggests that for the quantities given, and the conditions during the trial, oil and grease has a greater affinity to rise than does the TOC component in the Welland River Sediment. This, naturally, is a generalized comment and may change as the original sediment changes, or the conditions of treatability are altered.

5.0 DISCUSSION OF RESULTS

The result of the bench scale trials can be summarized as:

- Fe, Al and Mg are elements which are natural components of clay. By and large, the Acres processed Welland River Sediment is estimated at over 90% clay. Because the quantities of Fe, Al and Mg can vary considerably in a clay soil package, the quantities of each which comprises contamination and/or sediment is difficult to determine. Nevertheless, each of these elements was treated as if it were contamination and is presented in the data.
- The original sediment characterization (unprocessed by Acres) showed Cu, Ni, Cr, Zn, and Fe concentrations greater than SEL's for the PSQG. Concentrations of Pb and Mn approached but did not exceed the SEL.
- With the exception of Mg and Zn, all metals of concern to the PSQG were of a significantly higher concentration in the Acres processed sediment than in the original unprocessed sediment.
- Characterization of the processed sediment revealed Fe, Mn, Cu, Ni, Cr, and Pb concentrations greater than the SEL for the PSQG: Zn, Cd, and As concentrations did not exceed the SEL.
- Attrition scrubbing on its own yielded useful interim results. Firstly, solitary attrition scrubbing of the sediment did not significantly release metals from the soil into a liquid phase. Secondly, after attrition scrubbing, the sediment would settle, and within a period of days separate into a dark bottom layer ('base' sediment), and a top coloured (or oxidized) layer. The top coloured layer was almost exclusively orange and 1 to 3 cm in depth. Samples of the coloured layer and the 'base' sediment were analyzed and revealed that the coloured layer contained less metal than the 'base' layer. This would indicate that the metal content settled preferentially to the finer sediment; thereby indicating that the metal may be contained within the sediment in discrete particles, and have a quicker settling velocity than a considerable portion of the sediment.
- The combination of attrition scrubbing, chemical addition and flotation removed some selected metals in differing quantities from the Welland River sediment. The metals which were removed in the overflow froth from the flotation cell were, interestingly, above atomic number 30. Metals which were generally not removed, and therefore concentrated in the base sediment remaining in the flotation cell, were below atomic number 30. This was the

only pattern found, and at the point of publication of this report, still presents an undefined occurrence.

- Zn and Pb were removed in considerable quantities, both to below the SEL for the PSQG. These metals had the most successful rate of removal throughout the treatability trials.
- High acid value trial runs (leaching) did not remove a significant amount of contamination metal to the liquid. Therefore, it is felt that the metal is in a highly insoluble state; small micron sized shavings of metal trapped in tightly bound clay.
- SEM through backscatter, and EDAX spectrum analysis reveal the metal contamination to be visually similar to the clay. EDAX spectrum also displays differing components in each metal particle found in the sediment, however, the major component by far was always Fe.
- Concentrations of oil and grease, as well as TOC, were both reduced through the **ALTECH** soil washing process. The treatability trials were concentrating significantly on the metal contamination, however, optimization towards oil and grease and TOC removal appears to be possible in the future.

A general series of recommendations from **ALTECH** may be outlined as follows:

- Because the metal is insoluble, leaching does not produce any significant results, (see Alt-3rs results). Therefore, physical processes with chemical enhancement are the only option if straight landfill is ruled out. The soil washing was partially successful in removing metal with atomic numbers greater than 30, and for this reason, it is felt that with further treatability studies the system may be improved to yield significant results.
- Although not part of the scope of the treatability study, it was noticed that the dried sediment was very magnetic. However, work on the Frantz magnetic separator could not separate insoluble metal from the sediment with any great degree of success. A more intensive magnetic separation process may be combined with the soil washing treatability process as outlined in the report.

6.0 CONCLUSIONS – SITE SPECIFIC

The metal contamination in the Welland River Sediment does not appear to be significantly leachable. The sediment package of fine metallic particles in tightly bound clay is the hardest sediment package to clean using the **ALTECH** soil washing principle. Nevertheless, because the metal is not significantly leachable, the only remaining option open for remediation is physical removal, enhanced proportionately by chemical promoters.

The SEM and EDAX analysis have proven the metal to be visually similar to the surrounding clay. Further, the study involved insoluble metal particles entrained in tight clay, possibly the most difficult situation for remediation.

ALTECH has had limited success in removing the metal from the sediment. Zn and Pb were removed in the largest quantities and this, at the very least, proves that the metal can be removed from the sediment through the soil washing process. Contaminant specific methodologies were unable to be fully developed under the scope of the treatability program, nevertheless, with further optimization and trial, it is felt that the combination of physical and chemical treatments will be able to remove a significant portion of each metal in the sediment.

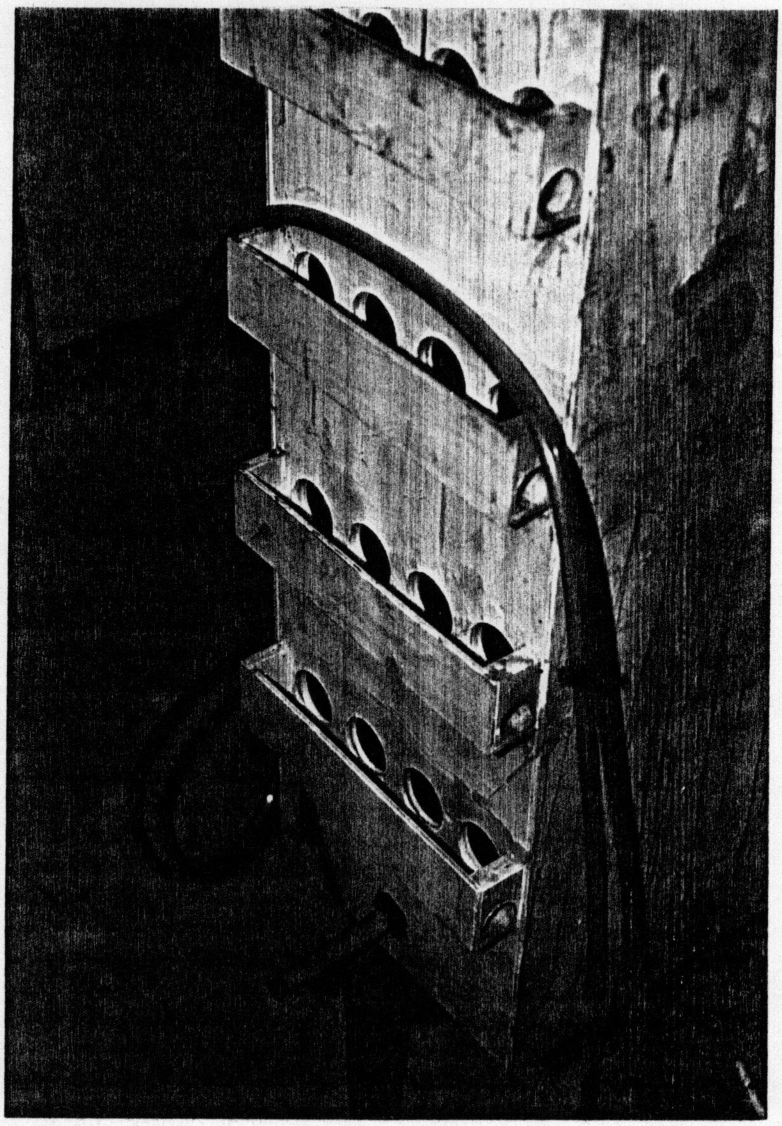
Significant quantities of oil and grease as well as TOC were removed through the ALTECH soil washing process. At this point, however, physical removal of oil and grease to meet the PSQG oil and grease parameters of 0.15% or 1,500 ppm (carried over from open water disposal guidelines), does not present itself as being easily attainable without further optimization of the system.

7.0 COMMERCIALIZATION PLAN

Future plans for the **ALTECH** soil washing system include additional optimization. The Welland River Studies have proven that metal removal through physical and chemical processes is possible, although complicated.

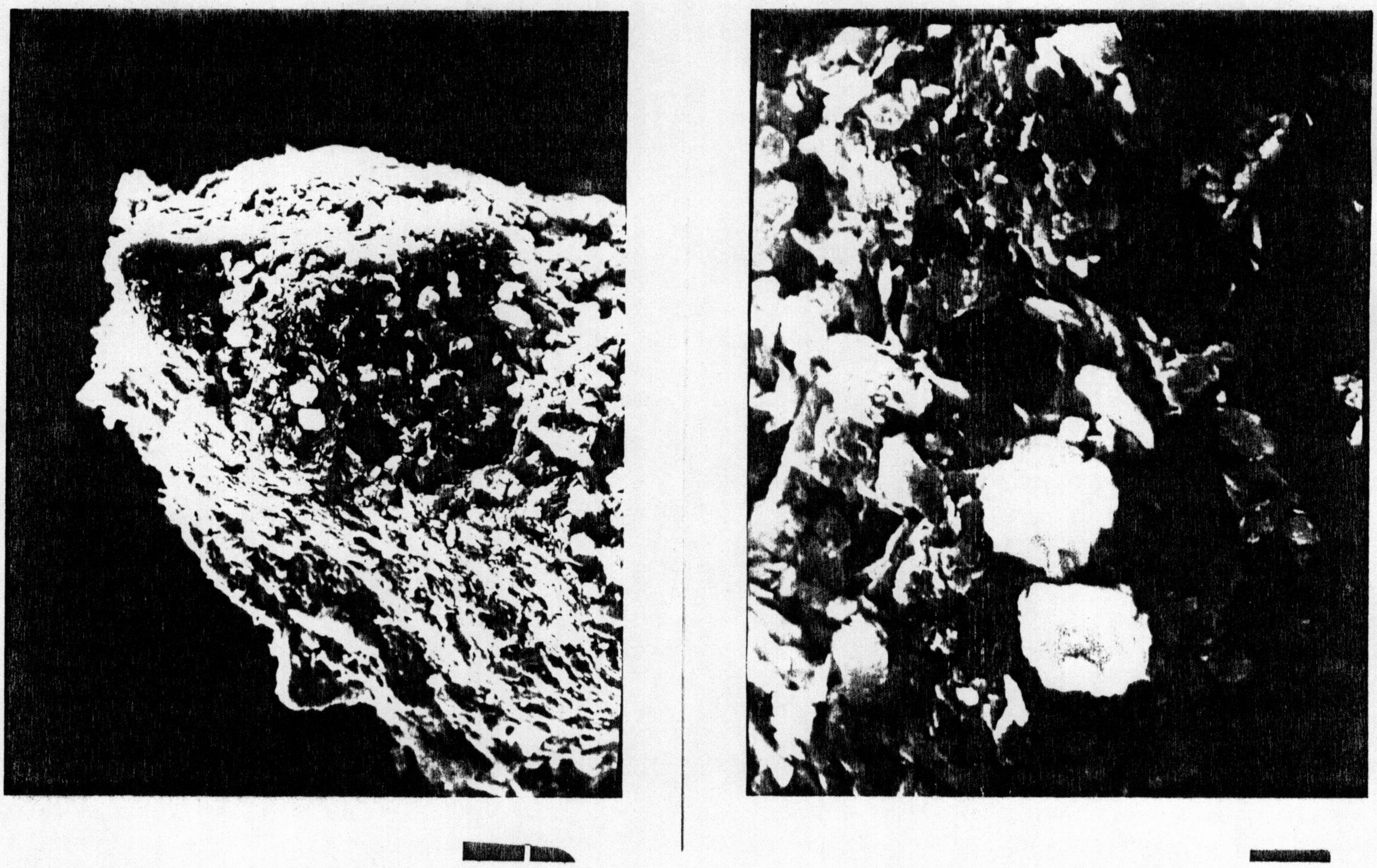
The commercialization plan includes developing methodologies for the removal of specific metals from specific sediment types. This type of research and development will require a concerted effort and additional bench scale treatability trials with prepared sediment. Nevertheless, with the encouraging results for Pb and Zn removal from the Welland River Sediment, further optimization of the system seems possible, with full commercialization closely to follow.

PLATE #1



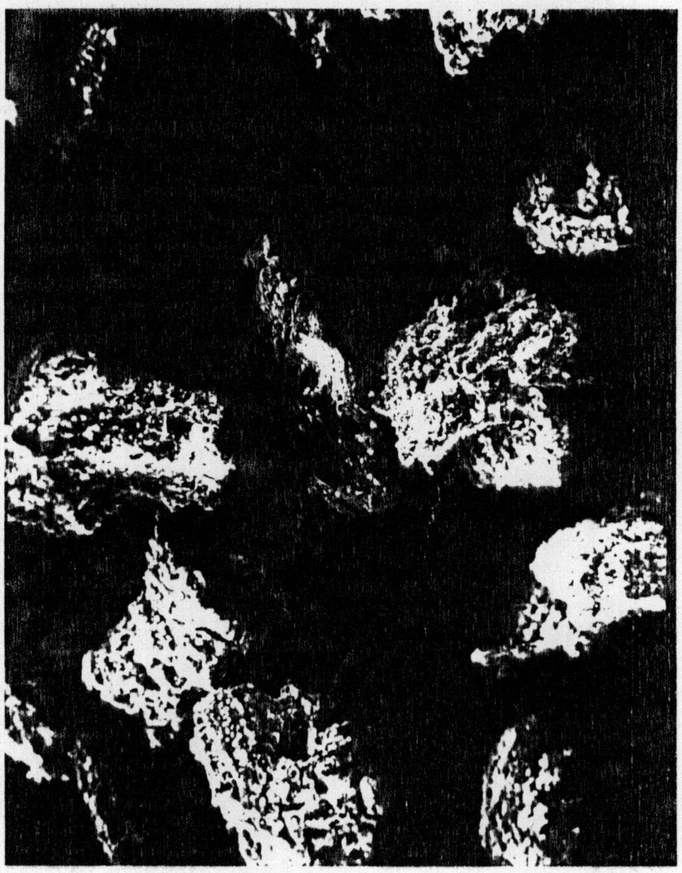
Float Cell and Sampling Ports

PLATE #2



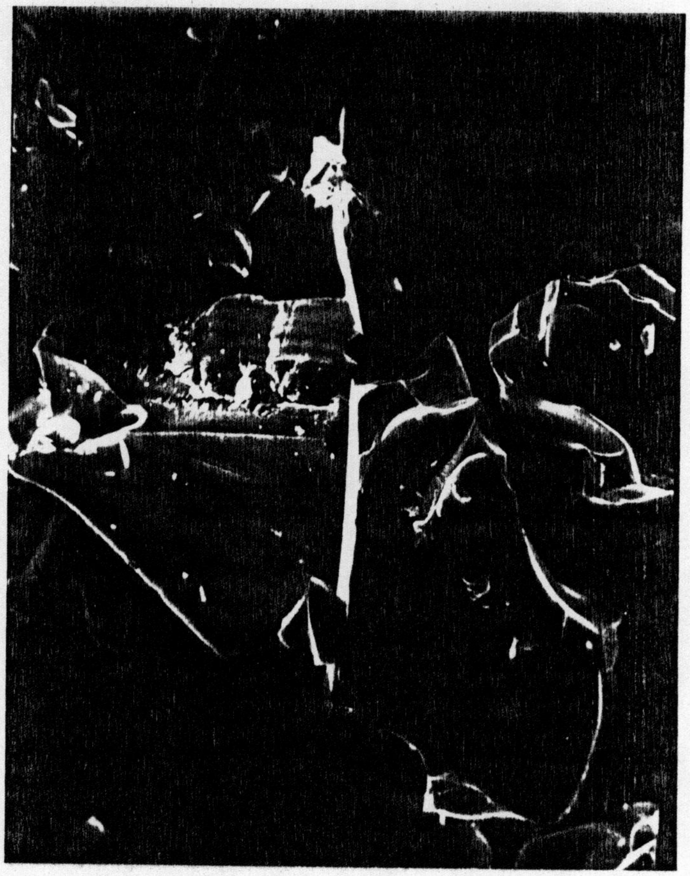
Original Sediment, SEM

PLATE #3



Acres process sediment, less than 75 microns nominally; untreated

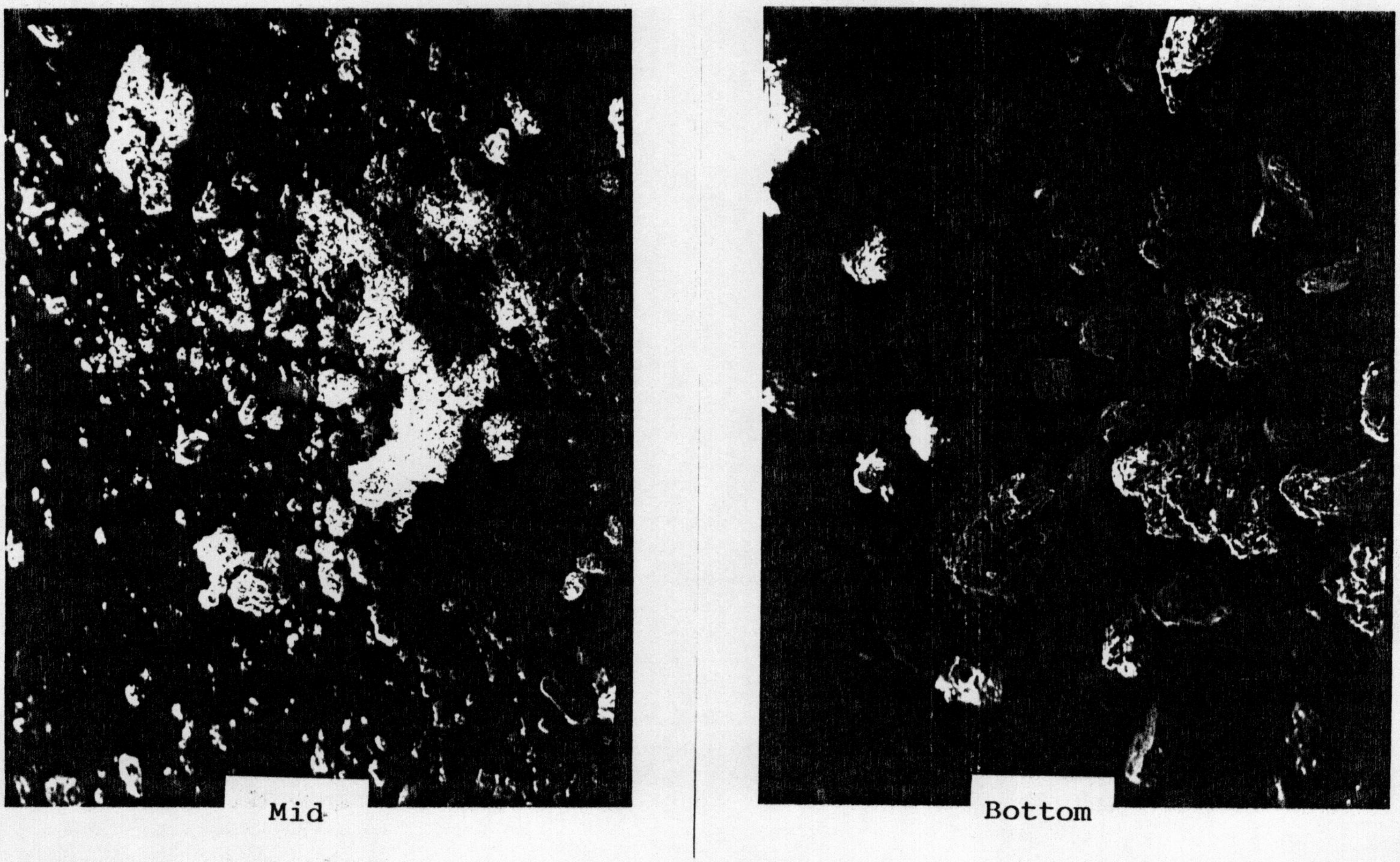
PLATE #4



SEM of collector chemical; dried

PLATE #5

Top

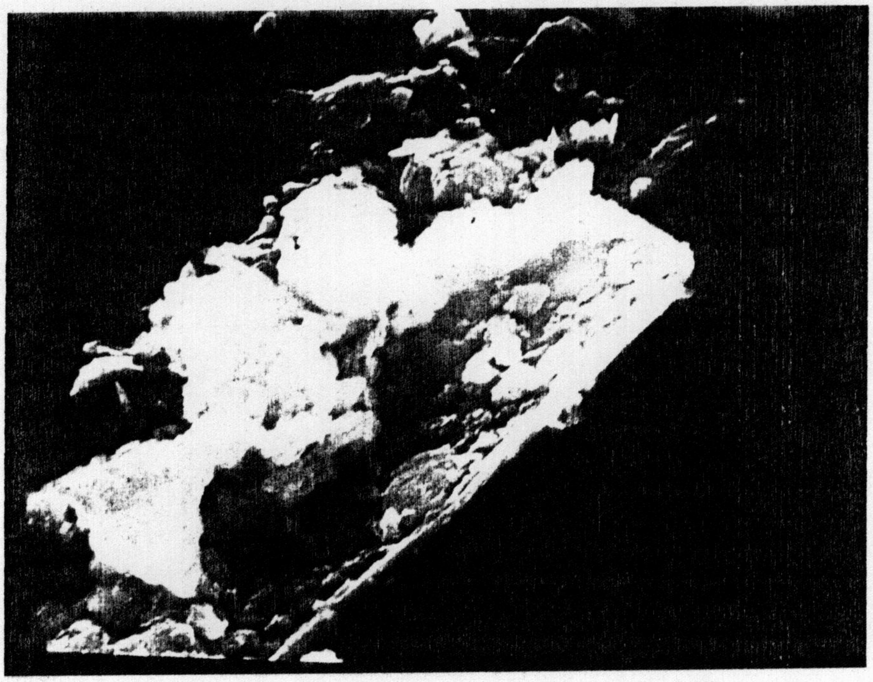


Mid

Bottom

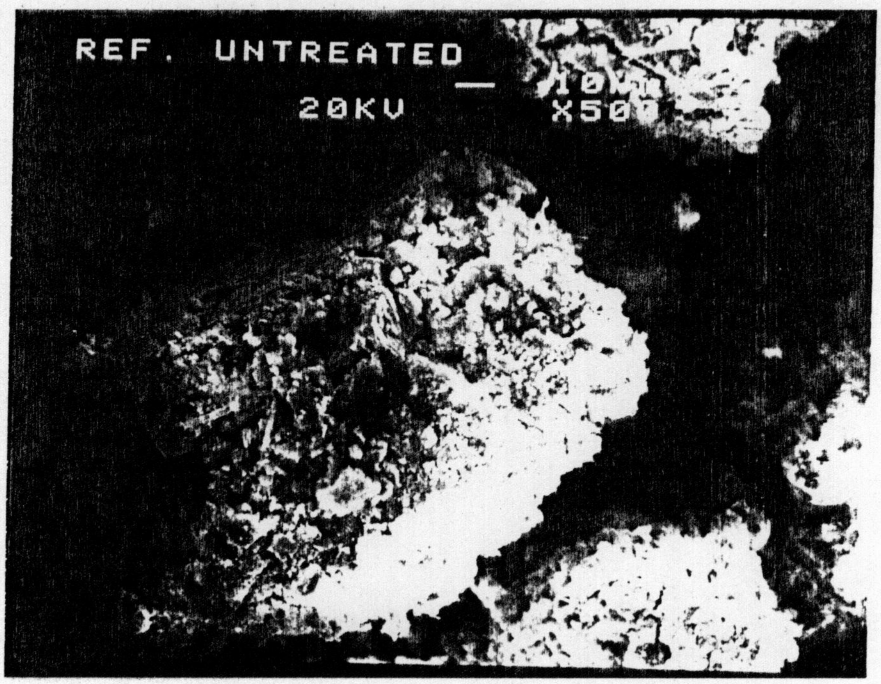
Stratified samples; top=froth, mid=liquid, bottom=base sediment

PLATE #6



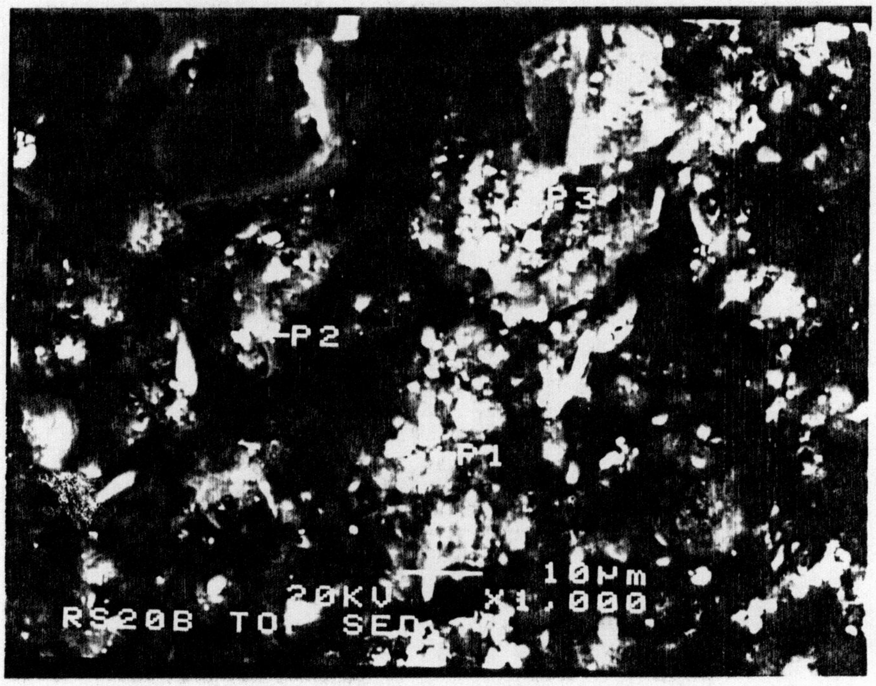
Unspeciated Particle

PLATE #7



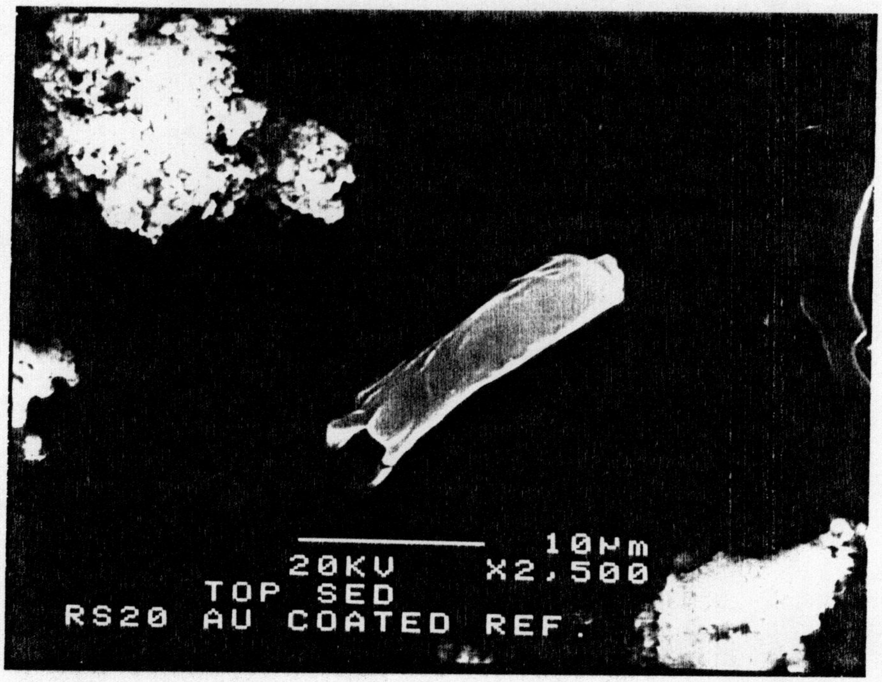
Reference sediment, untreated

PLATE #8



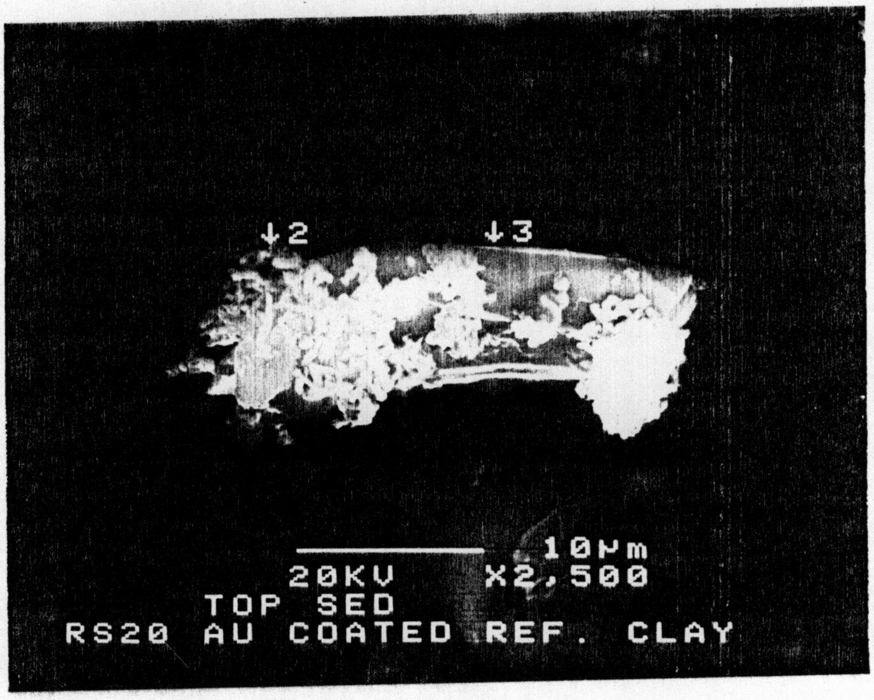
Coloured settled sediment with identified metal particles

PLATE #9



Silicone cylinder

PLATE #10



Clay and gypsum particle

APPENDIX #1

TERRA CHEM
Environmental Analysts & Consultants
81 Keefer Road, Thorold, Ontario, Canada L2V 4M8 (416) 227-4156

Alt-1RS

Mon, Mar 30, 1992 6:27 PM

	#	Cu	Ni	Co	Cr	Ag	Al	Zn	#
1	Un1S	0.001	0.064	0.001	0.001	0.001	0.24	0.046	Un1S
2	Un2S	0.001	0.086	0.002	0.001	0.001	0.29	0.039	Un2S
3	Un3S	0.001	0.089	0.001	0.001	0.001	0.35	0.039	Un3S
4	Un4S	0.001	0.063	0.004	0.001	0.001	0.58	0.046	Un4S
5	Un5S	0.001	0.061	0.001	0.001	0.001	0.35	0.038	Un5S
6	Un6S	0.002	0.073	0.001	0.001	0.001	0.35	0.046	Un6S
7	Overs	0.001	0.093	0.002	0.001	0.001	0.32	0.033	Overs
8									
9	Sed1	263.081	837.466	57.107	780.945	3.612	69670.05	829.754	Sed1
10	Un1-sed	247.336	572.541	47.131	451.844	4.508	84631.15	1096.311	Un1-sed
11	Un2-sed	252.477	677.739	49.097	603.147	3.934	69055.94	1000.874	Un2-sed
12	Un3-sed	296.939	841.545	60.058	962.099	4.810	67419.83	880.466	Un3-sed
13	Un4-sed	275.130	629.766	47.660	571.274	4.116	79484.40	909.879	Un4-sed
14	Un5-sed	271.591	522.917	41.667	385.417	4.545	92803.03	1251.894	Un5-sed
15	Un6-sed	273.124	463.787	39.703	320.681	4.581	95397.03	1332.897	Un6-sed
16	Over-sed	201.500	522.100	42.400	298.200	4.100	91540.00	1049.000	Over-sed
17	Sed2	276.214	842.411	60.894	795.594	3.774	77570.38	944.512	Sed2
18									
19	NBS 2704m	96.056	46.173	23.135	130.418	2.050	71310.04	465.638	NBS 2704m
20	NBS actual	98.600	44.100		135.000	2.171	61100.00	438.000	NBS actual

Alt-1RS Mon, Mar 30, 1992 6:27 PM

	Mn	Fe	Mg	Se	As	Cd	Pb
1	0.014	0.281	11.400	0.001	0.000	0.001	0.01
2	0.019	0.280	10.940	0.001	0.004	0.001	0.01
3	0.021	0.324	11.050	0.001	0.001	0.001	0.01
4	0.015	0.325	11.020	0.001	0.000	0.001	0.01
5	0.015	0.191	11.090	0.001	0.001	0.001	0.01
6	0.020	0.300	10.870	0.001	0.004	0.001	0.01
7	0.045	0.194	11.500	0.001	0.000	0.001	0.01
8							
9	846.349	60152.284	13666.537	0.292	21.563	1.687	158.32
10	713.115	45963.115	16803.279	0.538	19.205	2.582	165.49
11	654.138	51617.133	15894.522	0.689	40.131	2.226	105.94
12	813.411	65758.017	15597.668	0.608	26.230	2.117	144.31
13	712.738	58730.503	19540.728	0.617	39.443	2.270	114.90
14	685.606	37821.970	14678.030	0.672	39.142	2.780	17.73
15	599.913	41906.632	19284.468	0.788	40.766	3.595	19.76
16	588.000	39310.000	14160.000	0.778	24.207	1.784	68.10
17	757.854	67962.056	14585.883	0.363	26.374	1.563	175.46
18							
19	513.471	38861.773	11069.895	1.018	25.411	3.081	166.63
20	555.000	41100.000	12000.000	1.120	23.400	3.450	161.00

APPENDIX #2

QA/QC

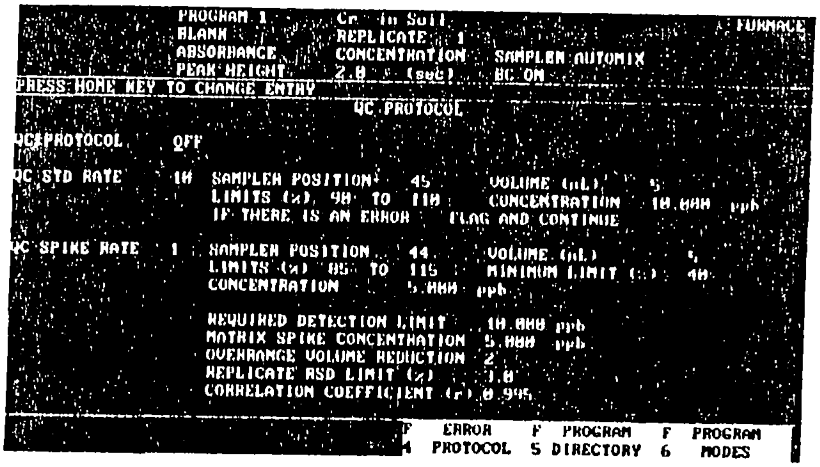
ATOMIC ABSORPTION SPECTROMETER
BROCK UNIVERSITY

3 Parameter Descriptions

NBS SRM 2704
(Buffalo River Sediment)

This section provides a guide to the QC Protocol software page, describing each on-screen parameter field and its function. In the following pages element specific features are highlighted with an **(E)**, while automatic run features are highlighted with an **(A)**. Quick Reference Guide 1 at the rear of this manual indicates which parameters are element specific and which are automatic run specific.

Section 4 describes the operation of each QCP function, including those that do not require operator entries, with real examples of QCP reports. The QC Blanks, Duplicates and Matrix Spike facilities are not discussed in this section as they do not require operator input on the QC Protocol page. Figure 1 displays the layout of the QC Protocol software page, showing the parameter fields discussed in the following sections.



```
             PROGRAM 1        Cr In Soil                       FURNACE
             BLANK            REPLICATE   1
             ABSORBANCE       CONCENTRATION   SAMPLER AUTOMIX
             PEAK HEIGHT      2.0   (sec)     BC ON
PRESS HOME KEY TO CHANGE ENTRY
                                 QC PROTOCOL

QC PROTOCOL     OFF

QC STD RATE     10   SAMPLER POSITION   45     VOLUME (uL)        5
                     LIMITS (%)  90  TO  110   CONCENTRATION    10.000  ppb
                     IF THERE IS AN ERROR    FLAG AND CONTINUE

QC SPIKE RATE    1   SAMPLER POSITION   44     VOLUME (uL)          5
                     LIMITS (%)  85  TO  115   MINIMUM LIMIT (%)  40
                     CONCENTRATION      5.000  ppb

                     REQUIRED DETECTION LIMIT     10.000 ppb
                     MATRIX SPIKE CONCENTRATION   5.000  ppb
                     OVERRANGE VOLUME REDUCTION   2
                     REPLICATE RSD LIMIT (%)      3.0
                     CORRELATION COEFFICIENT (r)  0.995

                                          F  ERROR     F  PROGRAM    F  PROGRAM
                                          4  PROTOCOL  5  DIRECTORY  6  MODES
```

Figure 1. The QCP software screen lay-out

NOTE All readings and calculations in SpectrAA instruments are calculated to five significant figures but are reported to the number of significant figures used on the standards page (except in scale expansion). In the %R calculation the measured concentration might be 7.4444 reported as 7.4 and the specified concentration 7.5. The %R will be correctly reported as 99.3% not 98.7% as would seem correct from the print out.

Where applicable the concentration units and number of decimal places are automatically set to be the same as those entered on the STANDARDS page.

3.1 QC Protocol ON/OFF

When QC PROTOCOL ON is selected the activated features on this page will be applied to each of the analytical methods in the automatic run sequence. After turning QCP ON and exiting the QC Protocol page, the letters QC appear in the top right hand corner of the screen.

If QC PROTOCOL is OFF the tests set on page 23 will not be applied to the selected program(s) in the automatic run. Operation will revert to normal SpectrAA-300/400 operation with the exception that a manual sampling automatic run will not be available.

3.2 QC Standards

NBS SRM

QC standards are used to monitor the continuing validity of analytical calibrations and are also frequently referred to as check standards.

(E) QC STD RATE XX

The frequency at which the QC standard is measured. If 5 is selected the QC standard will be analysed every 5 samples. To turn OFF the QC Standard check enter '0'. Valid range 0 to 45. Default setting 0.

(A) SAMPLER POSITION XX

The position of the QC Standard in the sampler carousel (position 45 is recommended). Valid range 1 to 45, default setting 45.

(E) VOLUME (µL)

This is the volume of the QC Standard to be injected. The volume should be selected so that the absorbance is around the middle of the calibration range. Valid range 0 to 70, default setting 5.

(E) LIMITS (%) XXX TO YYY

The calculated recovery %R will be tested against these limits. For example, for a particular element the limits could be 110% to 90%. If the calculated recovery is outside these limits the selected error action will be taken. Valid range of each limit 0 to 200. Default settings 90 and 110.

The recovery, %R, is calculated by:

$$\%R = \frac{\text{Measured concentration of QC Standard}}{\text{Specified concentration of QC Standard}} \times 100$$

(E) CONCENTRATION

This is the concentration of the QC Standard and should be selected so that the expected result is well within the calibration range. Valid range 0.001 to 9999, default 1.000.

(A) IF THERE IS AN ERROR

This field defines the action to be taken if the calculated recovery for the QC Standard is outside the selected limits. User selectable options are:

RECALIBRATE AND REPEAT
RESLOPE AND REPEAT
FLAG AND CONTINUE
STOP

Default selection, STOP. Typical operation examples of these options are given in Section 4.2.

3.3 QC Spikes

A QC Spike is performed when a known volume of a known concentration standard solution is added to a sample. The percent recovery of the added analyte is calculated and compared to user specified action limits. Spikes are used as an indication of the effects of adding analyte to the sample matrix. If interferences occur this will be reflected in a poor recovery and so the Method of Standard Additions might be required. If the recovery is acceptable the analysis can be carried out using normal calibration techniques.

(E) QC SPIKE RATE XX

The frequency at which the QC Spike is measured. If 5 is selected the QC spike will be analysed every 5 samples. To turn OFF the QC Spikes, enter '0'. Valid range 0 to 45, default setting 0.

(A) SAMPLER POSITION XX

The position of the QC Spike solution in the programmable sample dispenser (PSD) carousel (position 44 recommended). Valid range 1 to 45, default setting 44.

(E) VOLUME (µL) XX

The volume of QC Spike solution to be added to the total sample volume (sample + blank + modifier) of the currently selected program. Valid range 0 to 70, default setting 5.

(A) LIMITS (%) XXX TO YYY

The percent recovery action limits for the QC Spike procedure. QCP will test the calculated spike recovery against these limits. Valid range for each limit 0 to 200, default settings 85 and 115.

(A) MINIMUM LIMIT (%) XXX

The minimum acceptable result for the spike recovery. Valid range 0 to 200, default setting 40.

(E) CONCENTRATION XXXX

This is the concentration of the QC Spike standard, the units and decimal places are automatically set to those selected on the

STANDARDS page. Note that the user does not have to enter the SPIKE **ADDED** CONCENTRATION, this is automatically calculated by QCP. Valid range 0.001 to 9999, default setting 1.000.

The following formula is used:

$$\text{SPIKE ADDED CONC} = \frac{\text{QC SPIKE VOLUME}}{\text{SAMPLE VOLUME}} \times \text{QC SPIKE CONCENTRATION}$$

3.4 Overrange Volume Reduction (OVER)

The OVER feature will automatically reduce the volume of any solution when the calculated concentration result *exceeds* the calibration range by more than 10%. In all cases only the sample volume will be reduced. The selected blank and modifier volumes will remain the same to ensure that the blank correction being applied to the solutions remains valid.

The OVER procedure will be repeated as many times as it takes to get the result in range or until the REDUCED VOLUME required is less than 1 microlitre.

This facility is valid in the CONCENTRATION and STANDARD ADDITIONS modes of calibration.

(A) OVERRANGE VOLUME REDUCTION X

The selected sample volume for the current program will be reduced by this factor if the solution result is greater than the upper standard concentration plus 10%. Valid range 1 to 5, default setting 2.

3.5 Matrix Spike

The MATRIX SPIKE is a spike that has been prepared manually by the user and taken through the sample preparation procedure. One matrix spike is used per sample batch and is assumed to be in the third sample position of the batch. This facility is available in the CONCENTRATION mode of calibration.

(E) MATRIX SPIKE CONCENTRATION XXXX

The SPIKE ADDED concentration for the MATRIX SPIKE.

Valid range 0.001 to 9999, default 1.000.

3.6 Replicate %RSD Test

This facility automatically tests the %RSD or coefficient of variation, of all solutions except the BLANK, against an action limit. The test is valid in all calibration modes.

(E) REQUIRED DETECTION LIMIT XXXX

If the calculated concentration of any analysed solution is greater than this limit the %RSD test will be applied. If the result is less than this limit the %RSD test will not be applied. Valid range 0.001 to 9999, default setting 1.000.

(A) REPLICATE %RSD LIMIT (%) XX

Enter the required action limit for the %RSD. The calculated %RSD (coefficient of variation) for the current solution will be tested against this limit. If this field is set to '100' the facility will be turned OFF. Valid range 0 to 100, default setting 20.

3.7 Correlation Coefficient, 'r'

QCP provides four goodness of fit parameters for the linear least squares regression line of best fit for the STANDARD ADDITIONS calibration mode.

(A) CORRELATION COEFFICIENT (r) XXXX

This field allows the user to enter a limit test for the regression coefficient, 'r'. Valid range 0.000 to 1.000, default setting 0.995.

The regression coefficient is calculated as recommended by the US EPA:

$$r = \frac{((N*SUM(Xi*Yi))-(SUM(Xi)*SUM(Yi)))}{(((N*SUM(Xi^2))-(SUM(Xi))^2)^{0.5})*((N*SUM(Yi^2))-(SUM(Yi))^2)^{0.5}}$$

QA/QC

TECHNITROL EXPERTISE INC.
TORONTO, ONTARIO

(full document available upon request)

TECHNITROL EXPERTISE INC.

Technitrol Experitise Inc.

Quality Control Manual

Version 1   Revision 0

Issue: September 4, 1990

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1-2 Revision Status
1-3 Statement of Certification
1-4 Distribution List
1-5 Organization
1-6 Laboratory Facilities and Equipment
1-7 Manual Review

2. SYSTEM FUNCTION DESCRIPTIONS

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2-2 Document Control and Technical Library
2-3 Control of Laboratory Equipment
2-4 Quality Assurance Program
2-5 Corrective Action
2-6 Quality Records
2-7 Management Quality Audit
2-8 Subcontractor Quality Surveillance

3. APPENDICES

Appendix A Facsimiles of Technitrol Expertise forms currently in use.

TECHNITROL EXPERTISE INC.

SUBJECT: **FOREWORD**

Technitrol Expertise Inc. is a laboratory that has been engaged in chemical, physical, and environmental testing since 1983.

Means are provided to ensure detection of discrepancies and for timely corrective action in all areas affecting contractual requirements and professional services rendered.

In extension to the Quality Control Manual, Quality Control Procedures and Testing Procedures are provided to achieve the quality objectives of the Company.

Quality Policy

It is the policy of Technitrol Expertise to provide the customer with an accurate assessment of the work performed, and that all services will meet the quality expectations of its customers.

It is the policy of the company to continue to meet the demands of today by employing highly qualified professionals applying well maintained equipment, capable of performing to the needed specifications and tolerances and maintaining a high and consistent standard of quality.

In order to achieve the intent of this policy, we have designed and developed this Quality Control Program that will ensure a coordinated effort throughout all phases of the organization and will reflect the requirements, whenever such a program is required by contract, by the jurisdiction, or by both.

SUBJECT: FOREWORD

Implementation of the Policy

This manual describes the company's quality system that will ensure that the necessary procedures and requirements are followed as and when applicable.

The Q.C. Manager is the appointed Quality Representative of the company to deal with customers and the jurisdiction in relation to service and subcontract quality and quality program requirements.

The quality control program will reflect the basic requirements of the following:

a. ASME Boiler and Pressure Vessel Code sections III, VIII and IX.

b. ASTM Specifications as applicable.

c. CSA Standards as applicable.

d. DND Specification D-QA-001-002/SF-001.

e. NIOSH Standards as applicable.

f. EPA Standards as applicable.

g. Ontario Ministry of the Environment (MOE) Standards as applicable.

h. USP Standards as applicable.

i. BP Standards as applicable.

j. CGSB standards as applicable.

k. Customer specifications and/or other specifications as required by the customer.

SUBJECT: REVISION STATUS

We hereby certify that this manual has been prepared and revised as necessary to reflect the current quality program of the company.

Sections affected: ____________________

____

____

____

____

____________________ ____________________

Q.C.Manager Date

TECHNITROL EXPERTISE INC.

SUBJECT: **STATEMENT OF CERTIFICATION**

We hereby certify that this Quality Control Manual accurately describes the policies and objectives of Technitrol Expertise with respect to its Quality Program.

Prepared by: 31/8/90
David Gordon Q.C. Manager Date

Approved by: Barbara J. Kovensky Aug 31, 1990
Barbara Kovensky, Vice-President Date

SUBJECT: **STATEMENT OF CERTIFICATION**

Manual No. 1

Issued To: Technitrol-Expertise
In house copy

Date: 31/8/90

This volume of the Quality Control Manual is issued to the individual listed above, who will be responsible for the incorporation of the revisions as they are furnished. All revisions will be forwarded to the registered manual holder with a transmittal form to be returned to the undersigned indicating receipt and incorporation of the revisions.

All further inquiries concerning the Quality Control Manual shall be directed to:

Quality Control Manager
Technitrol Expertise Inc.
637 Petrolia Road,
Downsview, Ontario,
M3J-2X8

Q.C. Manager

This manual is intended for the use of the volume holder and shall not be reproduced without written consent of the above.

APPENDIX #3

LEGEND FOR ANALYTICAL RESULTS

Attrition Scrubbing Trials

L= liquid
D= duplicate

different chemical group trials= 2,3
4,5,6
7,8,9
10,11
13,14,16
18
19,20,21
23,24,25
27,28,29

TW= tap water

TERRA CHEM

Environmental Analysts & Consultants

81 Keefer Road, Thorold, Ontario, Canada L2V 4M8 (416) 227-4156

Alt-2RS

Mon, Mar 30, 1992 6:28 PM

	#	Fe	Zn	Mn	Cu	Ni	Al	#	V
1	C2L	0.259	0.004	3.885	0.013	0.034	0.014	C2L	0.033
2	C2LD							C2LD	
3	C3L	6.575	0.033	5.242	0.041	0.161	1.322	C3L	0.011
4	C4L	14.080	0.805	7.273	0.691	1.245	22.164	C4L	0.200
5	C5L	10.086	0.148	2.789	0.217	0.344		C5L	
6	C6L	11.990	0.223	6.085	0.230	0.721	6.333	C6L	0.056
7	C7L	1.822	0.042	5.587	0.043	0.280	0.933	C7L	0.011
8	C7LD							C7LD	
9	C8L	0.794	0.022	3.823	0.027	0.138	0.544	C8L	0.044
10	C8LD							C8LD	
11	C9L	1.896	0.043	4.594	0.068	0.182	1.411	C9L	0.033
12	C10L	0.850	0.049	9.400	0.043	0.732	0.578	C10L	0.089
13	C10LD							C10LD	
14	C11L	10.049	0.204	11.100	0.242	1.405	5.811	C11L	0.133
15	RS13L	26.130	0.772	6.940	0.547	0.979	20.465	RS13L	0.078
16	RS14L	2.400	0.057	4.931	0.048	0.298	1.189	RS14L	0.022
17	RS14LD	2.668	0.057	5.343	0.053	0.312	2.877	RS14LD	0.100
18	RS16L	1.750	0.029	4.840	0.033	0.189	0.811	RS16L	0.078
19	RS16LD	1.722	0.027	4.835	0.030	0.179	0.889	RS16LD	0.044
20	RS18L	0.134	0.006	0.442	0.011	0.022	0.222	RS18L	0.033
21	RS18LD	0.144	0.003	0.367	0.004	0.014	0.133	RS18LD	0.022
22	RS19L	0.735	0.029	3.901	0.031	0.137	0.667	RS19L	0.056
23	RS19LD	0.779	0.031	4.227	0.037	0.134	0.778	RS19LD	0.078
24	RS20L	4.372	0.082	4.071	0.106	0.240	3.122	RS20L	0.056
25	RS21L	0.477	0.018	3.154	0.017	0.111	0.278	RS21L	0.033
26	RS21LD	0.456	0.011	2.984	0.014	0.099	0.167	RS21LD	0.022
27	RS23L	3.515	0.057	5.009	0.051	0.169	2.022	RS23L	0.078
28	RS23LD	3.916	0.060	5.109	0.058	0.189	2.278	RS23LD	0.089
29	RS24L		0.382	5.793	0.320	0.674	2.011	RS24L	0.044
30	RS25L	3.606	0.068	4.650	0.061	0.223	2.566	RS25L	0.100
31	RS25LD	3.882	0.071	4.965	0.069	0.234	2.800	RS25LD	0.100
32	RS27L	2.748	0.074	11.050	0.070	0.948	1.444	RS27L	0.089
33	RS27LD	2.663	0.074		0.071	0.958	1.500	RS27LD	0.089
34	RS28L	0.868	0.042	9.300	0.051	0.825	0.689	RS28L	0.111
35	RS28LD	0.739	0.042		0.052	0.769	0.789	RS28LD	0.111
36	RS29L	2.591	0.113	10.320	0.097	1.177	2.411	RS29L	0.011
37	RS29LD	2.736	0.119		0.102	1.181	2.611	RS29LD	0.011
38	TW	0.313	0.056	0.745	0.603	0.096	0.167	TW	0.011
39	TW-D	0.252	0.052	0.780	0.633	0.093	0.133	TW-D	0.022

Alt-2RS Mon, Mar 30, 1992 6:28 PM

	Cr	Cd	Pb	Mg
1	0.029	0.001	0.006	40.329
2				41.996
3	0.037	0.001	0.006	47.717
4	0.131	0.001	0.081	50.773
5	0.082	0.001	0.006	46.051
6	0.284	0.004	0.110	44.718
7	0.074	0.002	0.006	42.107
8				37.385
9	0.067	0.001	0.006	36.941
10				35.719
11	0.112	0.003	0.030	41.051
12	0.133	0.002	0.006	54.106
13				54.883
14	0.333	0.004	0.064	52.217
15	0.650	0.004	0.314	50.106
16	0.183	0.002	0.128	44.162
17				36.274
18	0.083	0.001	0.018	39.885
19				
20	0.020	0.001	0.006	11.777
21				
22	0.061	0.001	0.006	35.441
23				
24	0.180	0.001	0.033	37.163
25	0.059	0.001	0.006	34.663
26				
27	0.140	0.001	0.014	39.996
28				
29			0.134	
30	0.167	0.001	0.018	40.052
31				
32	0.168	0.001	0.014	55.272
33				
34	0.153	0.001	0.006	54.772
35				
36	0.192	0.001	0.006	55.717
37				
38	0.028	0.001	0.006	9.277
39	0.022	0.001	0.006	9.221

LEGEND FOR ANALYTICAL RESULTS

Attrition Scrubbing Trials

TS (alone)= sediment characterization
TS (preceded by a letter/number sequence)= coloured top sediment layer in sample jars
S= solid
C= sample identification symbol
RS= sample identification symbol

different chemical group trials= 2
3,4
5
6,7
8
9
10,11

TERRA CHEM

Environmental Analysts & Consultants

81 Keefer Road, Thorold, Ontario, Canada L2V 4M8 (416) 227-4156

Alt-2aRS

Thu, Apr 2, 1992 10:17 AM

	#	Fe	Mg	Mn	Zn	Cu	Ni	Cr
1	C2S	208054.0	12707.0	4658.9	786.7	947.4	1936.1	3339.9
2	C2DS	223460.9	12135.0	4272.9	697.5	930.3	1968.2	3445.1
3	C3S	208401.3	11714.9	2903.8	568.9	814.6	1963.7	3748.0
4	C4S	191439.8	12231.4	2670.1	574.9	811.2	1929.6	3705.2
5	C5S	213880.0	11927.1	3705.7	638.7	874.9	1965.9	3757.5
6	C5DS	221157.9	12112.6	4009.6	679.4	864.7	2010.1	3807.6
7	C6S	219637.2	12046.5	4480.5	705.8	973.0	1890.8	3524.3
8	C7S	208417.5	12409.1	2893.1	579.0	896.3	2024.1	4162.3
9	C8DS	181346.9	16259.7	3599.8	566.9	920.5	2160.9	4587.2
10	C9S	194067.1	10886.9	3336.0	582.3	792.0	1702.0	3372.0
11	C10S	200653.0	12257.5	3710.8	671.6	883.4	1904.9	3968.3
12	C11S	195113.3	12849.0	2983.6	621.2	831.6	2005.3	4437.5
13	C11ds	194291.3	12734.9	2884.0	609.7	839.5	1919.9	3850.1
14	TS	193831.0	13138.2	2830.2	580.4	757.7	1678.5	3623.4
15								
16	RS21S	183958.0	13301.0	3197.0	696.0	886.0	1940.0	4352.0
17								
18	C9F	167500.0	22500.0	3668.0	1012.0	512.5	468.8	593.8
19	RS21TS	107582.0	16311.5	2349.2	377.5	319.7	569.3	988.9
20	RS2TS	237009.8	21813.7	3720.6	1272.1	1014.7	1808.8	3426.5
21	RS10TS	103031.6	13787.4	2206.8	611.7	614.6	1189.8	2387.5
22	RS11TS	76785.7	10912.7	2024.3	490.1	466.8	817.0	1282.2
23								
24	short-liq	786.2		101.5	23.3	0.2	35.5	12.8
25	long-liq	797.9	867.0	109.2	24.9	0.2	43.5	8.6
26	xlong-liq	930.6	719.0	133.2	35.7	0.5	54.3	7.8
27	xlong-Dliq	951.5		134.3	35.7	0.6	54.3	8.0
28								
29	short-solid	236077.1	11387.5	2899.0	480.9	764.6	1099.3	3319.2
30	long-solid	219575.8	10446.5	3397.8	479.8	810.4	1087.2	3872.2
31	xlong-solid	218633.0	11320.4	3138.9	472.8	746.6	1142.9	3899.7
32	short-D solid	224600.2	11278.3	2605.0	505.9	764.7	1173.3	3737.9
33	long-D solid	250645.2	10917.3	2268.1	500.5	748.0	1239.9	4538.3
34	xlong-D solid	226163.0	11436.1	2616.1	518.8	847.5	1183.4	3694.1
35	SCR	561725.1	680.3	8777.1	44.6	727.7	1318.0	3679.8
36	NBS 2704	39248.9	12968.3	560.5	402.8	97.7	77.4	145.3

Alt-2aRS

Thu, Apr 2, 1992 10:17 AM

	Pb	Cd
1	238.3	
2	259.4	
3	224.3	
4	230.4	
5	233.8	
6	241.4	
7	230.7	
8	231.1	
9	229.8	
10	205.9	0.9
11	237.4	
12	256.4	1.8
13	242.6	1.7
14	224.9	
15		
16	265.0	1.7
17		
18	293.8	25.0
19	140.6	1.6
20	311.3	22.1
21	251.7	2.9
22	154.8	2.0
23		
24	0.3	0.0
25	0.8	0.0
26	1.6	0.0
27	1.4	
28		
29	274.9	2.7
30	264.1	1.7
31	271.1	1.2
32	263.6	1.7
33	270.6	1.5
34	252.2	1.3
35	425.9	
36	151.1	3.4

APPENDIX #4

TERRA CHEM
Environmental Analysts & Consultants
81 Keefer Road, Thorold, Ontario, Canada L2V 4M8 (416) 227-4156

Alt-3RS

Mon, Mar 30, 1992 6:30 PM

	#	Fe	Zn	Mn	Cu	Ni	Cr	#
1	TW	0.313	0.056	0.745	0.603	0.096	0.028	TW
2	TW-D	0.252	0.052	0.780	0.633	0.093	0.022	TW-D
3								
4	short-liq	786.200	23.300	101.500	0.225	35.520	12.820	short-liq
5	long-liq	797.900	24.900	109.200	0.174	43.510	8.580	long-liq
6	xlong-liq	930.600	35.700	133.200	0.549	54.280	7.830	xlong-liq
7	xlong-Dliq	951.500	35.700	134.300	0.562	54.280	7.980	xlong-Dliq
8								
9	short-solid	236077.063	480.886	2898.968	764.563	1099.312	3319.175	short-solid
10	long-solid	219575.763	479.803	3397.758	810.398	1087.167	3872.246	long-solid
11	xlong-solid	218633.029	472.839	3138.878	746.630	1142.942	3899.683	xlong-solid
12	short-D solid	224600.162	505.856	2605.008	764.742	1173.263	3737.884	short-D solid
13	long-D solid	250645.161	500.504	2268.145	747.984	1239.919	4538.306	long-D solid
14	xlong-D solid	226163.039	518.850	2616.110	847.454	1183.443	3694.131	xlong-D solid
15	SCR	561725.128	44.643	8777.105	727.679	1318.027	3679.847	SCR
16								
17	NBS 2704	39248.853	402.810	560.493	97.668		145.260	NBS 2704

Alt-3RS

Mon, Mar 30, 1992 6:30 PM

	Cd	Pb	Mg	As	Se
1	0.001	0.006	9.3		
2	0.001	0.006	9.2		
3					
4	0.012	0.350		2.000	0.090
5	0.020	0.770	867.0	1.900	0.096
6	0.030	1.590	719.0	2.100	0.090
7		1.430			
8					
9	2.731	274.918	11387.5	23.280	1.180
10	1.739	264.092	10446.5	24.640	1.580
11	1.190	271.118	11320.4	20.040	1.270
12	1.666	263.603	11278.3	21.800	1.270
13	1.512	270.562	10917.3	20.880	1.500
14	1.312	252.155	11436.1	19.400	1.000
15		425.925	680.3	3.720	0.660
16					
17	3.440	151.055	12968.3	24.600	1.340

APPENDIX #5

LEGEND FOR ANALYTICAL RESULTS

Trial no. 1 and no. 2, Flotation Cell

L= liquid
D= duplicate
S= solids
V= vacuum filtered

little froth, trial no. 1
5,6= froth
7= aggitation zone
8= base sediment remaining in flotation cell

substantial froth, trial no. 2
9= froth at beginning
11= base sediment remaining in flotation cell
12= froth at end

TERRA CHEM
Environmental Analysts & Consultants
81 Keefer Road, Thorold, Ontario, Canada L2V 4M8 (416) 227-4156

Alt-4RS

Mon, Mar 30, 1992 6:31 PM

	#	Fe	Mn	Zn	Cu	Ni	Cr	#	Mg
1	5L	125.3	19.32	0.620	0.023	3.786	0.007	5L	30.4
2	6L	96.8	15.32	0.285	0.021	3.145	0.001	6L	31.4
3	7L	59.0	14.49	0.189	0.013	3.082	0.001	7L	27.6
4	9L	53.6	6.35	0.919	0.013	2.121	0.101	9L	19.3
5	9LD						0.086	9LD	16.9
6	12L	46.6	6.63	0.434	0.010	2.290	0.009	12L	25.3
7	12LD	46.4	6.46	0.439	0.010	2.257		12LD	
8	5LV	123.5	19.75	0.608	0.021	3.781	0.018	5LV	28.7
9	6LV	87.0	13.88	0.293	0.014	3.160	0.011	6LV	20.9
10	7LV	49.3	14.87	0.169	0.010	2.567	0.002	7LV	26.0
11	9LV	52.4	6.40	1.041	0.030	2.216	0.052	9LV	21.5
12	9LVD						0.044	9LVD	19.8
13	12LV	40.8	6.92	0.476	0.009	2.436	0.010	12LV	33.0
14	12LVD	40.7	6.80	0.473	0.009	2.446		12LVD	
15									
16	5S	88119.8	2685.95	938.017	836.777	1448.347	378.099	5S	14927.7
17	6S	86930.2	3774.71	1048.810	814.845	1435.054	492.840	6S	13200.9
18	7S	105047.2	2469.22	802.216	796.061	1619.819	453.426	7S	14464.5
19	8S	292186.9	5607.13	296.013	1022.956	2464.760	624.648	8S	9242.9
20	9S	103272.0	3058.28	958.078	912.065	1510.225	395.910	9S	11912.1
21	11S	239126.1	5543.70	473.863	985.634	2293.496	1197.127	11S	12779.3
22	12S	91106.1	2955.01	1087.495	960.586	1548.700	359.162	12S	12205.9
23	12SD	83871.6	2913.81	1162.697	974.970	1429.148	405.632	12SD	11283.8
24	5SV	96011.5	2473.27	1056.743	935.444	1595.395	442.434	5SV	13980.3
25	6SV	106219.7	2993.74	929.927	858.239	1559.976	462.137	6SV	12308.2
26	7SV	117909.2	4386.14	1362.439	939.019	1531.605	492.504	7SV	13897.9
27	9SV	103626.9	2856.72	1125.947	1031.287	1597.250	448.784	9SV	10990.4
28	12SV	94973.2	2923.36	1185.620	996.086	1555.418	455.707	12SV	14225.4
29	12SVD	95914.4	2837.55	1160.506	1012.646	1538.911	452.237	12SVD	9883.3
30									
31	2704-1	36024.5	593.17	469.799	92.775	34.540	152.685	2704-1	10511.3
32	2704-2	39248.9	560.49	402.810	97.668	39.000	145.260	2704-2	12968.3
33	actual	41100.0	555.00	438.000	98.600	44.100	135.000	actual	12000.0

Alt-4RS

Mon, Mar 30, 1992 6:31 PM

	Ag	Pb	Cd	As	Se	Be	Sb
1	0.028	0.065	0.018	0.017	0.082	0.004	0.38
2	0.024	0.051	0.015	0.007	0.071	0.002	0.37
3	0.021	0.052	0.015	0.005	0.067	0.002	0.32
4	0.012	0.090	0.019	0.025	0.054	0.003	0.18
5	0.012	0.087	0.021	0.027	0.055	0.004	0.18
6	0.012	0.041	0.017	0.007	0.039	0.003	0.16
7							
8	0.025	0.049	0.017	0.011	0.088	0.004	0.33
9	0.024	0.055	0.012		0.069	0.005	0.32
10	0.021	0.056	0.007		0.062	0.004	0.41
11	0.013	0.088	0.002	0.023	0.047	0.001	0.21
12	0.013	0.083	0.003	0.025	0.045	0.001	0.25
13	0.013	0.032	0.001	0.007	0.044	0.014	0.25
14							
15							
16	6.921	384.401	2.583	35.114	1.763	1.756	5.06
17	6.656	349.839	2.521	31.616	1.634	1.714	6.76
18	6.668	337.300	1.334	21.779	0.877	1.641	6.16
19	5.236	157.471	0.101	9.968	1.605	1.108	5.44
20	7.055	397.648	1.534	33.180	1.748	1.738	5.01
21	5.088	188.348	0.100	9.876	1.647	1.197	6.68
22	7.738	416.839	2.579	33.822	1.984	1.651	5.47
23	7.368	396.750	3.129	36.304	2.019	1.615	4.74
24	6.785	384.252	3.392	26.120	1.640	1.439	3.29
25	6.765	340.468	3.736	22.769	1.575	1.616	4.54
26	6.787	364.465	5.065	28.859	1.692	1.519	5.37
27	7.174	404.942	6.377	31.128	1.591	1.594	4.88
28	7.829	429.543	6.180	61.300	1.894	2.163	4.43
29	7.393	421.109	5.642	63.881	1.859	2.043	4.86
30							
31	2.171	166.107	2.961	21.713	1.081	1.974	3.36
32		151.055	3.440		1.282	2.156	3.65
33		161.000	3.450	23.400	1.120	2.065	3.80

LEGEND FOR ANALYTICAL RESULTS

Trial no. 3 and no. 4, Flotation Cell

L= liquid
D= duplicate
S= solids

13= sediment characterization

substantial froth, trial no. 3
14= froth at beginning
15= froth at end
16= base sediment left in flotation cell

substantial froth, trial no. 4
17= froth at beginning
18= froth at end
19= base slurry, no base sediment at end of trial

TERRA CHEM

Environmental Analysts & Consultants

81 Keefer Road, Thorold, Ontario, Canada L2V 4M8 (416) 227-4156

Alt-5RS

Mon, Mar 30, 1992 6:33 PM

	#	Fe	Mn	Zn	Cu	Ni	Cr	#	Mg
1	L14	0.09	3.38	0.059	0.010	0.392	0.011	L14	22.2
2	L14D	0.10	3.08	0.043	0.009	0.403	0.017	L14D	19.3
3	L15	0.18	2.82	0.025	0.009	0.301	0.015	L15	21.9
4	L15D	0.02	2.83	0.017	0.005	0.304	0.002	L15D	22.9
5	L17	0.05	0.21	0.013	0.002	0.070	0.010	L17	7.6
6	L17D	0.03	0.21	0.011	0.005	0.050	0.021	L17D	7.9
7	L18	0.02	0.15	0.083	0.006	0.073	0.004	L18	6.7
8	L18D	0.01	0.15	0.022	0.003	0.047	0.022	L18D	6.7
9	L19	0.07	0.13	0.021	0.005	0.055	0.032	L19	6.0
10	L19D	0.08	0.12	0.021	0.004	0.047	0.014	L19D	6.7
11									
12	13S	166252.07	2279.23	698.590	742.123	2236.733	6285.240	13S	11525.7
13	13SD	178067.42	2393.18	751.666	799.686	2296.158	6566.053	13SD	10829.1
14	14S	92543.68	1450.63	1081.877	655.221	1478.058	3304.551	14S	10453.1
15	14SD	86938.94	1400.12	1043.267	624.747	1421.351	2964.011	14SD	11878.3
16	15S	89098.33	1421.49	1183.443	695.686	1469.102	3527.983	15S	14156.6
17	16S	213890.03	2778.01	585.761	830.940	2731.842	10084.120	16S	11181.8
18	16SD	208032.85	2670.27	551.883	825.321	2551.082	7411.859	16SD	11017.6
19	17S	97653.14	1542.36	1044.153	624.503	1481.702	4492.840	17S	12708.8
20	17SD	77996.72	1524.01	1045.772	612.685	1399.836	3887.521	17SD	12879.7
21	18S	85687.96	1611.38	916.257	575.348	1415.848	3451.065	18S	11670.8
22	19S	111929.75	1681.47	1003.230	681.268	1683.488	4401.494	19S	14675.0
23	19SD	107069.46	1627.62	995.684	669.955	1623.510	4657.830	19SD	14231.4
24									
25	2704	42788.27	601.65	436.922	106.469	42.186	133.588	2704	10084.4
26	certified	41100.00	555.00	438.000	98.600	44.100	135.000		12000.0

Alt-5RS

Wed, Apr 1, 1992 3:42 PM

	Ag	Pb	Cd	As	Se	Be	Sb	Al
1	0.006	0.012	0.003	0.001	0.012	0.003	0.27	0.050
2	0.006	0.010	0.002	0.002	0.012	0.003	0.14	0.070
3	0.006	0.012	0.003	0.002	0.010	0.002	0.28	0.300
4	0.006	0.013	0.002	0.002	0.011	0.002	0.21	0.200
5	0.001	0.004	0.002	0.008	0.001	0.001	0.06	0.080
6	0.002	0.002	0.003	0.004	0.001	0.002	0.13	0.100
7	0.002	0.002	0.001	0.005	0.001	0.002	0.05	0.500
8	0.001	0.002	0.001	0.006	0.001	0.001	0.05	0.300
9	0.001	0.004	0.001	0.004	0.001	0.004	0.02	0.010
10	0.001	0.003	0.001	0.006	0.001	0.001	0.02	0.010
11								
12	4.871	260.365	2.280	21.092	1.085	1.451	7.36	50766.998
13	4.998	284.300	1.764	27.979	1.009	1.470	6.57	58800.470
14	5.486	335.331	3.048	21.892	1.331	1.829	3.86	61966.680
15	5.661	336.231	2.527	25.627	1.577	1.820	3.84	54266.074
16	5.538	349.106	2.623	23.951	1.244	1.943	4.37	54391.761
17	5.027	225.687	0.923	15.952	0.769	1.539	6.87	33381.206
18	4.607	230.569	1.703	14.173	0.731	1.402	7.01	37139.423
19	4.873	297.235	2.586	27.595	1.144	1.889	3.58	51173.429
20	5.131	306.548	2.668	24.682	1.344	1.847	5.34	39798.851
21	4.812	296.376	2.559	18.786	0.983	2.048	3.07	47481.572
22	5.248	312.979	2.826	21.245	1.151	1.817	6.56	41522.002
23	5.240	300.658	2.466	15.875	0.997	1.850	5.75	62741.471
24								
25	2.109	157.091	2.812	25.462	1.111	2.009	3.72	
26	2.171	161.000	3.450	23.400	1.120	2.065	3.80	

APPENDIX #6

LEGEND FOR ANALYTICAL RESULTS

Final Flotation Cell Trials

OS= sediment characterization
AS= attrition scrubber
LS= liquid slurry -- solids
BS= base sediment left in flotation cell after trial run
5= 5 min. overflow -- solid
10= 10 min. overflow -- solid
LLS= liquid slurry -- liquid
FCW= float cell water
5L= 5 min. overflow -- liquid
10L= 10 min. overflow -- liquid

TERRA CHEM

Environmental Analysts & Consultants

81 Keefer Road, Thorold, Ontario, Canada L2V 4M8 (416) 227-4156

Alt-6RS Mon, Mar 30, 1992 6:35 PM

	#	Fe	Mn	Zn	Cu	Ni	Cr	#	Mg
1	CS	170272.44	2022.84	731.32	685.347	1884.065	4967.452	CS	10259.836
2	CSD	165954.42	1967.59	664.22	672.873	1858.618	4759.168	CSD	13610.541
3	AS	158094.10	2402.91	993.02	708.333	1809.809	6166.316	AS	14142.215
4	ASD	166700.82	2072.75	708.91	688.883	1885.502	5056.416	ASD	13762.285
5	LS	83333.33	1579.39	1102.53	617.834	1284.398	3043.649	LS	12602.252
6	LSD	84368.74	1486.22	1000.75	606.413	1286.924	2756.349	LSD	12668.517
7	BS	279370.77	2890.03	461.22	813.272	2398.347	7499.582	BS	18959.120
8	BSD	283457.25	3035.04	474.77	836.571	2512.872	8059.913	BSD	20134.294
9	5	74744.38	1568.76	1392.33	555.368	1084.663	2010.706	5	12791.268
10	5D	72213.18	1361.63	1040.89	561.076	1599.369	2316.467	5D	15412.978
11	15	76642.34	1252.94	1008.21	560.979	1127.991	2207.583	15	14315.501
12	15D	74190.28	1459.97	1288.77	595.344	1074.545	1754.211	15D	15765.405
13									
14	LLS	0.00	0.84	0.10	0.009	0.178	0.009	LLS	0.137
15	LLSD	0.00	0.83	0.10	0.005	0.175	0.001	LLSD	0.126
16	FCW	0.03	0.01	0.10	0.152	0.001	0.027	FCW	0.064
17	FCWD	0.04	0.01	0.10	0.149	0.001	0.029	FCWD	0.062
18	5L	0.00	0.88	0.06	0.005	0.118	0.031	5L	0.110
19	5LD	0.00	0.88	0.06	0.009	0.143	0.022	5LD	0.104
20	15L	0.00	0.79	0.03	0.006	0.104	0.022	15L	0.090
21	15LD	0.00	0.80	0.03	0.007	0.112	0.011	15LD	0.120
22									
23	NBS	39442.39	560.46	468.18	94.821		130.576	NBS	10932.993
24	certified	41100.00	555.00	438.00	98.600	44.100	135.000		12000.000

Alt-6RS

Mon, Mar 30, 1992 6:35 PM

	Ag	Pb	Cd	As	Se	Be	Sb	Al
1	6.210	267.328	3.305	14.976	0.911	1.402	6.76	48027.60
2	5.698	255.495	2.646	14.050	1.521	1.425	6.46	41396.85
3	5.981	280.403	3.090	10.056	1.626	1.396	7.53	53645.18
4	6.148	254.508	4.406	14.836	1.499	1.434	7.43	43369.98
5	6.245	333.640	4.505	14.406	1.413	1.740	5.17	61100.45
6	6.112	321.844	4.208	16.493	1.385	1.804	4.91	51074.83
7	5.675	169.853	3.286	9.470	1.195	1.195	7.37	29622.24
8	5.483	176.952	3.996	7.959	1.045	1.115	8.88	28704.28
9	6.237	338.548	4.499	13.722	1.372	1.738	4.45	41395.54
10	6.408	352.624	4.373	13.377	1.439	2.034	4.27	49154.29
11	6.792	355.637	3.751	12.437	1.394	1.622	5.12	65078.65
12	6.680	363.664	4.352	15.668	1.535	1.518	5.16	70325.85
13								
14	0.001	0.005	0.001	0.001	0.004	0.001	0.05	0.01
15	0.002	0.005	0.001	0.001	0.003	0.001	0.05	0.03
16	0.001	0.009	0.001	0.001	0.001	0.001	0.05	0.17
17	0.001	0.009	0.001	0.001	0.001	0.001	0.05	0.23
18	0.003	0.002	0.001	0.006	0.002	0.001	0.10	0.04
19	0.002	0.004	0.001	0.003	0.003	0.001	0.25	0.11
20	0.003	0.006	0.001	0.003	0.002	0.001	0.21	0.11
21	0.002	0.005	0.003	0.006	0.003	0.001	0.33	0.06
22								
23	2.390	147.013	3.088	24.255	1.272	1.793	3.69	57232.22
24	2.171	161.000	3.450	23.400	1.120	2.065	3.80	61100.00

TECHNITROL EXPERTISE INC.

637 PETROLIA ROAD, DOWNSVIEW, ONTARIO M3J 2X8
TEL.: (416) 665-2134 FAX: (416) 665-9251

March 23, 1992

Altech Environmental Consulting Ltd.
225 Sheppard AVenue West
Toronto, Ontario M2N 1N2

ATTENTION: Mr. Rod Shaver

RE: Samples (7) of Solid and Liquid Wastes for Analysis
Our Project No: 92-0242

On March 17, 1992, we received in our laboratory seven (7) samples of solid and liquid Wastes for analysis for Oil and Grease plus total organic carbon as per our client's specifications.

The results were as follows:-- (attached)

B.J.Kovensky
B.A.Chem.
M.Sc.
P.Chem.
C.Chem.

attached

Page 2.
March 23, 1992
TEchnitrol to Altech
Lab No. 92-0242

SAMPLE IDENTIFICATION	RESULTS, ppm (Unless otherwise stated)	
	Oil and Grease	Total Organic Carbon
1. 5 minute overflow (liquid)	3832.5	1679
2. Attrition Scrubber Sediment (Solid)	22,399.2	17.14%
3. Water from flotation Cell (liquid)	24.2	5.1
4. Orig. Sediment (solid)	24,441.2	21.2%
5. 15 Minute Overflow (Liquid)	3696	1720
6. Liquid Slurry (Liquid)	3603.3	1692
7. Bottom Sediment No. 2 (solid)	21,455.8	14.7%

APPENDIX #7

Wastewater Technology Centre
operated by RockCliffe Research Management Inc.

Centre Technique des Eaux Usées
dirigé par Gestion de Recherche RockCliffe Inc.

867, chemin Lakeshore Road
P.O. Box / C.P. 5068, Burlington
Ontario, Canada, L7R 4L7
(416) 336-4855
Fax/Fac (416) 336-4765

File-N/Reference
Project 28896

28 July 1992

Mr. Alex R. Keen, President
Altech Environmental Consulting Ltd.
225 Sheppard Avenue West,
Willowdale, Ontario
Canada M2N 1N2

Dear Mr. Keen:

Re : Altech Bench-Scale Treatability Studies - WTC Laboratory Audit

During the "audit run" of the treatability studies performed by Altech on pre-screened Welland River sediment I obtained seven split samples. These samples were submitted to the Wastewater Technology Centre (WTC) analytical laboratory for analysis of solids (the samples ranged from water to approximately 50 % solids), total organic carbon and a number of metals (by ICP). I have received the results of these analyses and have compared them with the equivalent results reported in Altech's draft report (Appendix #6) describing the treatability studies. The WTC results are enclosed with this letter. Both this letter and the results should be included within the final report.

With the exception of Sb, Cd, Ag, As, Zn and Se the results from the Altech and WTC laboratories for the metals compared well. Of the exceptions, Sb, As and Zn are the only metals of concern, as the concentrations reported for each metal by each laboratory were "high" while differing by at least a factor of 2.

For the remaining metals analyzed, differences in concentrations reported were usually less than 25%. For most metals, Altech reported higher concentrations than the WTC's laboratory. It is conceivable that this difference is due to operational procedure.

In all cases, as the concentrations reported by the two laboratories differed by constant factors, the statements made by Altech regarding <u>trends</u> in the system response will still hold true.

Sincerely,

Paul Bucens
Contaminated Sediment Treatment Technology Program
Site Remediation Division

enclosure

cc. C. Wardlaw

altec2.pgb

PRETREATED WELLAND RIVER SEDIMENT SAMPLE (Screened to sub 75um in ACRES pilot)
Results from ALTECH treatability testing (WTC Split Samples)
Collected: 16 March 92 File:WRWTC6
Submitted: 16 March 92
Received: 27 July 92

WTC id	Original Sediment	Attrition Scrubber Sediment	Bottom Sediment	Liquid Slurry Solid	Liquid Slurry Liquid	5 Minute Overflow Solid	5 Minute Overflow Liquid	15 Minute Overflow Solid	15 Minute Overflow Liquid	Water from Flotation Cell
ALTECH id	OS	AS	BS	LS	LLS	5	5L	15	15L	FCW
TSS ug/gm				20040		23480		26830		2
w.c. %	55.6	53.6	58.3	96.9		97.0		96.7		100
pH										
TOC %C	7.10	7.40	6.41	11.8		11.6		11.6		
TOC C ug/gm										4.8
O&G										
	ug/g	ug/g	ug/g	ug/g	ug/mL	ug/g	ug/mL	ug/g	ug/mL	ug/mL
Sb	98	115	99	72	T.10	82	T.10	61	T.10	T.05
As	35.3	33.7	31.4	27	N/A	29.2	N/A	25.2	N/A	T.0004
Ba	257	294	226	248	0.06	303	0.08	276	0.07	0.47
Cd	W	W	W	W	W	W	W	W	W	W
Ag	W	W	W	W	W	W	W	W	W	W
Zn	500	559	406	558	T.04	675	T.04	533	0.09	0.1
Se	0.384	0.475	0.292	0.401	N/A	0.502	N/A	0.412	N/A	T.0005
Cr	2489	5000	2863	1495	T.03	1462	T.03	996	0.05	T.010
Co	150	178	146	90	W	92	W	63	W	W
Fe	20 %	23 %	21 %	7.5 %	1.2	7.8 %	1.6	6.5 %	3.2	1.4
Ni	1461	1722	1440	860	T.04	952	T.04	693	0.05	T.02
Na	3781	4440	3481	3852	31	4344	33	3280	31	50
Sn	3755	4715	3918	1919	0.25	2103	0.27	1244	0.3	T.06
Al	4.1 %	4.6 %	3.5 %	4 %	1.5	4.8 %	1.8	4.7 %	3	13.5
Be	3.1	3.8	3.2	1.9	W	2.1	W	1.5	W	W
Cu	602	687	595	415	0.05	460	T.03	373	0.05	0.17
Mg	1.5 %	1.6 %	1.3 %	1.2 %	13	1.5 %	14	1.3 %	14	8.4
S (%)	0.685	0.697	0.862	0.96	N/A	1.07	N/A	1.002	N/A	0.004 ug/mL
Tl	5771	5140	4611	1470	W	1443	W	978	W	W
Mn	2381	2652	2462	867	0.21	993	0.5	743	1.05	T.02

T =< MDL
W = NOT DETECTABLE
N/A = NOT AVAILABLE
Blanks were not analysed