CAMECO STAR **LAKE**

STABILITY OF COPPER IN STAR LAKE MUSKEGS

FINAL REPORT

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EXECUTIVE SUMMARY

The effluent treatment system for the Star Lake gold operation utilized a sprinkling system in two muskeg areas, contained by two dykes. This novel approach to treatment performed effectively removing both Cu and CN from the effluent. The waste management area of the Star Lake operation is to be decommissioned. Studies evaluating the performance of the system and the fate of the copper in the muskeg indicated that two requirements were needed before determining the decommissioning approach for the site. Firstly, the spacial distribution of copper in the two muskegs had to be ascertained. Secondly, the environmental stability of the copper had to be evaluated. The literature was consulted and samples were collected from the muskeg to address these objectives.

The literature on uses of wetlands for mine effluent treatment indicates that they are effective for removal of copper (Cu). The main removal mechanism which is believed to be working in these systems is microbial sulphate reduction taking place in anaerobic sediments. Confirmation that sulphate reduction and subsequent precipitation of CuS is taking place is wanting in the literature.

In the Star Lake muskegs, sulphate reduction is taking place. The Eh/pH conditions in the muskeg sediments, however, indicate that this microbial process cannot be the major process which has been removing the Cu from the sprayed effluent. The Eh/pH range of the Star Lake muskeg pore-water suggests that Cu might be precipitated as carbonates (malachite), oxides (tenorite) or hydroxides (cuprite). The literature indicates ion exchange, organic complexation and adsorption are likely the dominant Cu removal processes, under the prevailing conditions.

Copper is reported to stay in suspension when present in colloidal form (identified in the tailings effluent) and thus not available to form a stable precipitate. Colloidal Cu forms together with Fe(III) oxyhydroxide. In the Star Lake muskegs Cu is cycled

seasonally, along with reductive dissolution of and re-precipitation of Fe(III) oxyhydroxide in the prevailing Eh/pH range. Copper in the muskegs is also likely bound to peat by both cation exchange and complexation. In the literature, reports on Cu removal capacity of peat ranges between 14,000 mg.kg⁻¹d.w. to 24,300 mg.kg⁻¹d.w.

In the present study, the Cu concentrations in T-2 muskeg in the directly sprayed zone were around 2% Cu (20,000 mg.kg⁻¹d.w.), close to the saturation concentrations reported for ion exchange and complexation. The T-2 areas below the spray zone have Cu concentrations ranging from 78 mg.kg⁻¹d.w. to 15,100 mg.kg⁻¹d.w. of Cu. In the T-3 area, Cu concentrations in the directly sprayed zone were lower with a concentration range of 103 mg.kg⁻¹d.w. to 2,790 mg.kg⁻¹d.w. and lower again in the unsprayed zone T-3, 47 mg.kg⁻¹d.w. to 921 mg.kg⁻¹d.w.. Such concentration ranges are reported for sediments in Cu mineralized areas. In the Star Lake area, the control muskeg Cu concentrations were 15 mg.kg⁻¹d.w. to 38 mg.kg⁻¹d.w., which are lower as can be expected since the area has no copper mineralization. The utilization of the muskeg as a treatment system elevated the areas into the mineralized concentration range.

Copper load for the sprayed and non-sprayed zones of the muskeg areas were estimated by multiplying the mean Cu concentrations in solid samples from a zone by the estimated volume of 'influenced' sediment in that zone (depth x area of zone). The loadings of Cu estimated in this manner are higher than the load estimated using Blindman Lake effluent concentrations and monitored flow rates from the sprinkling system. This provides evidence, that the copper was retained in the muskeg area and that effluent concentration limits were met due to the wetland removal processes, not due to dilution in the drainage basin.

The spacial distribution of the total Cu load leads to the conclusion that 85% of the Cu is removed in the T-2 muskeg area. Of this percentage, **45%** remained in the direct

spray zones of T-2. It can be concluded that the forest soil has retained most of the Cu and it has remained there since its application with the sprinkler system in 1989.

The literature suggests that the dominant retention mechanism of copper in the Star Lake muskeg is postulated as organic complexation and adsorption of colloids or free Cu⁺². In order to confirm or refute the suggested retention mechanisms of Cu in the muskeg and the sprayed forests soils, samples were examined with energy dispersive X-ray spectroscopy (EDS). One hundred EDS scans were investigated. This examination confirmed that Cu is removed from the water by cation exchange and complexation. Formation of stable precipitates is also possible based on the Eh/pH measurements obtained in the muskegs.

In addition **to** the EDS investigation, sequential extractive methodology was applied to the muskeg samples. This procedure removes in sequence the Cu held by the material as ion-exchange and complexation, both relatively mobile fractions, followed by the extraction of readily soluble precipitates and finally insoluble (stable) precipitates. The forest soil in the direct sprayed zone in T-2 has retained 60 % to 70 % of Cu in an organic complexed form. The exchangeable fraction of Cu in this zone was less then 0.01 %. Four (4) % to 6 % of Cu is present in the more stable precipitate form. The fraction of stable precipitates is higher in T-3, although only 20 % of the copper could be accounted for in the extractions, due to the low copper concentrations in the material.

Copper appears to be displaced from the material in the direct spray zone of T-2 by thawing. This results in increased copper concentrations during spring run-off **as** evidenced by the monitoring data for 1992, 1993 and 1994. For the remainder of the year, the effluent concentrations are below the authorized guideline concentration. Although the approach to effluent treatment was very effective in removing Cu, concentrating it in the forests soils, in future uses of this treatment approach, consideration should be given to optimize the formation of more stable Cu forms during

operation of the system. This study leads to the following conclusion. The effectiveness of the treatment for both CN and Cu was proven. The good accountability of the Cu load added to the system and its distribution within, suggest that utilization of muskeg as a treatment approach for gold mill effluents is an environmentally acceptable approach. With optimization, it represents a sustainable solution both for operation and decommissioning.

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1.0 Introduction

The Star Lake gold deposit was expected to have a relatively short life. The Star Lake Mill was commissioned in 1986 with a 200 t.p.d CIP mill. The tailings effluent contained Cu, which originates from the mineralisation associated with the gold, which is not extracted during the milling process. A high proportion of Cu in the tailings effluent was in colloidal suspension. To remove colloidal Cu conventionally, ultrafine filtration would be required (MacMarlane and Smith, 1989). The presence of colloidal Cu was also supported by the findings of Riveros (1992). He studied the chemical behaviour of Cu in tailings filtrate, reclaim water, T3 overflow, as well as interactions of these effluents with lake sediments.

In the past 5 to 10 years, wetlands have gained a reputation as passive biological treatment systems for mining effluents. The advantage of passive systems over chemical treatment systems is the fact, that no additional chemicals are used in the treatment process and that natural systems are utilized in the treatment.

Star Lake provided an ideal situation to test the applicability of a passive system for the treatment of gold mill effluents. The cation exchange capacity *of* bog/muskeg material provides one of the main natural cleansing processes operating in wetlands. However, colloidal Cu is not likely to be bound permanently to cation exchange sites available in the peat or organic matter in the muskeg. The possibility that the Cu colloid would break down and therefore Cu would be available for cation exchange existed. On the other hand the colloidal Cu may get adsorbed directly onto the organic matter in the muskeg. Both possibilities warranted using the two muskeg areas located below the tailings pond to treat the effluents.

A sprinkling system to disperse the effluent over the two natural "muskeg areas" T-2 and T-3 was installed. The discharge from the "muskeg areas" was within the required compliance concentrations of Cu and cyanide during operations of the sprinkling

system. The treatment approach was considered effective

In November 1990, an assessment was carried out by Gormely Associates (Gormely et al., 1990) of wetlands for gold mill effluent treatment. This included a literature review of wetlands for treatment of mine effluents and the two wetland treatment systems Jolu and Star were described in detail. The conclusions from the assessment indicated that cyanide is degraded, either chemically or microbially in the treatment approach. It was concluded on the other hand that Cu is mainly adsorbed or filtered by the muskeg. The stability of its form was not known but it was speculated that the release of Cu would not be significant.

Gormely addressed the effectiveness of the treatment approach through an evaluation of concentrations of metals in above and below ground biomass growing in the muskeg areas. The concentrations of Cu in the above ground biomass, based on vegetation samples taken during the study, were very small (42.6 mg.m⁻²). A very small percentage of the daily load at peak operations of 6,160 mg.m⁻²day⁻¹ (May 1989) was taken up into the above ground biomass.

These data of Gormely indicate that Cu uptake by biomass is not an important Cu removal process. This finding also alleviates potential concern, that this toxic element could be available in the food chain, if wetlands/muskeg would be used as a treatment facility. Below ground biomass concentrations were reported about 15 times higher than above ground concentrations, which suggests that Cu remains in the muskeg material. In 1990 Gormely described the vegetation as very stressed and unhealthy. At a site visit in 1992 by M.Kalin, the vegetation in both T-2 and T-3 muskeg areas had recovered to a healthy state in contrast to the deterioration described earlier. This suggests that Cu present in the bogs is not exerting in toxic effects on the vegetation. A study following the Gormely work was initiated by Cameco to determine the distribution of the Cu concentrations in organic materials in T-3 muskeg area (Wittrup and Nelson, 1992).

Areas with high Cu concentrations were found in the direct spray zone, with decreasing concentrations in the remainder of the muskeg areas. The removal efficiency of both muskeg areas combined was calculated to be between 93 to 98 % of the Cu. Copper was considered stable but its chemical form and the removal mechanism remained unclear (Wittrup and Nelson, 1992). These two aspects are very relevant with respect to decommissioning. Boojum Research was retained, in 1994, to assess the long term fate of Cu in the two muskeg areas.

The first step addressing the objectives involved a literature review of wetlands which have been used to remove Cu from mining waste streams to compare the Cu removal capacity of those systems to that of the T-2 and T-3 muskegs (Section 2.1). This was followed by a literature review of Cu forms in aquatic systems, which should provide the background to determine which Cu form is to be expected in the muskegs (Section 2.2). Finally the muskeg areas were sampled to derive a detailed description of the spatial distribution of the Cu in the muskeg. Copper concentrations in these profiled sample cores were determined as well as sequential extractions were carried out on selected samples to determine the chemical form of Cu (Section 4.6). The material was investigated by EDS/electron microscopy which identifies the type of material with which the copper is associated (Section 4.4). The results from all these approaches are used together with the monitoring data (Section 4.5) to determine the type of copper form present in the T-2 and T-3 and its long term fate in the system.

2.0 Literature Review

2.1 Copper Removal in Passive Treatment Systems

Most constructed wetlands for AMD treatment have been treating coal drainage in which Cu concentrations are not generally of major concern. A few wetland-type systems have been designed for base metal mine drainage. These are generally designed to enhance anaerobic microbial processes, in particular, bacterial sulphate

reduction, which can indirectly remove metals through elevation of pH and precipitation of metals as hydroxides or sulphides (Mills et al., 1989). These precipitates are insoluble and stable as long as they are not exposed to oxidising conditions.

Effective removal requires interception of AMD by an anaerobic substrate without disrupting the functioning of that process and also requires diffuse flow through sediments or 'sediment-like' conditions.

Passive systems described in the published literature have not been specifically designed for Cu removal except for Star Lake and Jolu (Gorrnely, 1990; Gormely et al 1991). A few systems have been set up in water where copper is a significant contaminant (Gallinger et al 1991; Wildeman 1993).

Eger and Lapakko (1988) determined the fate of Ni and Cu from a base-metal stockpile drainage in a white-cedar wetland in northern Minnesota. They determined that almost all the Cu was removed as the drainage passes through the wetland. At least 77 % was held in the peat rather than in higher plant biomass or pore-water. The chemical form by which Cu was removed was not determined.

Eger et al. (1994) studied wetland removal of metals from a mine drainage in northern Minnesota which like Star Lake has a near neutral pH. The water from the Minnesota mine contained approximately 2 mg.L⁻¹ Ni and low concentrations of other metals including Cu (0.04 to 0.19 mg.L⁻¹). An average of 86.5 % of Cu was removed by the Minnesota wetland, a similar figure to that achieved for Star Lake. Sequential analysis to identify the forms of copper in the peat substrate are under way.

Bench-scale tests (Bolis et al 1991) achieved almost 100 % removal of Cu and other metals over a 19 week period from acid mine drainage containing 70 mg.L⁻¹ Cu and with a pH of 2.3-2.7. The substrate was cow manure/planter soil mix selected on the basis of buffering capacity (mushroom compost was considered inadequate). These

experiments were conducted to optimize the role of bacterial sulphate reduction for metal removal. Much of the buffering capacity in this system is due to ammonia release from cow manure denitrification. The neutralization capacity and thereby the treatment capacity of these substrates will eventually run out. A system which can continuously treat effluents requires a continuous supply of decomposable organic matter to act as electron donor and carbon source for the bacteria, which would drive denitrification. The floating vegetation mat in the Star Lake condition can perform such a role.

At the Big Five Tunnel in Colorado, effective Cu removal from water containing 0.73 mg.L⁻¹ of Cu was achieved in conditions favourable for sulphate reduction (EPA, 1993). **A** mass balance for sulphur for the Big Five wetland indicates that most sulphur removal is converting it to an acid-volatile fraction along with the metals. However it has not been possible to identify specific crystalline, metal sulphide compounds (e.g. by X-ray spectroscopy). Therefore the conclusion that metal sulphide precipitation is the main method of metal removal in anaerobic conditions favourable for bacterial sulphate reduction *is* tentative. It has certainly been established that metals including copper can be removed from the waste stream in anaerobic conditions. A constructed, anaerobic or sub-surface flow wetland at Bell Island Copper, treating effluent from the tailings pond was adsorbing copper readily. As the wetland was dominated by moss the effluent acidified slightly and some copper increases in the effluents can be expected. **A** change in pH will bring about alterations to the adsorption characteristics of the decaying organic matter (personal comm., M. Kalin with A. Sobolewski, field trip of 16th. annual B.C. Mine Reclamation Symposium, Smithers, B.C., June 15-18, 1992).

The U.S. Bureau of Mines has investigated the potential of SRB activity to remove metals, including Cu, from AMD. These studies include pilot scale reactors (Dvorak et al., 1991, 1992) and data from a constructed wetland (Hedin et al., 1988, 1989; McIntire et al., 1990).

Bacterial sulphate reduction specifically requires the following conditions:

-Exclusion of oxygen -A source of sulphate -A source of organic carbon as an electron acceptor -The presence of SRBs -Suitable pH, Eh

Sediments usually provide suitable Eh and pH environments for sulphate reduction and generally contain decomposable organic material to feed the process. The mine drainage usually contains more than enough sulphate as an electron donor. The main limiting factor may be effective contact between the drainage water and the potentially sulphate reducing sediment environment.

One solution is to extend the favourable conditions through as much of the water column as possible and also provide long retention times (Kalin, 1993). **A** test cell system receiving seepage from the Copper Cliff (INCO, Sudbury) tailings achieves this in **1** m deep cells through a floating cattail mat which prevents wave action and provides organic material and a sulphate reducing sediment. This ARUM (Acid Reduction Using Microbiology) system removed more than 80 % of copper from a seepage containing approximately 1mg.L⁻¹. Thus the floating vegetation mat in the T-3 muskeg would serve as a similar system.

The measured Eh of Star Lake muskeg-sediment pore-water is not sufficiently low for sulphate reduction (theoretically -0.22 V, Stumm and Morgan (1981)). However a few negative readings have been obtained. The smell of H_2S noted during sample collection, indicated that this process was occurring in the muskegs. The fact that H_2S smell is detected, means that the H_2S is escaping as either the pH is not adequate for metal sulphide precipitation or no metals available for precipitation.

Wetlands with anaerobic sediments for treatment of acid mine drainage will likely remove much of the Cu and other metals through organic complexation as well as through sulphide precipitation (Machemer and Wildeman, 1991). Overall, it is clear that peaty materials and organic sediments are very effective in Cu removal from water. The form removed is not often reported but this is of great importance in determining the long-term stability of Cu with changes in hydrology, water chemistry etc. In particular, it is essential to be able to predict the consequences of changes in environmental conditions such as that caused by changes in drainage and the subsequent drying-out of the wetlands.

One study has examined the effect of exposure to drying and oxidising conditions on the forms of heavy metal in contaminated sediments from a Japanese Lake (Saeki et al., 1993). Sequential analysis of sediments was used to determine changes in various fractions containing Cu, Zn, Pb and Cd. These sediments have a 0.36 to 7.35 % carbon content and are therefore much less organic than the Star Lake muskeg sediments (L.O.I. > 70 %, 1994 data). The chemistry of the Star Lake sediments and changes thereof on drying out may be very different. However, Saeki et al., clearly indicate that stable precipitates such as sulphides may be converted to less stable forms (oxides) in the presence of oxidising conditions. Reportedly, much of the copper adsorbed onto both clayey and sandy sediments remained as an organic/sulphide fraction. These findings suggest that it is important to avoid drying out of sediments in T-2 and T-3. The conversion of previously precipitated copper sulphides could be expected, if changes in water levels occur or the sediments are exposed to oxygen.

2.2 Chemistry of Copper in the Aquatic Environment

Copper is an essential element for all living organisms but is also potentially very toxic (Demayo and Taylor, 1981). The chemistry of Cu in aquatic environments is summarized here with emphasis on likely Cu forms prevailing in muskegs.

The chemistry of Cu in aquatic environments has been extensively reviewed (Leckie and Davis, 1979; Pickering, 1979; Spear and Pierce, 1979; Thornton, 1979). Cu can exist in the oxidation states Cu(III), Cu(II), Cu(I) and Cu(0). In the environment, Cu(II)

predominates. Copper chemistry closely parallels that of other **divalent** heavy metals such as Zn, Cd, Pb and Ni. Copper speciation in aquatic environments is controlled predominately by pH and Eh as indicated in Schematic 1, also alkalinity and complexing agents (Spear and Pierce, 1979). Models predicting the various forms of inorganic Cu present have been developed on the basis of equilibrium and stability constants (Ernst et al., 1975; Stumm and Morgan, 1980).

Eh and pH have a profound effect on which species of Cu will precipitate from solution. Phase diagrams for Cu, such as Schematic 1, indicate that in the conditions pertaining in the bulk pore-water in Star Lake muskeg sediments (pH 5.5-6.5, Eh 0.14-0.42 V), forms of Cu can be carbonates (malachite), oxides (tenorite) or hydroxides (cuprite) are indicated. Furthermore it should be borne in mind that the microenvironment around peat and bacterial surfaces may differ markedly from the bulk pore-water chemistry. Although these minerals are predicted based on pH and Eh conditions, their occurrence would have to be confirmed through SEM studies, which have been carried out on selected samples in this study.

In most aquatic systems, Cu is present as dissolved forms (Cu⁺²) or inorganic complexes or associated with suspended colloids or particulates. The latter includes Fe(III) oxyhydroxides and organics and complexes of these two. Particulates may settle with low flow rates, whereas colloids may remain in suspension. Thus, Cu may remain in the water column in colloidal form. Humic and fulvic acids prevailing in bog environments are known to form complexes with Cu and other metals (Pettersson et al., 1993).

Samples for ICP analysis are routinely filtered through filters with a pore size of 0.45 μ m. Colloidal material and some Cu adsorbed onto Fe(III) oxyhydroxides are smaller in size than the filter pores and will thus pass through. Acidification of the filtered sample will dissolve the colloidal or adsorbed Cu and consequently, dissolved Cu concentrations can be substantially overestimated. In environments where organics are abundant, such as muskeg sediments, this is an essential consideration. It should also



Schematic 1: Eh/pH phase diagram for copper (25°C). From Spear and Pierce (1979) and indicate measurements of pore-waters of T-2 and T-3 sediment samples respectively collected in April, 1994 be borne in mind that such colloidal forms e.g. humic acid, may be mobile and carry Cu over long distances (Pettersson et al., 1993). In addition complexation 'protects' Cu from the ambient chemistry by keeping it 'out of solution'. Copper was therefore retained in suspension in conditions otherwise favourable for precipitation. Overall, dissolved organic material (DOM) may be detrimental for Cu removal and will certainly complicate predictions of what may happen to Cu in the long-term. These considerations are likely relevant to Cu which is retained in the T-2 and T-3 muskegs.

Copper is readily adsorbed to Fe(III) oxyhydroxide precipitates which are formed in oxic conditions when the pH is greater than approximately 3.5, the exact value depending on the iron concentration and Eh as indicated in Schematic 2 (Stumm and Morgan, 1981). The pH/Eh phase diagram for Fe indicates that the conditions in the pore waters of both T-2 and T-3 muskeg sediments are very near the boundary between Fe^{+2} and $Fe(OH)_3$. In other words the iron present is likely actively cycling between dissolved Fe^{+2} and Fe(III) oxyhydroxide precipitates. Therefore, any Cu adsorbed to such precipitates is not in a stable form. According to Deng and Stumm (1994), the iron hydroxides which can be formed in wetland sediments, do coprecipitate other metals such as Cu but these hydroxides are also cycling. This would mean that the Cu is rereleased.

If the Fe(III) oxyhydroxide precipitates are subsequently exposed to reducing conditions, the co-precipitated Cu and other cations will be rapidly released into solution as the dissolution and reduction of Fe(III) is taking place (Stumm and Sulzberger, 1992). Therefore, adsorption of Cu to Fe(III) oxyhydroxides in the muskeg environment is likely to be a temporary removal process.



Schematic 2: Eh/pH phase diagram for iron ([Fe] = 10⁻⁵ M, 25°C). After Sturnm and Morgan (1981). and indicate measurements of pore-waters of T-2 and T-3 sediment samples respectively collected in April, 1994

The foregoing considerations derived from the literature would indicate that seasonal changes in the bogs would result in different microbial activity and along changes in hydrology. Cu releases showing a seasonal pattern can be expected. Microbial decomposition will be more rapid during the summer months and water fluctuations would he greatest during run-off events.

Humic substances such as peat are well known for their ability to remove cations from solution. Copper is bound to peat through ion exchange and complexation processes (Volesky, 1990; Chen et al., 1990). The contaminant removal properties of peat by both of these mechanisms and applications are reviewed in Couillard (1994). In the study of Chen et al. (1990), the relative amounts of copper removed by ion exchange and complexation are reported to be dependant on the type of the peat (moss or vascular vegetation), the pH and the Cu concentration. At concentrations of 6 mM (380 mg.L⁻¹) and 14 mM (890 mg.L⁻¹) Cu water added to the two peats tested, ion exchange was the dominant removal mechanism in Chen's experiments (30 g peat d.w.L⁻¹, pH 2-4). With increase in pH, there was an increase in removal by both ion exchange and complexation, the former being more pH sensitive. The total Cu removal capacity for the two peats were 14,000 and 24,300 mg.kg⁻¹d.w. for the moss and vascular origin of the peats respectively.

The maximum Cu value found in the Cameco 1992, T-3 muskeg survey (1,100 mg.kg⁻¹d.w.) (Wittrup and Nelson, 1992) is much lower than the removal capacity reported in Chen et al. (1990) of 14,000 to 24,000 mg.kg⁻¹d.w. of peat. Areas in the direct spray zone above T-2 may be near saturation values for ion exchange and complexation with concentrations of 20,000 mg.kg⁻¹d.w. of organic matter. Much of the peat literature is on moss, mainly *Sphagnum* sp. peat which is generally more acidic and will have a different surface chemistry than the Star Lake muskeg peat which will be closer to vascular plant dominated peat. The Star Lake muskeg is a fen-like bog,

dominated by vegetation such as sedges, which prevail in alkaline conditions which enhances Cu removal.

3.0 Materials and Methods

3.1 Site Description

The two muskeg areas are located in separate drainage basins which both flow in the same direction towards David Lake, which ultimately drain into Star Lake. Map 1 shows an overview of the area. Blindman Lake, which contains the tailings, is naturally draining into T-2 but during operations the outflow was controlled by a dam. The tailings effluent was pumped to the T-2 drainage basin and distributed through a sprinkler system. From the T-2 muskeg the water was collected at a dyke and pumped to the T-3 areas where again the water was distributed through sprinklers.

From an ecological point of view, the T-2 area was originally not muskeg but a sedge/shrub meadow which has considerably less of a wetland status than a muskeg. This is in contrast to T-3 which represents a floating muskeg area.

The spray zone in each of the respective muskegs was located at the head of the drainage basin and sampling by Cameco in 1993 indicated large differences in copper concentrations throughout the muskegs. The sampling campaign in the field trip April 11-14th, 1994 aimed to cover areas which had not been sampled before and areas which were known to have high copper concentrations, as determined in the Cameco Study (Nelson and Wittrup, 1992). Both muskegs T-2 and T-3 are divided into zones and described in detail below.





3.1.1 T-2 Muskeg Area

The T-2 area comprises four zones, differentiated according to, first, whether Blindman Lake tailings pond water was sprayed over the particular zone or not, and second, according to the substrate type (dictated in large part by the level of substrate saturation and subsequent plant community development). The sampling zones are indicated in Map 1 and sampling locations are given in Map 2.

<u>Direct Spray Zone 1</u>: In this zone, the sprinkler system sprayed Blindman Lake water directly over the pine-spruce forest substrate. Therefore, the copper content of Blindman Lake water likely came in relatively even contact with the forest substrate covering this area. The spray solution percolated through the substrate, and moved down the natural grade as sub-surface flow through Zone 3 to pool in Zone **4**.

<u>Direct Spray Zone 2:</u> Peat and clay excavated from the Crown Pillar area was placed on the west slope of the T-2 area. Waste rock was spread over the peat to maintain access for vehicles. Sprinklers sprayed Blindman Lake water over this area. Water likely percolated through this pile, draining toward the perimeter of Zone **4**, located at the base of Zone 2.

<u>Forest Zone 3:</u> This area is populated with healthy pine and spruce, and the substrate is covered with thick moss. Although this area did not receive direct spraying, water was passing through the area, possibly below the organic stratum and along the clay/overburden/bedrock interface.

<u>Muskeg Zone 4</u>: The water level in this area was raised by approximately 1 m by a dyke constructed during operation of the T-2 sprinkler system. Sedges populate this area, as well as some willow and birch. Surface and sub-surface flow from the Zones 1, 2 and 3 passes through this area before being collected in the pond at the T-2 dyke.



3.1.2 T-3 Muskeg Area

The T-3 muskeg area can be divided into the zone where water was sprayed by the most recently operated sprinkler system and the non-sprayed zone. Generally in T-3 a peat layer, which was frozen, underlain by an peaty sediment slurry. This substrate was not frozen and is referred to as soup or gyttya when sampled in the study. This substrate type was encountered at six of the eight locations sampled in the T-3 area. A sample was collected at each of these six locations.

<u>Direct spray Zone 5</u>: This is the area receiving spray directly from the spigot line, i.e. within 30 m of the line. Vegetation in this zone is dominated by sedges. A few shrubs and trees (tamarack and black spruce, bog birch) were present.

<u>Muskeq Zone 6</u>: This is the remainder of the muskeg zone above the T-3 dam. Vegetation in this zone is dominated by sedges. Some shrubs and trees (tamarack and black spruce, bog birch) were present.

Control **Muskeg (Zone** 7): Samples were collected in a muskeg area located 1200 m by road southwest of the Jolu-Star Lake Roads junction, and 80 m due north of the Star Lake-Jolu road. Vegetation in this area was dominated by sedges. This muskeg was used to determine background conditions.

3.2 Field Sampling

A site visit was conducted between April 11 and 14, 1994 for collection of substrate and water samples in the T-2 and T-3 areas. At this time the ground was still frozen, facilitating sampling of the underlying water and allowing for the collection of relatively undisturbed cores of the muskeg root layers. Sampling locations (indicated on Map 2) are:

<u>Direct Spray Zone 1</u>: Three locations were sampled in this zone, T2-1, T2-2 and T2-3. At all three locations, a frozen surface (0-5 cm deep) and frozen subsurface (5-10 cm) layer sample was taken. These samples were kept frozen until sample processing.

<u>Direct Surav Zone 2</u>: Two samples were collected in this area, T2-5 and T2-6. T2-5 was collected by digging 10 cm into the peat-waste rock frozen surface layer, and sampling the peat. The sample was frozen until processing. T2-6 was collected from the peat-clay embankment as 4.2 cm diameter cores. Because of a frozen clay strata, the maximum depth of the cores was 25 cm. In addition, a surface (0-10 cm) bulk grab sample was excavated using an axe.

<u>Forest Zone 3:</u> A single location, T2-4 was sampled at the centre of this zone. A 5 cm thick frozen moss layer, the surface frozen peat layer 5 - 10 cm deep, and the lower frozen peat layer 10-15 cm deep were collected. These were stored in a freezer.

<u>Muskeg Zone 4:</u> Samples were collected from a total of 5 locations (T2-7 - T2-11) in this area. Extracted cores were as long as 37-60 cm but had to be ended due to the presence of tree-roots or rocks. In addition, grab bulk samples from 0 to 10 cm were collected at each location. **No** field measurements were taken at the substrate sampling locations because of frozen ground conditions in the T-2 area.

<u>Direct Spray Zone 5:</u> Substrate samples were collected at four locations in this zone (T3-3, T3-4, T3-5 and T3-7). A core and a "mush" (ice auger frozen cuttings) sample were collected at each station. Unfrozen peat sediment slurry (soup), present at T3-5 and T3-7 was also sampled. A polycarbonate tube was used to penetrate the slurry and the sample was retrieved into a plastic bag with a vacuum pump. The soup samples were kept at 4°C until processing. H_2S was detected from the unfrozen peat layer at T3-5.

Muskeq Zone 6: Substrate samples were collected at four locations in this zone

beyond the sprinkler system (T3-1, T3-2, T3-6 and T3-8). At T3-1, the muskeg was overlain by a 25 cm thick layer of ice. The frozen peat layer itself was relatively thin (15 cm) and comprised mainly a sedge root mass. Hand coring of this root layer proved impossible, and a soup sample alone was sampled from this location.

At T3-2, the top 40 cm of the muskeg was frozen. Because of the root layer, coring was again unsuccessful. Instead, frozen cuttings from ice auguring were sampled and kept frozen (mush samples). The peaty sediment slurry soup was sampled at this location.

At T3-6, the frozen peat layer was 40 cm thick. Upon drilling through the frozen peat, layer, the unfrozen peat layer degassed continuously (bubbling) for approximately 5 minutes (CO, + minor H_2S). Here a solid core, core cuttings (mush) and soup sample were collected.

Run-off (snow-melt) water was pooling over the 15 cm thick ice layer in the vicinity of station T3-8. An H_2S smell was detectable during sampling of core and soup samples.

<u>Control Zone 7</u>: Control samples (core, mush and soup) were collected in a muskeg area located 1200 m by road southwest of the junction Jolu-Star Lake road, and 80 m due north of the Star Lake-Jolu road. At both sampling locations, a frozen peat layer (28-38 cm thick) overlay by a peaty sediment soup layer. Thus the Control Muskeg is similar in structure to T-3.

3.3 Laboratory Measurements

Sample storage and preparation: All samples collected were maintained frozen or refrigerated in their original bags. The air was removed from the bags on site to maintain field conditions for Eh. Field measurements of the unfrozen peat slurry in the boreholes were not taken, due to weather conditions. Within 48 h of return to the

laboratory all liquid samples were processed for the determination of pH, Em and conductivity. Acidities and alkalinities were determined as well.

The frozen grab samples where thawed and the measurements were made after the sample was mixed and had reached room temperature. The frozen core samples, were cut with a band saw. Half of the core was melted and used for the physical/ chemical determinations. The other half of the cores were melted and fixed for the microscopic investigations.

Physical/Chemical determinations: The pH was measured with a Canlab probe and Jenco meter, Em with Fisher probes and Corning Model 103 meter, and conductivity with an Orion 140 meter and probe. Em, the measured redox potential, was converted to Eh by means of the formula:

$$Eh(mV) = Em(mV) + (241 - 0.66(T-25))$$

where T is the measured temperature ($^{\circ}$ C). The 241 value is a constant specific to the type of electrode used (Wetzel 1983). Acidity and alkalinity were determined by titration with 0.01 N NaOH or 0.01 N H₂SO₄ respectively with a Brinkman Metrohm 702 SM Titrino autotitrator.

For Loss On Ignition (L.O.I.) determination, the material was air dried followed by oven drying to a constant weight. The sample was split, one portion submitted for ICP analysis and 1 g sub-sample ground in a mortar and pestle. The material was ignited at 475° C for 1 h and the sample reweighed. Percent L.O.I. was determined as the final weight over initial weight x 100.

ICP analyses are carried out by a certified environmental chemical laboratory in Toronto; the laboratory's QA/QC procedures are given in Appendix 2.

3.4 Sequential Extraction Analysis

The sequential analysis of sediment is designed to determine the form of metal in the sediment, to derive conclusions with respect to the chemical stability. The sequential extractant methodology is well established and described in detail by Salomons and Forstner 1984, Bupp and Ghosh 1991, Henrot and Wieder 1990. All authors essentially employ the same methodology which lead to the following interpretation:

The extraction steps are:

1st Extraction:	1 M KNO, solution for <u>exchangeable metals</u> , solidIsolution ratio of 1:50, 2 hours shaking.
2nd Extraction:	$0.1 \text{M} \text{Na}_4 \text{P}_2 \text{O}_7 + 0.01 \text{N}$ EDTA solution for <u>organically-bound metals</u> , solidlsolution ratio 1:50, 24 hours shaking.
3rd Extraction:	1M ammonium acetate solution (pH=5) for metal carbonates, solid/solution ratio 1:50, 5 hours shaking.
4th Extraction:	concentrated HNO, solution for <u>other metal precipitates</u> (arsenates, hydroxides and sulphides), solidIsolution ratio 1:50, 2 hours heating at 120°C.

After each extraction which is carried out on the same material, the material is collected on a 0.45 μ m filter and subjected to the next extraction. The filtrate or extract is submitted to ICP for copper concentrations. Thus for extracts Cu concentrations are obtained. The percent extractable Cu is calculated against the total Cu content determined in a subsample

3.5 Electron Microscopy

Sample fixation: For each sample from the T-3 soup, 10 mL was added to 0.8 mL gluteraldehyde (25 %) in a scintillation vial. Solid samples from the forest Zone 1 were thawed and mixed. From the resulting slurry, 10 g (wet weight) was added to 0.8 mL gluteraldehyde (25 %). All samples were refrigerated until investigated.

Sample mounting: Whole mounts of fixed material were prepared for electron microscopy by floating Formvar and carbo-coated ZOO-mesh aluminum grids on small droplets of a mixed water/peat slurry. After several minutes, excess material was carefully removed from the grids with filter paper. For thin sections, specimens were washed in a solution of 0.05 M N-2-hydroxyethylpiperazine-N'-2-ethane-sulfonic acid (HEPES) buffer (Research Organics Inc., Cleveland), pH 7.2, to remove excess gluteraldehyde.

The mounted samples were dehydrated through a graded acetone series and embedded in epoxy resin (Epon 812, CanEM, Guelph). Thin sections, approximately 60 nm in thickness, were obtained with a Reichert-Jung Ultracut E ultramicrotome, and mounted on aluminum grids. To increase electron contrast of cytoplasmic material inside intact cells, some sections were stained with uranyl acetate and lead citrate.

Instrumentation: Grids were viewed with a Philips EM400T transmission electron microscope (TEM) equipped with a model LZ-5 light element detector and an exL multichannel analyzer (both from Link Analytical). The TEM was operated at 100 keV, with a liquid N₂-cooled anticontamination device in place. Energy-dispersive X-ray spectroscopy (EDS) was conducted using electron beam spot sizes of 400 nm² with a beam current of 0.1 μ A. Spectra were obtained by collecting counts for 100 sec (live time). The d-spaces for crystalline mineral phases were examined by SAED (selected area electron diffraction) with a camera length of 800 mm. The elemental composition of amorphous phases were characterized using EDS spot analysis run with the Link quantification program to determine stoichiometric ratios of elements.

4.0 Results

4.1 Chemistry of Field Samples

Cores of muskeg material from the T-2 and T-3 muskegs were collected during spring, **1994.** The samples generally consist of root and coarse black peat with occasional clay layers. A detailed description of the material is given in Appendix 1 (Table A2).

In Table 1, the characteristics of the samples are summarized. Water from the regular monitoring stations was also collected during the field trip. The Cu concentration in 0.45 µm filtrates from selected soup samples and from Blindman Lake were determined to assess background concentrations of the dissolved phase of Cu in the muskeg. Blindman Lake concentrations were 0.141 mg.L⁻¹ and the soup filtrate from T3-5 was lower with 0.062 mg.L⁻¹. The Cu concentration in the soup sample from the Control Muskeg (CM-1) was 0.017 mg.L⁻¹. The complete ICP analyses are given in Appendix **2.** These results suggest that indeed, particulary considering the wet density of the soup the amount of Cu in the pore-water represents a small fraction of the Cu which is associated with the solids (Table 1).

The field pH values of soil/sediment slurries fell within the range 5.87 to 6.89 which is that expected range for alkaline fens. This range is favourable for removal of Cu by ion-exchange, organic adsorption and for the precipitation of Cu as carbonate or sulphide. It is also favourable for Fe(III) oxyhydroxide precipitation to which Cu may adsorb and coprecipitate. The Eh values indicate mildly oxidising conditions in the sediments throughout the muskeg areas. The H₂S smell often detected when sampling the sediments along with redox potential is sufficiently low (Eh <-220 mV), indicates that sulphate reduction is taking place. All samples had low alkalinity and acidity and are thus well buffered.

L.O.I. values were generally very high (>80 %). This, coupled with densities generally a little above 1.0, indicates highly organic materials, namely peat. Exceptions to this

Table 1: Chemistry of Star Lake soil/sediment samples

ocatior	Sample	Depth	Wet	Wet wt	LOI	pН	Eh	Cond.	Acidity	Alkalinity	Cu
	type	(cm)	density	dry wt	(%)		(mV)	(mS/cm)	(mg/L	(mg/L	(mg/k <u>c</u>
			<u>(g/mL)</u>						CaCO3)	CaCO3)	
m e 1 -	ect sp	/ zone	wood!	pove 1							
T2-1	grab	0-5	1.44	2.55	26.1	6.89	332	54			3440
	grab	5-10	1.18	2.46	45.8						2410
T2-2	grab	0-5	1.24	3.03	59.3	6.81	374	224			22200
	grab	5-10	1.13	2.98	69.2	6.71	381	58			1710C
T2-3	grab	0-5	1.22	2.75	52.1	6.62	379	32			201 0C
	grab	5-10	0.77	2.67	41.6						19000
m e 2 - c	direct spra	ay zone c	on bridge	with add	ed peat						
T2-5	grab	0-10	1.52	1.32	12.1						747
T2-6	core	0-26	1.21	2.27	24.6	6.62	379	485			2000
me3-r	ion-spray	ed zone	in woods	above T	2						
T2-4	grab	0-5	1.24	2.65	79.2						78
		5-10	0.62	2.72	85.2						15100
me4-T	2 muske	g									
T2-7	core	0-53	1.05	4.45	79.3	6.47	306	364			80.2
T2-8	core	0-57	1.08	2.91	25.7	6.71	333	258			1220
T2-9	core	0-55	1.02	4.32	83.2	6.51	269	182			1300
T2-10	core	0-49	0.99	3.95	81.7	6.81	250	252			1280
T2-11	core	0-37	1.04	4.50	78	6.32	305	136			2080
2 <u>ne 5 - 1</u>	3 spray-	zone									
T3-3	core	0-42	0.96	4.57	92.8	5.89	415	133			262
T3-4	core	0-22	0.94	3.24	89.8	6.79	371	223			2790
T3-5	core	0-33	0.99	3.77	87.5	6.45	376	278			1490
	soup		0.97	36.06	90.4	6.42	136	206	7.2	57.4	103
T3-7	core	0-37	1.00	4.42	91.4	6.58	337	262			1510
	soup		1.07	92.57	91.5	5.54	454	153	7.4	16.6	196
								1]	
T3-1	soup		1.00	40.88	82	6.37	273	240	7.4	68.8	169
T3-2	soup		1.03	30.55	91.4	6.12	338	135	4.5	22.5	46.6
T3-6	core	0-24	1.14	3.16	89.4	6.48	342	316			921
	soup		1.00	149.97	90.8	5.87	425	221	11.5	47,3	53.4
T3-8	core	0.33	0.88	4.33	73.3	6.36	400	79			255
	soup		1.02	131.63	72.7	6.33	408	139	3.1	23.1	375
sne 7 -C	ontrol m	uskeg									
CM-1	core	0-28	1.02	3.65	88.5	6.54	353	98			15.4
	soup		1.01	23.06	88.9	6.04	320	150	2.6	28.9	15.9
CM-2	core	0-38	0.95	4.58	88	6.43	368	110			38.2
	soup		0.96	33.44	89.4	6.05	389	95	3.9	18.6	21.4

are T2-1 and T2-5, which had substantial clay contents with L.O.I. of 26 % and 12 % respectively.

Cu content of Soil/sediment Samples 4.2

Cu contents of the samples are summarised in Table 2.

A - Bo	ojum data (April 1994)						
Zone	Description	Sample	# of	Mean Cu	sd	Minimum	Maximum
		type	samples	(mg/kg)		(mg/kg)	(mg/kg)
1	Direct sprayed zone in woods above T-2	Grab 0-5cm	3	15,247	8,392	3,440	22,200
		Grab 5-10cm	З	12,837	7,413	2,410	19,000
2	Direct spray zone on peat-amended dyke	Grab/core	2	1,374	627	747	2,000
3	Non-sprayed zone in woods above T-2	Grab 0-5cm	1	78		78	
		Grab 5-10cm	1	15,100			15,100
4	T-2 muskeg	Core	5	1,192	640	80	2,080
5	T-3 direct spray zone	Core	4	1,513	894	262	2,790
		Soup	2	150	47	103	196
6	T-3 non-sprayed zone	Core	2	588	333	255	921
		Soup	4	161	133	47	375
7	Control muskeg	Core	2	27	11	15	38
		Soup	2	19	3	16	21

Α

Zone	Description	Sample	#of	Mean Cu	sd	Minimum	Maximum
		type	samples	(mg/kg)		(mg/kg)	(mg/kg)
5	T-3 direct spray zone	Core	6	225	247	23	690
6	T-3 non-sprayedzone	Core	18	110	257	7	1100
7	Control muskeg	Core	2	3	0	3	3

Zone 1 (Zone 2 - Zone 6) is a reflection of both an inadequate retention time for removal and the fact that saturation of Cu in Zone 1 (direct-spray zone) has taken place. The lower values found in Zone 1, at sampling location T2-1 relative to T2-2 and T2-3 may be due to a lower peat content and therefore, lower Cu ion-exchange and complexation removal capacity. Cu values for the samples from 5-10 cm depth were generally a little lower than the surface samples.

The samples at stations T2-5 and T2-6 (Zone 2) are located in the area of the dyke between Blindman Lake and the T-2 muskeg. These two samples had much lower Cu concentrations than those in Zone 1. This may be in part attributable to the location at the end of the spray line where sprayed volumes of effluent were less than at the centre of the spray line. The physical distribution of the spray could not be expected to have been even. Furthermore, the peat is much deeper here and the Cu may have penetrated to greater depths than in Zone 1.

The data for T2-4 (Zone 3) are particularly interesting. This location is not in the direct spray zone but is in the flow path of water between the spray zone and the T-2 (Zone 4) muskeg. The trees in this area appear healthy and there is a ground-cover of living mosses. In contrast, in the direct spray zone, most trees are dead and the soil surface is devoid of vegetation. The T2-4, 0-5 cm sample was peaty and contained much living moss. The Cu content of this material was relatively very low (78 mg.kg⁻¹d.w.) in contrast to the Cu content of the underlying peaty soil which was high (15,100 mg.kg⁻¹d.w.). This indicates that water containing Cu flows over or through the soils of this area from the direct-spray zone above.

In the T-2 muskeg (Zone 4), sample Cu contents were in the range of 80 to 2,080 mg.kg⁻¹d.w.. The low value was for T2-7 at the base of the peat-amended slope. These values are greater than those of the control muskeg (15 to 38 mg.kg⁻¹d.w.). For most of the spraying period (1989-1993) this area was flooded. As concentrations are well below saturation levels for ion-exchange and complexation (Chen et al., 1990), it
suggests that the Cu form might not have been available by these two processes.

Concentrations of Cu in core samples from the T-3, direct spray zone (Zone 5, mean of 1,513 mg.kg⁻¹ d.w.) were similar to those in the T-2 muskeg (Zone 4, mean of 1,192 mg.kg⁻¹ d.w.). Much less Cu was found in the soup at these locations indicating that most Cu removal takes place on contact with the solid material. The Cu concentrations in soup (mean of 150 mg.kg⁻¹ d.w.), although much lower than in the floating solid layer, were still higher than those of the control muskeg (Zone 7, mean of 19 mg.kg⁻¹ d.w.). In the non-sprayed zone (Zone 6), mean Cu content (588 mg.kg⁻¹ d.w.) was lower than in the direct sprayed zone similar to T-2 again indicative of rapid removal of Cu from spray water on contact with the surface material.

Cu concentrations in T-3 samples in this study are generally somewhat higher than those reported for 1992 in the Cameco Star Lake muskeg sampling report (Wittrup and Nelson, 1992) as shown in Table 2. This could be due, in part to the additional spraying in 1993 or generally reflecting the heterogenous distribution of the Cu in the muskegs.

4.3 Cu Load to the T-2 and T-3 Areas

The Cameco-Star Lake annual reports give estimated flow rates for the sprinklers and also Cu concentrations in the effluent of T-2 (station 2.3.5), Blindman Lake (station 2.3) and T-3 (station 2.5). This data has been used to estimate load of Cu discharged to the T-2 and T-3 muskegs summarised in Table 3. Estimates made for 1993 monitoring data indicated that only 13 kg for T-2 and 19.5 kg for T-3 were added for the entire spraying period (June to September). Of the total load to T-2 for the period 1989-1993 (3.4 t), 70 % was added in 1989 and another 17 % (0.57 t) in 1990.

To demonstrate that estimates of total Cu load can be variable, the estimate of Gormely of 1,918 kg for T-2 is included in Table 3. Here the loading was estimated based on

flow rates per sprinkler and areas of wetland irrigated.

Year	T2	T3
	(kg)	(kg)
1989	2,422.2	251.5
1990	570.8	79.6
1991	344.3	48.9
1992	97.4	40.8
1993	13.0	19.5
Total load, 1989-1993	3,447.7	440.3

Table 3:	Estimated Cu load in T-2 and T-3	3

	T2	T3	
1989 estimated Cu load (kg)	1,918	179	
Boojum estimate (k on soil/	sediment C	u content)	
	T2	T3	
Total load (1989-1993)	T2 7,290	T3 1,310	

The variability in the estimates derived from the spraying records was evaluated by comparing the Cameco effluent load with Gormely's estimates. Clearly, very different estimates of Cu load can be obtained. The Boojum estimates, which are approximately twice as great as those reported by Cameco, are also prone to variability. Firstly in the estimates of the areas of the zones and secondly the variability of the samples, resulting in a non-representative mean Cu concentrations. Whatever the magnitude of variability, it is abundantly clear that most of the copper discharged to the T-2 and T-3 muskeg areas is found in the direct spray zone in T-2.

Copper load for each of the 6 zones in the T-2 and T-3 areas were estimated from the Cu content of the soil/sediment samples and are summarised in Table 4.

Area	Control area	I	T-2	Area		T-3/	Area	Grand
Zone	Zone 7	Zone	Zone	Zone	Zone	Zone	Zone	total
	<u>.</u>	1	2	3	4	5	6	
Area (ha)	0.0001	0.67	0.13	0.74	1.24	0.62	2.23	5.63
Cu in zone (t)		3.87	0.23	1.28	1.91	0.72	0.59	8.60
Cu background (t)*	2.92E-06	0.020	0.004	0.021	0.036	0.018	0.065	0.16
Cu from spraying (t)		3.85	0.22	1.26	1.88	0.70	0.52	8.44
Cu (t/ha)	0.0292	5.771	1.816	1.739	1.544	1.154	0.265	
Sprayed Cu (t/ha)		5.742	1.787	1.71	1.515	1.125	0.235	

* Background Cu in Zones 1-6 based on concentration in control muskeg (Zone 7)

The Cu load are based on areas of the zone derived from Maps 1 and 2. The area reached by the spray was assumed to be 30 m away from the line installation. 'Volume of material' in each zone was based on the depth sampled (grab sample depth or core depth), **As** there is more volume of bog to the depth of sampling than, the load calculated will represent an conservative estimate. Copper may well be present at greater depth in some zones. For soup sediment samples a depth of 1 m was assumed.

The mass of sediment material was calculated by first multiplying the sample depth by the area of the zone, to give the volume of material. This volume was multiplied by the wet density to give mass of wet material. This value was then divided by the wet weight/dry weight ratio given for each sample in Table 1, resulting in mass of dry material. These calculations were carried out for each sample. The mean of the copper content for each zone was the average of estimated mass multiplied by the Cu concentration in each sample. Where samples were collected from two depths in the same location, the Cu concentrations for the two layers were added. For the soup sediment layer of the T-3 muskeg (Zones 5 and 6) a depth of 1 m was assumed for

calculations. Background Cu values were estimated by carrying out the same procedure for the samples from the Control muskeg, both for solid core and soup sediment samples (Zone 7). Detailed calculations are given in Appendix 1 (Table AI).

Zone 1, the direct spray zone in the woods above T-2, had a total estimated Cu content in the top 10 cm of 3.9 t (Table 4). This exceeds estimated total T-2 effluent load based on flow rates and Blindman Lake copper concentrations of 3.4 t. wwever, given the assumptions made for the estimates, the two figures are very close. Given the fact that in the spray zone most of the Cu was applied in 1989, the analyses of the 1994 samples suggest that most of the Cu has remained in this zone for 5 years, Furthermore the material in this zone has been subjected to changes in water saturation and in oxidation/reduction conditions. This indicates that indeed this material has bound copper relatively strongly which is also supported by the results of the sequential extractions (Table 5, page 54).

The lower load estimated for Zone **2** may be in part a result of being at the end of the sprinkler line and therefore receiving a smaller volume of Blindman Lake water. Zone 3 was not directly sprayed but the extremely high Cu content of a sample from below the surface (15,000 mg.kg⁻¹d.w.) indicates that much Cu-laden water flows through or over the soil here. This material is near saturation for ion exchange/complexation, the dominant Cu removal mechanisms. Zones 3 and 4 of the T-2 muskeg had a total estimated load of 1.3 t of which most is contained within the floating, surface layer with 0.83 t compared to 0.06 t in the soup. The total load of Cu again exceeds the estimate of 0.4 t based on flow rates and station 2.3.5 water quality data. The Cameco 1992 study suggested that the latter loads may be underestimates, due to flow monitoring problems of the sprinkler system.

The advantage of muskegs as a treatment system for gold mill effluents is evident when the distribution of Cu is compared on a per unit area basis. Much of the copper

is retained in the direct spray zone, where the overall Cu content per ha was approximately 3 times that of all other utilized areas. The area of this zone (0.8 ha) is only a small fraction of the total area utilized (5.63 ha).

From the zonal Cu distribution estimates, an interesting observation can be derived. The direct spray Zone 1, which had the highest concentrations of copper, consisted essentially only of forest soil and had relatively high organic matter content (mean of 49 %) compared to Zone 2 (mean of 18 %) (Table 1). Zone 2 was also part of the direct spray zone and contained the excavated peat and clay from the crown pillar. Zone 2 material appeared to be much less effective in retaining copper than the more organic-rich natural forest soil. This is expected as the sequential extraction analyses indicate that organic complexation is the major process for Cu removal from the effluent. The loads in Zones 3 and 4 indicate that the materials here likely retain Cu by similar mechanisms as in Zone 1, since the total mass retained in these areas is very similar. For T-3 the same observation applies, as there is no real difference between the retention of total copper in both Zone 5 and Zone 6.

The % L.O.I. values (organic contents) in all other zones in both muskegs are generally higher ranging between 70 % and 90 %. If the copper which reaches these zones, was all available for organic complexation or ion exchange, then essentially no copper should be found in T-3. This however is not the case, and demonstrates, that the form copper removal process is likely different than that of Zones 1 and 2.

The more mobile fractions of the discharged Cu leaving Zones 1 and 2 consists of two types. The first is the Cu found in the lower part of T-2 (Zone 3 and **4**) which has moved directly from Zone 1 and has remained mobile and not bound. The second type of Cu would be that which represents pore water of Zone 1, possibly from the breakdown of complexes. Only these mobile forms of Cu will be transferred to T-3. The distribution of Cu in T-3 is more or less even throughout the muskeg and is similar to the lower part of T-2 (Zone **4**).

Thus a small fraction of the total Cu loaded into the muskegs is found downstream of Zones 1 and **2**. This interpretation is corroborated by the results obtained from the sequential extractions which suggest, that most of the Cu in Zones 1 and 2 is organically bound and thus only mobile when changes in the pore water take place. The sequential extractions are discussed in detail later in this report. Some Cu release might be expected during spring run-off events, as indicated by the 1994 monitoring data.

4.4 Electron Microscopy Study

The role of bacteria in metal stabilization in the Star Lake muskegs should also be considered. Most microbial growth occurs in biofilms. These environments are dominated by sharp gradients which may have profound effects on exchange processes between surfaces and the bulk liquid medium. All the chemical characteristics reported for the water samples, represent bulk chemistry, and thus do not describe the conditions which prevail at the surface sites, i.e. the biofilm where Cu is complexed or bound in other ways. Biofilms (Ferris et al., 1989) and bacterial surfaces (Mullen et al., 1989) are sites of enhanced accumulation of Cu and other metals in contaminated aquatic ecosystems. Microscopic examination of Star Lake muskeg material may provide further insight into selective association with microbial surfaces.

In Section 4.4.1, the results from the microscopic evaluation of the samples collected in this study are presented. As evident from the Eh-pH phase diagrams, the precipitation processes which can lead to Cu stabilization in muskeg sediments are complex. Therefore direct evidence of the presence or absence of true precipitates, other than amorphous precipitates (hydroxides) was sought using EDS scans. The work was carried out at the University of Guelph under the supervision of Dr. G. Ferris of the Department of Geology, University of Toronto.

4.4.1 Electron Diffractive X-Ray Spectroscopy

Electron diffractive X-ray spectroscopy (EDS) essentially displays the spectrum of Xrays emitted from a sample bombarded by electrons. The X-rays have a wavelength characteristic of the element bombarded. The peak size is a reflection of the number of X-rays of a particular wavelength emitted. Each element emits X-rays with several different wavelengths. Therefore, for some elements, more than one peak is present in the scans. A selected sample area is bombarded in the electron microscope. A total of approximately 100 scans were collected and they are all presented in Appendix 4.

All scans have a large Al peak, much of which is attributable to the aluminum grids used to support the specimens. Therefore, Al is not discussed in the interpretation of the scans. The Cl peak is also ignored as chloride is present in the fixative for the mounts. Only elements with an atomic weight of about 20 g or greater can be separated by this method. The large peak to the left of scans represents the sum of the lighter elements such as N, O and C, which can not be quantified.

Scans which showed particularly interesting features where selected for interpretation in this section. Images of scanned areas were interpreted as organic matter, mineral particles or bacterial cells, indicated on the labels beneath each scan. All three types of material were found in all the samples examined. The organic matter always predominated. Figs. 1-20 show a selection of scans to represent the variety which was encountered in the material.

Figs. 1-12 show scans for samples from T2-3, the direct spray zone in the woods above the T-2 muskeg. Figs. 1-4 show scans for mineral particles (angular-crystals). Fig. 1 scan is dominated by Si with minor peaks for Fe, Cu and Ca. This is clearly a silicate or quartz crystal. Fig. 2 shows large peaks for K and Fe and Si and smaller Cu and Ca peaks. This may be a K-feldspar. The scans in Figs. 3 and **4** have the same peaks as Fig. 2 but Fe is the largest peak. The identity of these mineral phases is not

known.

Figs. 5-8 are scans for organic particles in the T2-3 samples. Large copper peaks are present in all samples and are sometimes accompanied by large Fe peaks (Fig. 8), Ca peaks (Figs. 6 and 7) or Si peaks (Fig. 8). The dominance of Cu in this material is indicative of complexation or cation exchange of Cu with the organic material.

Figs. 9-12 are scans of bacterial cells in the T2-3 samples. All display a Cu peak (particularly large in Fig. 11) indicating that Cu is associated with bacteria. The photomicrographs (Plates 1 and 2) indicate that 'electron dense' areas, which appear relatively dark, are present on the outside and inside of the cells. As Cu is the dominant 'electron dense' factor on the scans, this indicates that Cu is removed both by association with materials outside the cells and also through uptake. All scans of bacterial cells have S and P peaks. No mineral phases were clearly associated with either the outside or inside of bacteria. There is no evidence in this material for precipitate formation around intact bacterial cells.

Figs. 13-16 show scans for samples from the T-3 soup. Inorganic (mineral) phases of iron (probably iron hydroxides) were present associated with bacteria in one sample (Fig. 13). As in the T-2 samples, mineral phase scans were often dominated by Si suggesting an abundance of silicates. Copper peaks were present in all scans.

Figs. 17-20 show scans for control muskeg (CM) samples. In contrast to the other samples, these scans are dominated by peaks for Ca. Small Cu peaks were present in all scans. As elsewhere, mineral phase scans were typically dominated by Si.



Fig 1: T2-3, 0-5 cm grab sample - mineral particle





Fig 3: T2-3, 5-10 cm grab sample - mineral particle









Fig 7:T2-3, 5-10 cm grab sample - organic particle



Fig 8: T2-3, 5-10 cm grab sample - bacterial cell



Fig 9: T2-3, 0-5 cm grab sample - bacterial cell



Fig 10: T2-3, 0-5 cm grab sample - bacterial cell



Fig 11: T2-3, 0-5 cm grab sample - bacterial cell



Fig 12: T2-3, 5-10 cm grab sample - bacterial cell





Plate 2: T2-1 5-10 cm Bacterial colony x 23,000, unstained whole mount



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Fig 16: T3-5 soup - bacterium



Fig 17: Control Muskeg (CM-2) soup - organic particle



Fig 18: Control Muskeg (CM-1) soup - mineral particle

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Fig 19: Control Muskeg (CM-1) soup - bacterial cell



Fig 20: Control Muskeg (CM-1) soup - organic particle

4.4.2 EDS Scans - Summary

The presence of Cu in all materials examined (mineral phase, organic phase and bacterial cells) suggests that the previously suspected removal mechanisms are responsible for Cu removal. The greater frequency of large Cu peaks in the organic materials indicates a predominance of removal by ion-exchange and complexation rather than precipitation reactions. Uptake of Cu by bacteria is also indicated in the photomicrographs. The apparent displacement of Ca by Cu in the organic material indicates that ion exchange may be very important as a means of Cu removal in the muskegs.

4.5 1992-1994 Monitoring Data

Monitoring data for pH, Cu and electrical conductivity for 1992-1994 for Blindman Lake (Stn. 2.3), T2 effluent (Stn.2.3.5) and T3 (Stn.2.5) are shown in Figs. 21-23. In 1992, there was a general increase in pH through the monitoring period at all 3 locations. In 1993, the early readings for Blindman Lake were higher than in 1992 data but then declined after spring run-off. A slight pH increase in the fall was evident for all three locations.

Over the observation period, there was a fairly steady decline in Cu concentration in the effluent from all three discharge points, with the exception of one peak concentration for Blindman Lake in spring 1992 with 1.6 mg.L⁻¹ of Cu. This peak is likely an error, as it was not clearly reflected in the T2 effluent. In 1993 and 1994, no increase in Cu concentration leaving Blindman Lake is evident during spring run-off. The Cu concentrations in T-2 show an annual spring run-off peak for all three years. Using the conductivity and pH of the effluents (Figures 21 and 23) as an indicator to determine the release process, it is evident that, pH declines (Figure 23), by about 0.5 of the pH unit and increase in conductivity (Figure 21) takes place. The increase in Cu concentration is clearly evident in the effluents from T-2. This is due to dissolution of





the pore-water in Zone 1. The Cu concentrations in T-3 are essentially not varying at the spring run-off although decrease in pH is associated with increases in electrical conductivity. This indicates that the Cu released in T-2 (below Zone 1) and in T-3 is not mobilized by increases in conductivity and decreases in pH. The trends observed in the monitoring data are confirm and corroborate the observations made in the EDS scans and the sequential extractions.

Unfortunately, Cu concentrations monitored during the spring exceeded regulatory limits in T-2. The consistency of this pattern in the two springs, the absence of a spring runoff Cu peak for the Blindman Lake effluent and the greater concentrations in T-2 effluent, than Blindman Lake effluent in the spring of 1993 and 1994 suggests that a fraction of Cu held in the soil/sediments of Zone 1 in T-2 is released.

4.6 Sequential Extraction Analysis

The Star Lake T-2 and T-3 muskeg sediments will undoubtedly contain copper derived from the sprayed contaminated water in both ion exchangeable and organically complexed forms. Cu may also be present as a coprecipitate of Fe(III) oxyhydroxide and possibly as precipitates of Cu hydroxides and Cu sulphides. Sequential analysis carried out on the material, represents a means to further substantiate the deductions made from the EDS investigation of the solid samples on the Cu forms present in the muskeg. Sequential extractions from the material can also support the Cu forms which are suggested based on the measurements of Eh, transferred to the Eh/pH phase diagrams.

Grab samples from the direct spray zone in T-2 (Zones 1 and 2) and from the T-3 muskegs were subjected to sequential extraction after Henrot and Weider (1990). This procedure fractionates metals as held in/on solid material by ion exchange (1st. extract - 1 M KNO,), organic complexation (2nd. extract - EDTA + 1 M Na₄P₂O₇), as soluble precipitates such as oxides and carbonates (3rd. extract - acetic acid) and as stable

precipitates such as sulphides (4th. extract - concentrated HNO,). The first two extractions yield the most environmentally mobile forms of the metal.

For the sequential extraction samples with relatively high Cu contents were selected, as the same material is extracted 4 times. If samples do not contain sufficient Cu originally, the concentrations of Cu in the extracts could be below the detection limit of the analytical method and the mass balance of the metal is more difficult to obtain, due to increasing methodological and analytical error with decreasing concentrations. The results for the T-2 and T-3 samples which have been subjected to sequential extractions are presented in Table 5.

Most of the Cu is extracted by EDTA/Na₄P₂O₇ the second extract in Zone 1. This suggests that the Cu in the material is mainly held by organic complexation. In the direct spray zone (Zones 1 and 2), 67 % of the Cu is complexed to organic matter and only a small fraction (0.07 %) is in ion-exchangeable form. The 3rd and 4th extractions yield about 3.5 % to 7 % of the copper in a more stable precipitate form. The extractions account for 75 % to 81 % of the total copper determined in the sample for T-2. The extracted Cu fractions for the two T2-2 (Zone 1) samples (0-5 cm depth and 5-10 cm depth) are very similar indicating the same Cu removal process(es) prevail in this zone.

The T2-6 (Zone 2) the second extraction contained a larger quantity of Cu 7.86 mg.g⁻¹ d.w. than was determined in the solid sample before extraction of 2.00 mg.g⁻¹d.w.. As a result, the second extraction appears to contain 393 % of the total Cu reported in the solid sample. This is due to the fact, that the material for the chemical analysis and for the extraction have to be subsampled. In the case of T2-6, the material is very heterogenous. Thus the results from the extractions of T2-6 material can not be considered as valid. Furthermore, as can be noted for the other samples which also contain only about 2 mg.g⁻¹ dry weight total Cu, the extractions account for only 20 % to 50 % as might have been expected with the decreasing Cu concentrations.

Table 5: Sequential extraction analysis - Cu in 1st. and 2nd. extracts

			Sample			
	T2-2 0-5cm	T2-2 5-10cm	T2-6	T2-I 1	T3-4	T3-6
	Zone 1	Zone 1	Zone2	Zone4	Zone5	Zone6
Volume of extract (mL)	100	100	100	100	100	100
Sample extracted-freshwt. (g)	10	10	10	10	10	10
Dry wt/wet weight	0.33	0.33	0.44	0.22	0.31	0.31
Sample extracted-dry wt. (g)	3.3	3.3	4.4	2.2	3.1	3.1
1st extract (1M KNO3)						
Cu in 1st extract (mg/L)	0.50	0.45	1.74	0.16	0.23	0.12
Cu in 1st extract (mg/g d.w.)	0.0152	0.0136	0.0395	0.0073	0.0073	0.0037
Total Cu in material (mg/g)	22.10	17.10	2.00	2.08	2.79	0.92
Cu in 1st extract (%)	0.07	80.0	1.98	0.35	0.26	0.41
2nd extract (EDTA/1M Na4P2O7)						
Cu in 2nd extract (mg/L)	494.00	379.00	346.00	7.37	9.75	1.16
Cu in 2nd extract (mg/g d.w.)	14.97	11.48	7.86	0.34	0.31	0.04
Cu in 2nd extract (%)	67.74	67,16	393.18	16.11	11.27	4.07
3rd extract (1M NH4 acetate)						
Cu in 3rd extract (mg/L)	50.5	20.5	28.5	0.982	1.47	1.47
Cui 1 3rd extract	1.53	0.62	0.65	0.04	0.05	0.05
Cu in 3rd extract (%)	6.92	3.63	32.39	2.15	1.70	5.15
4th extract (conc HNO3)						
Cu in 4th extract (mg/L)	47.7	21.7	29.9	13.4	6.3	3.88
Cu in 4th extract (mg/g d.w.)	1.45	0.66	0.68	0.61	0.2	0.13
Cu in 4th extract (%)	6.54	3.85	33.98	29.28	7,28	13.60
Estimated residual Cu (% of total)	18.74	25.28	(361.53)	52.11	79.48	76.78

1st extraction - exchangeable Cu 2nd extraction - organically bound Cu Zone 1 and 2 - direct spray zone in T2 Zone 4 - T2 muskeg 3rd extration - Cu carbonates 4th extration - other Cu precipitates Zone 5 - direct spray zone in T3 muskeg Zone 6 - non-sprayed zone in T3 muskeg However the muskeg samples (T2-11, T3-4 and T3-6) which have a lower total Cu content exhibit a different pattern in the sequential extractions, than the material from Zone 1. This can be expected as the mobile forms of Cu, which escape the direct spray zone in T-2 would be in a different chemical form, as the Cu which was complexed and remained in Zone 1.

In Zone 1 and 2 samples, very little Cu was held by ion exchange (first extraction) but a slightly larger fraction is held in this form in the lower part of T-2 and in T-3. For samples at the bottom of T2 (T2-11) and in T-3 the Cu in the second extraction yields a lower fraction with 4 Y_0 - 16 % of the total Cu. The third extract yield similar percentages in both T-2 and T-3 ranging between **2** to 6 %. The results are expected, as the formation of hydroxides and carbonates is mainly governed by pH which is similar in both muskegs.

However the fourth extract yields a higher fraction of Cu with 7 Y_0 to 29 Y_0 present as stable Cu precipitates mainly in the form of sulphides in T2-11, T3-4, T3-6 than in Zone 1 (3.5 % to 6.5 Y_0). This suggests that the Cu is present in a very stable form, such as oxides or hydroxides. Eh/pH phase diagrams for Cu suggested that tenorite (CuO) or malachite (CuCO₃(OH)₂ may precipitate in the conditions found in the muskeg pore-water (pH 6-7, Eh 300-400 mV Schematic 1). It would not be unreasonable to suggest, that the remainder of the Cu in the material in this area, given that only account for about 20 to 50 % of the copper, is present in environmentally stable forms, the largest fraction extracted from this material.

4.7 Melt Water and Distilled Water Leachate Analysis

Given the spring peaks in the Cu concentrations in the monitoring data, along with the findings from the literature, the Eh/pH conditions in the muskegs and the EDS observations, a simple preliminary laboratory assessment was carried out. The frozen

muskeg was melted and additions of distilled water, simulating spring run-off and heavy rainfall events was used to flush Cu out of the material.

A 500 mL frozen sample from the T-2 direct spray zone was allowed to melt in a 10 cm diameter column. The resultant melt water was collected through a spigot at the bottom of the columns. Approximately 250 mL was derived from the frozen wet volume (melt water, Table 6). Subsequently 250 mL of distilled water was added slowly (over a period of 15-20 min), replacing the melt water and the resultant leachate was collected. This sequence simulates spring when frozen soils and sediments melt and the pore water in the muskeg is displaced by run-off.

Sample	cu
description	(mg/L)
Melt water-filtered	0.221
Melt water-total	0.463
Leachate-filtered	0.854
Leachate-total	0.849

Table 6: Spray zone soil-Cu in melt water and leachate

Although this experiment is a very simple simulation of the field conditions, the results of the Cu concentrations in the dissolved ie. filtered (um 0.45) and total waters, given in Table 6 indicate, that concentrations of copper are comparable to those reported for spring run-off peak concentrations in the monitoring data for T-2 (Sta. 2.3.5). The Cu content of the melt-water and leachate are in the same range as observed for T-2 effluent (1992 to 1994 data). The dissolved copper concentrations ranged between 0.2 and 0.8 mg.L⁻¹ in the meltwater.

The concentration of total Cu is higher than the dissolved Cu. This suggests that the thawing process dislodges larger organic complexes, which in the field might release complexed Cu due to pH changes. The observation that the colour of both the distilled leachate and the melt water was a deep orange-brown is indicative of humic acid or

other organic substances being released from the material. The results from this one sample can not be used to derive an assessment the amount of Cu which could be released in total. It however does suggest, that the approach could be used to determine the total amount of Cu available for displacement in the muskeg material. Thus it can be concluded, given the indication of mobility of the organically bound and precipitated Cu in the muskegs, based both on experimental data and the literature, that a portion of the Cu will mobilize on a seasonal basis.

5.0 Long Term Predictions

The effluent treatment approach used to remove cyanide and Cu by spraying it over a muskeg area has effectively concentrated copper in the direct spray zone. Most *of* the Cu sprayed in 1989 is still retained in the soils and sediments of this area, but some appears to be mobile, particularly during spring run-off. The observed spring run-off peak for Cu concentrations in T-2 effluent in 1992, 1993 and 1994 is suggesting that this is the case. However the lower Cu concentrations in the monitoring data for T-2 reported for the summer and fall indicate that the thawing process is contributing to the replacement of the pore-water. The more stable forms of Cu noted in T-3 (sequential analysis) are likely formed during spring run-off, some estimates can be given for the length of time that spring peaks in Cu concentrations can are expected.

The drainage basin hydrology of T-2 and T-3 is summarised in Table 7. From the monitoring data, the three peak Cu concentrations in spring run-off water leaving T-2 result in a mean of $0.52 \text{ mg}.\text{L}^{-1}$ (mean of 1992-1994 data, Fig. **22**).

If all the run-off from the T-2 drainage basin occurred at this time and the Cu concentrations were as high as during the peaks, the Cu load leaving this area

	Mean Annual		
	Precipitation		
			ж.
Brabant Lake	530.1	mm/yr	
Cree lake	413.8	mm/yr	*
La Ronge	485.2	mm/yr	*
Stanley	434.9	mm/yr	*
Average Annual Precipitation	466	mm/yr	*
Evapotranspiration	300	mm/yr	*
Net Precipitation Available for Run-off	166	mm/yr	*
			**
Area of T-2 Drainage Basin	5.84	ha	44 ~~
Area of T-3 Drainage Basin	40.16	ha	~ ~
nnual Run-off from T-2 Drainage Basin	9,694	m3/yr	
nnual Run-off from T-3 Drainage Basin	66,666	m3/yr	
Iverage Flow from T-2 Drainage Basin	0.31	L/s	
Iverage Flow from T-3 Drainage Basin	2.11	L/s	

Table 7: T-2 and T-3 Drainage Basin Hydrology

* From: "Environmental Impact Assessment Describing Underground Mining at the Jasper Mine with Milling at the Star Lake Mill And Construction of a Haul Road", April 1989, Cameco, Vol. II, Appendix 4

** From: SMDC, 1985. Star Lake Project Tailings Pond Site Alternatives, Drawing Number 2.3.5

would be 5.62 kg.y⁻¹. With the total Cu content in the T-2 direct spray zone estimated added due to the treatment usage is 4.0 t (Table 4). Cu could be displaced for 714 years. For T-3, the estimated Cu leaving the area based on 1992 and 1994 data with a mean concentration of 0.1 mg.L⁻¹ would be 6.67 kg.y⁻¹. The total Cu content of T-3 at 1.2 t added due to spraying it would take 179 years and all the copper would be removed. If we assume that only about 50 % of the Cu can be mobilized the time frame would be lowered accordingly.

If on the other hand, each year some of the mobile Cu is converted into less mobile fractions, less Cu is displaced during spring run-off. During each summer a fraction of the Cu is converted to more stable Cu forms. Thus given that these processes can not be quantified, these predictions are unrealistic. Furthermore if it is considered that in northern Saskatchewan the spring run-off represent about 20 % of the annual run-off (snow pack La Ronge Airport), then copper removal would take longer accordingly. These consideration indicate, that although as an effluent treatment approach the muskeg spraying resulted in significant reduction in both Cu and CN, the design of the system should have taken removal processes and the Cu chemistry into account during operation. However it should be noted, that even during spring run-off the concentrations only slightly exceed the authorized levels, and that for most of the year the effluent concentrations are below these limits.

6.0 Conclusions

The distribution of total Cu in the muskegs used for treatment of gold mill effluents, along with the forms of Cu which are environmentally mobile, has been determined. Samples collected from different area and depth of the T-2 and T-3 muskeg have been subjected to EDS microscopy. This method determined that Cu is accumulated in bacterial cells but most frequently it is associated with organic material. Sequential extractions carried out on the samples suggested, that a large fraction of the copper **is** organically complexed 67 % in T-2 direct spray zones, but only about 7 % in T-3. Although, due to the low Cu concentration (2 mg.g⁻¹d.w.) in T-3 and the lower part of T-2, the sequential extractions account for only 20 % of the total Cu. The fraction of stable Cu in these locations increased to about 10 %.

It can be concluded based on the analytical results and the conditions (pH/Eh) that the formation of stable Cu precipitate is promoted. Thus, although the concentrations of Cu have increased in the muskegs (10 to 50 times above local background, Zone 4 T-2 and T-3), due to its use as an effluent treatment system, Cu it is not likely to be in very mobile form.

The copper concentrations determined for the control muskeg are within the expected range for sediments in mineralized areas as reported by Thornton (1979). In copper mineralized areas in England the concentration of Cu in sediments ranged from 42 mg.kg⁻¹ to 6500 mg.kg⁻¹ with a mean concentration of 1120 mg.kg⁻¹. As the Star Lake area is a gold mineralization the local background is considerably lower in Cu concentration. With the exception of the direct spray zone, the concentrations in zone 4 T-2 and T-3 range between 47 mg.kg⁻¹ and 2790 mg.kg⁻¹. It is therefore concluded, that the utilisation of the muskegs as a treatment system has elevated the muskeg areas into those concentration ranges which are typical for Cu mineralized areas.

Given the favourable economics and the potential environmental impact which would have been derived from utilisation of alternative treatment options, such as the production of treatment sludges, the approach used was environmentally sound. This is particulary the case as the spacial distribution of the Cu in the treatment system leads to the conclusion, that the forest soils in the direct spray zone were very effective in removing the colloidal Cu from the wastewater. Their capacity to remove organic complexed Cu was utilized to the capacity reported in the literature.

All of the copper which was added to the system through spraying (8.4 t), could be accounted for when Cu loads were estimated. This supports the conclusion that the Cu was retained, and effluent limits were met due to ongoing Cu removal processes. The performance was not a result of dilution in the drainage basin. Eighty five percent of the Cu removed was removed in the T-2 area, the first muskeg which received the effluent. Within this area 45 % of the Cu removed was retained in the T-2 'direct-spray' zone.

Although the Cu content of soils in T-2 'direct-spray' zone was approximately 2 % by weight, most of the Cu has remained in this material since its accumulation in 1989 to 1990. It is concluded that organic complexes due not break down. However, during spring run-off the pore-water is flushed resulting in the dissolution of organically complexed Cu. During summer months transformations to readily more stable precipitates of Cu take place.

The overall conclusion which can be derived from the present study is that the treatment approach could have been optimized. Cu was mainly removed by organic complexation, as demonstrated by the results obtained. The conversion of this copper to more stable Cu forms could have been promoted during operation of the system by promoting reducing conditions and supporting microbial activity.

Given the economics of the treatment approach and the environmentally acceptable result in meeting effluent limits during operation, the approach should be considered a success. The conclusion that the treatment resulted in retaining most of the added Cu in a defined area (mainly the direct spray zone) adds to the attractiveness of the approach. Furthermore, copper concentrations in the remainder of the muskeg are at the lower end of the concentration range reported for sediments in areas with copper mineralization thus not representing an environmental concern. Although during spring run-off some Cu is mobilized from the direct spray zone material, the effluent limits are met for the remainder of the year. In conclusion the treatment approach chosen appears to be environmentally sound and provides low operating and decommissioning costs, as expected from a passive treatment approach.

7.0 References

Bolis, J.L., Wildeman, T.R., and Cohen, R.R. 1991 The use of Bench Scale permeameters for preliminary analysis of metal removal from acid mine drainage by wetlands. Proc. 1991 National Meeting of the American Society for Surface Mining and Reclamation, Durango, Colorado, May 14-17, 1991.

Bupp, S. and Ghosh, S. 1991 Heavy metals uptake by microbial protein complexation. WPCF 64th Annual Conference and Exposition, Toronto, Oct. 7-10. Paper #AC91-025-003.

Chen, X., Gosset, T., and Thevenot, D.R. 1990 Batch copper binding and exchange properties of peat. Wat. Res., 24, 1463-1471.

Couillard, D. 1994 The use of peat in wastewater treatment. Wat. Res., 28, 1261-1274.

Demayo, A. and Taylor, M.C. 1981 Guidelines for surface water quality. Vol. 1: Inorganic Chemical Substances: Copper. Environment Canada, Ottawa. ISBN 0-662-11717-4.

Deng, Y. and Stumm, W. 1994 The reactivity of aquatic iron(III) oxyhydroxide -Implications for redox cycling of iron in natural waters. Appl. Geochem. **9**, 23-36.

Dvorak, D.H., Hedin, R.S., Edenborn, H.M. and Gustafson, S.L. 1991 Proc. of the 2nd. Int. Conf. on the Abatement of Acidic Drainage, Montreal, Vol. 1, pp 301-314.

Dvorak, D.H., Hedin, R.S., Edenborn, H.M. and McIntire, P.E. 1992 Treatment of metal-contaminated water using bacterial sulphate reduction: Results from pilot-scale reactors. Biotech. Bioeng. **40**, 609-616.

Eger, P. and Lapakko, K. **1988** Nickel and copper removal from mine drainage by a natural wetland. In 'Mine Drainage and Surface Mine Reclamation, Vol. 1: Mine Water and Mine Waste. USBM IC 9183, pp. 301-309.

Eger, P., Wagner, **J.R.**, Kassa, **Z.**, and Melchert, G.D. **1994** Metal removal in wetland treatment systems. Proceedings of Third Int. Conf. on the Abatement of Acidic Drainage, Pittsburgh, PA, April 24-29, 1994, Vol. 1, 80-88.

EPA (1993) SITE Emerging Technology Summary; Handbook for Constructed Wetlands Receiving Acid Mine Drainage. EPA/540/SR-93/523.

Ernst, R., Allen, H.E. and Mancy, K.H. **1975** Characterization of trace metal species and measurement of trace metal stability constants by electrochemical techniques. Water Res. **9**, 969-979.

Ferris, F.G., Schultze, **S., Witten, T.C.,** Fyfe, **W.S.** and Beveridge, **T.J. 1989** Metal interactions with microbial biofilms in acidic and neutral pH environments. Appl. Environ. Microbiol. **55**, 1249-1257.

Gallinger, R., Gormely, L., Kistritz, R. and Sobolowski, A. 1991 Constructed wetland experiment for treating AMD at Bell Copper. Proceedings 15th. Annual British Colombia Reclamation Symposium: Reclamation and Sustainable Development. Kamloops, June 24-28, 1991.

Gormely Process Engineering **1990** Assessment of wetlands for gold mill effluent treatment. Report for Saskatchewan Environment and Public Safety, Project No. 173-100.

Gormely, L., Higgs, T. and Kistritz, R. 1991 Wetlands for mine and mill effluent polishing. Proceedings of "Environmental Management for Mining: Annual
Saskatchewan Conference, Vol. 1."

Hammack, R.W., Dvorak, D.H., and Edenborn, H.M. **1994** Bench-scale test to selectively recover metals from metal mine drainage using biogenic H_2S . Proceedings of Third Int. Conf. on the Abatement of Acidic Drainage, Pittsburgh, PA, April 24-29, 1994, Vol. 1, 214-222.

Henrot, J. and Wieder, R.K. **1990** Processes of iron and manganese retention in laboratory peat microcosms subjected to acid mine drainage. J. Environ. Qual. 19: 312-320

Kalin, M. **1993** Treatment of acidic seeps using wetland ecology and microbiology. Final Report. CANMET project #:3.11.1, DSS #:015SQ.23440-2-92

Kersten, M. and Forstner, U. **1989** Speciation of trace elements in sediments. In G. E. Batley (ed.) "Trace Element Speciation: Analytical Methods and Problems", CRC Press, U.S.A. pp. 292-296.

McIntire, P.E., Edenborn, H.M. and Hammack, R.W. **1990** Incorporation of bacterial sulphate reduction into constructed wetlands for the treatment of acid and metal mine drainage. **1990** National Symposium on Mining, Lexington, Kentucky, May 14-18, 1990.

Machemer, S.D., and Wildeman, T.R. **1991** Organic complexation compared with sulphide precipitation as metal removal processes from acid mine drainage in a constructed wetland. J. Contam. Hydrol., **9**, 115-131.

MacMarlane, R. and Smith, D. **1989** The Star Lake mill, a low cost success story. Proc. 21st. Annual Meeting of the Canadian Mineral Processors. pp. 8-24. Mills, A.L., Bell, **P.E.** and Herlihy, A.T. **1989** Microbes, sediments and acidified water: The importance of biological buffering. In "Acid Stress and Aquatic Microbial Interactions", Ed. by S.S. Rao, CRC Press, Boca Raton, Florida, pp. 1-19.

Mullen, M.D., Wolf, D.C., Ferris, F.G., Beveridge, T.J., Flemming, C.A. and Bailey, G.W. 1989 Bacterial sorption of heavy metals. Appl. Environ. Microbiol. 55, 3143-3149.

Leckie, J.O., and Davis, J.A. **1979** Aqueous environmental chemistry of copper. In "Copper in the Environment, Part 1: Ecological Cycling", Ed. by J.O. Nriagu, John Wiley and Sons Inc., New York, pp. 89-121.

Petersson, **C.**, Hakansson, **K.**, Karlsson, **S.**, and **Allard**, **B. 1993** Metal speciation in a humic surface water system polluted by leachates from a mine deposit in Sweden. Wat. Res., **27**, 863-871.

Pickering, W.F. **1979** Copper retention in soil/sediment components. In "Copper in the Environment, Part 1: Ecological Cycling", Ed. by J.O. Nriagu, John Wiley and Sons Inc., New York, pp. 217-253.

Riveros, **P. 1992** Copper in Gold Plant Effluent (Saskatchewan MDA). Final Report. Work carried out by Cameco Corporation under the auspices of the Canada Centre for Mineral and Energy Technology, Energy, Mines and Resources Canada and the Federal Panel on Energy R & D (PERD). DSS Contract # 23440-1-9091/01-SQ.

Saeki, K., Okazaki, M., and Mutsumoto, **S. 1993** The chemical phase changes in heavy metals with drying and oxidation of the lake sediments. Wat. Res., **27**, 1243-1251.

Spear, P.A., and Pierce, R.C. **1979** Copper in the Aquatic Environment: Chemistry, Distribution and Toxicology, NRCC Associate Committee on Scientific Criteria for

Environmental Quality, National Research Council, Ottawa, NRCC No. 16454.

Stumm, W. and Morgan, J.J. 1981 Aquatic Chemistry. 2nd. Edition. Wiley, New York, 780 p.

Stumm, W. and Sulzberger, B. 1992 The cycling of iron in natural environments: Considerations based on laboratory studies of heterogenous substances. Geochim. Cosmochim. Acta, **56**, 3233-3257.

Thornton, I. 1979 Copper in soils and sediments. In "Copper in the Environment, Part 1: Ecological Cycling", Ed. by J.O. Nriagu, John Wiley and Sons Inc., New York, pp. 171-216.

Torma, A.E., Apel, M.L. and Brierley, C.L. (eds.) 1993 Biohydrometallurgical Technologies, Vol. II. Fossil Energy Materials, Bioremediation and Microbial Physiology. Proc. Int. Biohydrometallurgy Symposium, Jackson Hole, Wyoming, August 22-25, 1993, TMS Publications, Warrendale, Pennsylvania.

Volesky, B. 1990 Biosorption of Heavy Metals. CRC Press Inc., Boca Raton, FA.

Wetzel, R.G. 1992 Limnology, 2nd. edition. Saunders College Publishing, Philadelphia, 767 p.

Wittrup, M. and Nelson, J. 1992 Star Lake/Jasper Operation-1992 Muskeg Sampling Programme. Cameco, Environment and Safety Department

APPENDIX 1 BACKUP TABLES AND CALCULATIONS

Area						-2 Area							
Zone	iontrol, Z	Ione 7		Zone 1		lone 2		lone 3			one 4		
S.A., ha	per_m	2		0.67		0.13		0.74			1.24		
Station	CM-1	CM-2	T2-1	T2-2	T2-3	T2-5	T2-6	T2-4	T2-7	T2-8	T2-9	T2-10	T2-11
ayer 1, cm	0-28	0-36	0-5	0-5	0-5	0-10	0-26	0-5	0-53	0-57	0-55	0-4 9	0-37
Sample depth, m	0.28	0.38	0.05	0.05	0.05	0.1	0.26	0.05	0.53	0.57	0.55	0.49	0.37
Soil Vol, m3	0.28	0.38	335	335	335	125	325	369	6,569	7,065	6,817	6,073	4,566
Wet density	1.02	0.95	1.44	1.18	1.22	1.52	1.21	1.24	1.05	1.08	1.02	0.99	1.04
Wet/Dry ratio	3.65	4.58	2.55	2.46	2.75	1.32	2.27	2.65	4.45	2.91	4.32	3.95	4.5
Dry Soil wt, 1	0.078	0.079	189	161	149	144	173	173	1,550	2,622	1,609	1,522	1,060
[Cu], mg/dry Kg	15.4	38.2	3,440	22,200	20,100	747	2,000	78	80	1,220	1,300	1,280	2,080
t Cu in area	1.2E-06	3.0E-06	0.65	3.57	2.99	0.11	0.35	0.01	0.12	3.20	2.09	1.95	2.20
ayer 2, cm	28-128	38-138	5-10	5-10	5-10			5-10					
Sample depth, m	1	1	0.05	0.05	0.05			0.05					
Soil Vol, m3	1	1	335	335	335			369					
Wet density	1.01	0.96	1.18	1.13	0.77			0.62					
Wet/Dry ratio	23.06	33.44	2.46	2.98	2.67			2.72					
Dry Soil wt, t	0.04	0.03	161	127	97			84					
[Cu], mg/dry Kg	15.9	21.4	2,410	17,100	19,000			15,100					
t Cu in area	1.0E-06	5.1E-07	0.39	2.18	1.84			1.27					
u/area (t)	2.2E-06	3.6E-06	1.04	5.75	4.83	0.11	0.35	1.28	0.12	3.20	2.09	1.95	2.20
u in zone (t)				3.87		0.23		1.28			1.91		
u background (t)	2.9E-06	t/m2		0.020		0.004		0.021			0.036		1
u sprayed (t)						0.22		1.26			1.88		

Table A1: Estimated Cu loadings in the T-2 and T-3 areas based on soil/sediment Cu contents of samples collected in April 1994

Area		T-3 Area							
Zone		Zone 5				Zone 6			
S.A., ha		0.62				2.23			
Station	T3-3	T3-4	T3-5	T3-7	T3-1	T3-2	T3-6	T3-8	
ayer 1, cm	0-42	0-22	0-33	0-37	40-140	35-135	0-24	0-33	
Sample depth, m	0.42	0.22	0.33	0.37	1	1	0.24	0.33	
Soil Vol, m3	2,613	1,369	2,053	2,302	22,301	22,301	5,352	7,359	
Wet density	0.96	0.94	0.99	1	1	1.03	1.14	0.88	
Wet/Dry ratio	4.57	3.24	3.77	4.42	40.88	30.55	3.16	4.33	
Dry Soil wt, t	549	397	539	521	546	752	1,931	1,496	
[Cu], mgidry Kg	262	2,790	1,490	1,510	169	47	921	255	
t Cu in area	0.14	1.11	0.80	0.79	0.09	0.04	1.78	0.38	
ayer 2, cm			33-133	37-137			24-1 24	33-133	
Sample depth, m			1	1			1	1	
Soil Vol, m3			6,221	6,221			22,301	22,301	
Wet density			0.97	1.07			1	1.02	
Wet/Dry ratio			36.1	92.6			150.0	131.6	
Dry Soil wt, t			167	72			149	173	
[Cu], mg/dry Kg			103	196			53	375	
t Cu in area			0.02	0.01			0.01	0.06	
									Grand
u/area (t)	0.14	1.11	0.82	0.80	0.09	0.04	1.79	0.45	total (
u in zone (t)		0.72				0.59			8.60
u background (t)		0.018				0.065			
u sprayed (t)		0.70							8.44

able /	Star Lake	nplesof April 11-14, 1994 Desc	tion	d che	istry		<u></u>				
ample	Туре	Description	Wet	Wet	Dry	EDS	рН	Em	Cond.	Temp	LOI
Loc.			vol.	wt.	wt.	sample		mγ	mS/cm	С	
			(mL)	<u>(g)</u>	(g)	(cm)					
Control											
/luskeg											
CM-1	core	0-9cm ice				10-11,5					
		9-16cm - black peat, a few roots	125	127.3		25-26.{	6.54	103	98	11.8	
		16-28cm - brown peat, a few roots									
CM-I	soup		90	91.1	3.8		6.04	71	150	12.3	88.9
CM-2	core)-28cm - ice + few grassisedge leaves	175	188.9		6-7.5	8.43	123	110	18.9	
		28-38cm - peat with large roots				34-35.					
CM-2	soup		90	88.6	2.59		6.05	140	95	12.8	89.4
T2						-					
Auskeg											
T2-1	Grab 0-5cm		150	216.3			6.89	87	54	19.2	
T2-1	Grab5-10cm		150	176.7							
T2-2	Grab0-5cm		130	160.8			6.81	129	224	19.2	
T2-2	Grab 5-10cm		150	188.9			6.71	138	58	19.2	
T2-3	Grab0-5cm		110	134.5			6.62	133	32	19.2	
T2-3	Grab 5-10cm		205	157.2							
T2-4	Grab 0-5cm		120	148.5							
T2-4	Grab 5-10cm		300	187.2							
T2-5	Grab 0-10cm		500	761							
T2-6b	Core	0-3cm - ice, 3-8 cm peat	100	121.2		6-7.5	8.62	130	485	12.5	
		8-18cm · peat/clay, 18-26cm - clay									
T2-6	Grab										
T2-7	Core	3-23cm - coarse black peat with roots	260	273.2		6-7.5	8.47	61	364	18.7	
		23-53cm - fine black peal with roots				3 0-31.5					
T2-7	Grab										
T2-8	core	0-10cm - peat with roots	220	238.5			6.71	88	258	18.5	
		10-27cm- grey clay									
		27-57cm - black peat with roots									
T2-8	Grab										
T2-9	Core	0-32cm - black peat, a few roots	300	305.8		7-8.5	6.51	24	182	18.9	
		32-55cm -fine, black peat, a few roots				35-36.5					
T2-9	Grab										
T2-10	Core	0-16 cm - ice with a few twigs	177	175.5		18-20	6.81	5	252	18.8	
		16-49cm - black peat with roots				3 536.5					
T2-10	Grab										
T2-11	Core	0-10cm - ice with twig	200	207.2		10-11.5	8.32	54	136	10.5	
		10-23 cm - coarse fibrous peat									
		23-37cm - black peat, a few roots									
T2-11	Grab					3 0-31.5					

		1		i	1				l		
7.27	69	6E1	951	6.33		2.0	95.2	06		anos	8-81
									28-33cm - blackish peat		
								2	23-28cm - mainly ice, some peat and le		
	1.21	62	120	95.3	3.15-05		1.081	OZI	0-23cm - ice	Sore	8-E1
<u></u> ⊆'16	99	531	501	5.54		L	6.96	06		dnos	L-El
										ysnW	L-El
									30-37cm - brown peat with roots		
					34-32-2				16-30cm - black peat with roots		
	₽.81	562	52	88.8	G.8-7		182.5	182	or/stoods searb dfiw əbi dbum - mb ð l-0	Core	L-El
8'06	<u>8.5</u>	521	ELL	78.2		29.0	6'68	06		dnos	9-61
										цsnW	9-E1
					20-21.5				stoo r thiw lead - mɔəsːə		
	9.0T	316	16	84.8	G.8-7		8.81 T	001	0-4cm - peat	Core	9-E1
7 .06	3'81	506	60 L-	6.42		2.4	6.98	06		dnos	S-El
										ųsnįų	E-5T
					3.15-05				333cm - peat with sedge roots		
	61	872	131	6.45	9-7-8		6.641	125	90-3cm - ice	Core	S-El
										ųsnw	13-4
	3'8L	EZZ	126	67.8	G.T-ð		6.711	152	0-22cm - black peat	Core	t-£T
										ЧгиМ	E-EL
					33-34.5				16-42cm - coarse, brown peat with root		
					52-53.5				10-16cm - совгse, black peat with roots		
					11-01				səvsəl əgbəs\sssg diw əsi - mɔ01-ð		
	581	EEL	021	68. C	£.5-4		5.191	500	0-2cm - ice, 2-6cm- peat	Core	E-EL
16	15.3	132	68	51.8		3.02	97.6	06		dnos	2 - E l
										ЧзиМ	Z-El
6 18	9.11	540	53	26.8		72.2	668	06		dnos	1-El
											Bəysny
											El
					(ເພວ)	(6)	(6)	(ղա)			
	3	ພວ/ຣູພ	۸m		semple	,tw	-1W	.lov			.50J
101	meT	.bno-D	ᄢᆿ	Hq	EDS	Dry	teW	19W	Description	ed∕(⊺	Sample

Table A2: Star Lake Samples of April 11-14, 1994. Description and chemistry (continuation

APPENDIX 2 ASSAYER'S ICP DATA LABORATORY RAW DATA



Client.	Boojurn Resea 468 Queen St. Box 19 Toronto. ONT M5A 1T7	rch Ltd E Suite 400 C CANADA	Date Submitted: Date Reported: EPL Ref#: EPL Quote#: Client PO#:	April 2294 May 6/94 941681 verbal BROO684
Attn:	Mike Wanat		Ster Luke	
			51 0394. WKa	
			<u>[</u> 39. 2]	
		Certificate of Analy	ysis	
Analysis Per	formed:	18 element ICP S u n		
		Sulphur by ICP		
Methodolog	y:	 Analysis of trace metals in water by Plasma Spectrophotometry. U.S. EPA Method No. 200.7 (Ministry of Environment ELSCAN) Analysis of sulphur in water by Indu Plasma Spectrophotornetry. U.S. EPA Method No. 6010 (Ministry of Environment ELSCAN) 	 Inductively Coupled) uctively Coupled) 	
Instrumenta	tion:	1,2) Thermo Jarrell Ash ICAP 61E P	lasma Spectrophotorneter	
Sample Des	cription:	Water		
QA/QC:		Refer to CERTIFICATE OF QUALI	TY CONTROL report.	
Results:		Refer to REPORT of ANALYSIS atta Certified By J.N. Bishop Vice President, Stles and Certified By Certified By Certified By L. Munshaw, M.Sc., C. Cho Vice President, Laborator	ached Service em ry Services	

Environment Pro on Laboratories Inc.

Certificate of Quality Control

Client : Boojum Research Ltd Contact: Mike Wanat

May 6/94 941681 verbal BROO684

> EPL Quote#: EPL Ref # :

Client PO#:

Date Reported:

Analysis of Water

				Pro	cess Blar	¥	Proc	2058 % Re	KINA			ž	tric Spike	_		Overall
	SAMPLEID				Upper			DWOK	Upper				Lower	Upper		8
Parameter	(spike)	8	St ts	Result	Limit	Accept	Result	Limit	Limit	Accept	Result	Target	Limit	Limit	Accept	Acceptable
Aurthnum	4943 Blindman t.	0.025	mg/L	(q)pu	8	yes	104	8	120	yes	1.10	1.0	0.6	1.4	yes	yes
Barlum	đ	0.005	mg/L	(q) pu	0.01	yes	8	8	120	yes	٩ U	8 U	- 8 0	80	ĥà	yes
Beryllum	4943 Blindman L.	0.005	mg/L	(q)pu	0.01	yes	103	80	120	yes	1.17	1.0	0.6	1.4	yes	yes
Blemuth	4943 Hindman L	0.05	mg/L	(q) pu	0.1	yes	104	80	120	yes	1.07	1.0	0.3	1.7	yes	yes
Baron	4043 Bilndman L.	0.01	mg/L	(q) pu	0.02	yes	<u>8</u>	8	120	yes	1.06	1.0	0.6	4.1	y a s	yes
Cadmium	4943 Blindman L.	0.003	mg/L	(q) pu	0.006	yes	86	8	120	yes	1.07	0.1	0.6	4	yes	yes
Chromium	4943 Blindman L.	0.005	ng/L	(q)pu	0.01	yês	101	8	120	yes	1.10	1.0	0.6	1.4	yes	¥68
	4943 Blindman t.	0.005	mg/L	(q)pu	0.01	yes	104	8	120	yes	1.13	1.0	0.6	1,4	yes	yes
Copper	4843 Bilndman L.	0.003	mg/L	(q)pu	0.006	yes	103	8	120	yes	1.13	1.0	0.6	1,4	yes	yees
kon	4943 Blindman L.	0.005	mg/L	(q)pu	0.01	yes	67	8	120	yes	1.05	1.0	0.4	1.6	yes	yes
	4943 Blindman L	0.025	mg/L	(q)pu	0.10	yes	103	8	120	yes	1.11	1.0	4.0	1.6	yes	yes
Manganese	4943 Blindman L.	0.005	mg/L	(q)pu	0.01	yes	105	8	120	yes	1.14	1.0	0.6	1.4	yes	yes
Malybdenum	4843 Blindman L.	0.01	mg/L	(q) pu	0.03	y 0 8	97	8	120	yês	1.06	1.0	0.6	4.	y 0 3	yes
Nickel	4043 Bilndman L.	0.01	mg/t	(q)pu	0.02	yes	103	8	120	yes	1.11	1.0	0.6	4.1	yðs	yes
Bilver	2	0.003	mg/L	(q)pu	0.006	y e 8	101	8	120	yes	80	e U	ц В С	U.B.	쀨	88
Пл	4843 Blindman L	0.05	mg/L	(q) pu	0.15	yes	8	8	120	yes	1.06	0.1	4.0	1.6	ye 8	yees
Vanadium	4843 Bilndman L	0.01	mg/L	(q)pu	0.03	yes	8	8	120	yes	1.10	1.0	0.6	1,4	yes	500 X
Zine	4943 Blindman L	0.005	mg/L	(q)pu	0.02	yes	103	8	120	yes	1.13	1.0	0.6	4	y 8 8	yees
adphur a statement of the	an A	0.08	mg/L	(q)pu	0.16	yes	8	8	110	yes	80	ŝ	đ	đ	en	yes
			_										5	-		
00 = Limit of Quantitation = lower	st level of the para	meter the	it can be c	uantified 1	with confi	dence						A				
 — Unavaliable due to dilution re 	quired for analysis			_				- '	Authoriza	tion I.D.		1	N.			

LOQ = Limit of Quantitation = lowest level of the parameter that can be quantified with confidence
* = Unavailable due to dilution required for analysis
na = Not Applicable
nd = parameter not detected
nd = parameter not detected
ns = insufficient Sample Submitted
TR = trace level less than LOQ
TR = trace level less than LOQ
(b) = Analyte results on REPORT of ANALYSIS have been background corrected for the process blank.

Client : Boojum Research Ltd Contact: Mike Wanat

Report Date:	May 6/94
EPL Ref # 🗄	941681
EPL Quote #:	verbal
Client PO#:	BROO684

Analysis of Water

			4943	4940	4944-Control	4945-T3	
Parameter	100	Units	Blindman L.	Blindman L	muskeg CM-1	Area T3-5	
Date Sampled >			94/04/13	Replicate	94/04/13	W 1 3	
Aluminum	0.025	mg/L	0.099	0.0 99	0.095	0.197	
Barium	0.005	mg/L	0.036	0.035	0.011	nd	
Beryllium	0.005	mg/L	nd	nd	nd	nd	
Bismuth	0.05	mg/L	nd	nd	nd	nd	
Boron	0.01	mg/L	0.15	0.16	0.02	0.08	
Cadmium	0.003	mg/L	nd	nd	nd	nd	
Chromium	0.005	mg/L	nd	nd	nd	nd	
Cobalt	0.005	mg/L	nd	nd	nd	nd	
Copper	0.003	mg/Լ	0.141	0.145	0.017	0.062	
Iron	0.005	mg/L	0.521	0.514	0.277	0.372	
'Lead	0.025	mg/L	nd	nd	nd	nd	
Manganese	0.005	mg/L	0.330	0.335	0.018	nd	
Molybdenum	0.01	mg/L	0.03	0.03	nd	0.02	
Nickel	0.01	mg/L	0.01	0.02	nd	nd	
Silver	0.003	mg/L	nd	nd	nd	nd	
Sulphur	0.08	mg/L	28.0	26.3	1.13	2.55	
Tin	0.05	mg/L	nd	nd	nd	nd	
Vanadium	0.01	mg/L	nd	nd	nd	nd	
Zinc	0.005	mg/L	0.029	0.027	0.009	0.010	
1						\cap	
I							
1						./	
L						/_	
				Authorizatio	on I.D.	Alme	.

LOO = Limit of Quantitation = lowest level of the parameter that *can* be quantified with confidence.

nd = parameter not detected != LOO higher than listed due to dilution () Adjusted LOQ

SL	0394.WKQ [39.2]	EPL water ty	/ped May17-9	L	
1		===== 13.Mar.94	= = = = = = 13.Mer.94	= = = = = = = 13.Mar.94	
Ì	SAMPLE VOLUME	101101-04	10-11101-04	10-11121-04	
	ASSAYERS CODE	4943	4944	4945	
I	*********	=====			*====
	SAMPLING LOCATION	Star Lake	Star Lake	Star Lake	
		laka	musker	T3-5	
		23	CM-1		
	Processingcode	FA	FA	FA	
		化甲基苯苯二	*****		=====
	Temp. (C)				
	pH				
	Cond. (umhos/cm)				
	Eh (mV) Acidity (m.c./l)				
	Alkalinity (mg/l)				
	Ferric (Fe3 +)				
	Ferrous (Fe2+)				
	** I A R **	*************		·····	
	Temp. (C)	13.5	12.3	18.8	
	pH	6.79	6.04	6.42	
	Cond (umhos/cm)	567	150	206	
	Eh (mV) Acidity (mail)	76 7.0	71	109	
	Alkalinity (mg/l)	204	28.9	57.4	
	Ferric (Fe3+)				
	Ferrous (Fe2+)				
		======	==≃≈== 0003	===== 0.003	
1	Al	0.099	0 095	0.197	
ł	AS				
ļ	B	0.15	0 02	80.0	
1	Ba Ba	0.030	0.005	0.005	
i	Bi	0.05	0 05	0.05	
I	С				
	Ca	0.000	• •	0.002	
1	Ca	0.003	0 000	0 003	
İ	со	0.005	0.005	0 005	
ļ	Cr	0.005	0 005	0 005	
	C"	0.141	0 017	0 062	
	Ha	0.021	0211	0312	
ļ	K				
	La				
	Mg Mo	0.33	0.018	0.005	
	Мо	0.03	0 0 1	0 02	
ļ	Na				
	Nb	0.04	0.04	0.04	
ł	NI P	0.01	0 01	0.01	
İ	Pb	0.025	0 025	0 025	
ļ	S	28	11 3	2 55	
	Sb				
ł	Si				
i	Sn	0.005	0 005	0 005	
ļ	Sr				
	Te Th				
	Ti				
İ	Ŭ				
	V	0.01	0 01	0.01	
	W V				
İ	Zn	0.029	0 009	0.01	
I	Zr				



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Client:	Boolum Research Ltd	Date Submitted:	May 19/94
	468 Queen St. E. Suite 400	Date Reported:	June 2/94
	Box 19	EPL Ref#:	941928
	Toronto, ONT, CANADA		
	M5A 1T7	Client PO#:	BROO686
Attn:	Mike Wanat		

Certificate of Analysis

Analysis Performed	18 element ICP Scan , Digestion Required Sulphur by ICP. Digestion Required
	Moisture Content
√ethodology:	 Analysis of trace metals in soil by Inductively Coupled Plasma Spectrophotometry. U.S. EPA Method No. 6010 (Ministry of Environment ELSCAN) Analysis of sulphur in soil by Inductively Coupled Plasma Spectrophotometry. U.S. EPA Method No. 6010
	 (Ministry <i>cf</i> Environment ELSCAN) 3) Acid digestion of <i>s o 5</i> for metals determination by inductively coupled plasma atomic emission spectrometry and/or flame or furnace atomic absorption spectroscopy. U.S. EPA Method No. 3050(Modification) 4) Determination of the moisture content of soil by weight. ASTM Method No. D2216-80



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Client:	Boojum Research Ltd	Date Submitted:	May 19194
	468 Queen St. E. Suite 400	Date Reported:	June 2/94
	Box 19	EPL Ref#:	941928
	Toronto, ONT, CANADA		
	M5A 1T7	Client PO#:	BR00686
Attn:	Mike Wanat		

Certificate of Analysis

Instrumentation:	 1, 2) Thermo Jarrell Ash ICAP 61E Plasma Spectrophotometer 3) Thermolyne Hotplate/Hot Block 4) Precision Mechanical Convention Oven/Sartorius Basic Balance
Sample Description:	soil
QA'QC	Refer to CERTIFICATE OF QUALITY CONTROL report.
Results:	Refer to REPORT of ANALYSIS attached.

Certified J.N. Bishop vice President, Sales and Service Mul Certified By

F. Munshaw, M.Sc., C.Chem
 Vice President, Laboratory Services

Certificate of Quality Control

Client : Boojum Research Ltd Contact: Mike Wanat
 Date Reported:
 June 2/94

 EPL Ref # :
 941928

Client PO#:

llMu

Authorization I.D.

BROO686

Analysis of Soil, expressed on a dry weight basis

				F	ess B	¢	Pn	63 %	overy		Matrix Spike					Overail
	SAMPLE ID				Upper			Lower	Jpper				DWO	Upper		QC
Parameter	(spike)	LOQ	Units	Result	Limit	Чссер	Result	M	Limit	locept	Accult	larget	Limit	Umil	ссер	Acceptable
Aluminum	4986	1.0	rnghg	nd (b)	4.0	yes	102	80	120	yes	286	250.0	150.0	350.0	yea	yes
Aluminum	5001	1.0	mghg	nd (b)	4.0	yəs	102	80	120	yes			*		*	yes
Aluminum	5016	1.0	rnghg	nd (b)	4.0	yes	100	80	120	yes	301	250.0	150.0	350.0	yes	yes
Barlum	ne	0.2	mghg	nd (b)	0.6	yes	100	80	120	yes	na	na	na	па	na	y o s
Barium	na -	0.2	mg/kg	nd (b)	0.6	yes	101	80	120	yes	na	na	na	กล	na	yes
Barium	na.	0.2	mg/kg	nd(b)	0.6	yes	101	80	120	yes	na	na	na	na	na	yes
Beryllum	4986	0.5	mg/kg	nd (b)	1.5	yes	103	80	120	yes	280	250.0	100.0	450.0	уөв	yes
Beryllium	5001	0.5	mg/kg	nd (b)	1.5	yes	103	80	120	yes	267	250.0	1M.O	450 .0	yes	yes
Berytlum	5018	0.5	mg/kg	nd (b)	1.5	yəs	100	80	120	yes	273	250.0	100.0	450.0	yes	yes
Biemuth	na	2.5	mg/kg	4.3(b)	7.5	yes	105	80	120	yes	na	ាង	na	na	na	yes
Blamuth	ne	2.5	rnghg	4.3(b)	7.5	yes	105	80	120	yes	na	na	na	na	na	yes
Bismuth	na	2.5	rnghg	4.3(b)	7.5	yes	99	80	120	yəs	na	na	na	na	na	yes
Boron	4986	0.5	mg/kg	nd (b)	4.0	yes	100	80	120	yes	257	250.0	150.0	350.0	yes	yes
Boron	5001	0.5	mg/kg	nd (b)	4.0	yes	100	80	120	yes	247	250.0	150.0	350.0	yes	yes
Boron	5016	0.5	mg/kg	nd(b)	4.0	yes	101	80	120	yes	250	250.0	150.0	350.0	yes	yes
Cadmium	4960	0.5	mg/kg	(d) bn	2.0	yes	103	80	120	yəs	264	250.0	150.0	350.0	yes	yes
Cadmium	5001	0.5	mghg	nd(b)	2.0	yes	103	80	120	yəs	256	250.0	150.0	350.0	yes	yes
Cadmium	5018	0.5	rnghg	nd(b)	2.0	yes	101	80	120	yes	257	250.0	150.0	350.0	yes	yes
Chromium	4986	0.3	mg/kg	nd (b)	0.9	yes	101	80	120	yes	274	250.0	150.0	350.0	yes	yes
Chromium	5001	0.3	mg/kg	nd (b)	0.9	yes	101	80	120	yes	270	250.0	50.0	350.0	yes	yes

LOO = Limit of Quantitation = lowest level of the parameter that can be quantified with confidence

Unavailable due to dilution required for analysis

na = Not Applicable

nd = parameter not detected

ns = Insufficient Sample Submitted

TR = trace level less than LOQ

(b) = Analyte results on REPORT of ANALYSIS have bean background corrected for the process blank.

Certificate of Quality Control

Client : Boojum Research Ltd Contact: Mike Wanat

Date Reported: June 2/94 EPL Ref # : 941928

BROO686

Client PO#:

Mue

Authorization I.D.

Analysis of Soil, expressed on a dry weight basis

				Process Blank			Process % Recovery				Matrix Spike				Overall	
[SAMPLE ID				kpper			Lower	Joper				Lower	Upper		0 C
Parameter	(spike)	LOQ	Units	Result	Limit	Accept	Result	Limit	Limit	locep'	Result	Targel	Limit	Limit	locept	Acceptable
Chromium	5016	0.3	mg/kg	nd (b)	0.9	yes	101	80	120	yes	268	250.0	150.0	350.0	yes	yes
Cobell	4986	0.3	mg/kg	(d) bn	0.9	yes	104	80	120	yes	280	250.0	150.0	350.0	yes	yes
Cobell	5001	0.3	mg/kg	nd (b)	0.9	yes	104	80	120	yəs	273	250.0	150.0	350.0	yes	yes
Cobalt	5016	0.3	mg/kg	nd (b)	0.9	yes	100	80	120	yes	272	250.0	150.0	350.0	yes	yes
Copper	4986	0.2	mg/kg	nd(b)	0.8	yəs	103	80	120	yes	277	250.0	150.0	350.0	yes	yes
Copper	5001	0.2	mg/kg	nd (b)	0.8	yes	103	80	120	yes	311	250.0	150.0	350.0	yes	yea
Copper	5016	0.2	mg/kg	nd (b)	0.8	yes	101	80	120	yəs	274	250.0	150.0	350.0	yes	yes
kon	4986	0.3	mg/kg	(d) bn	1.2	yes	103	80	120	yes	257	250.0	150.0	350.0	yes	yes
Iron	5001	0.3	mg/kg	(d) bn	1.2	yes	103	80	120	yes			-	*	+	yes
Iron	5016	0.3	mg/kg	nd (b)	1.2	yes	103	80	120	yes	273	250.0	150.0	350.0	yes	yes
Lead	4986	1.0	mg/kg	nd (b)	4.0	yes	103	80	120	yes	285	250.0	150.0	350.0	yes	yes
Lead	5001	1.0	mg/kg	nd(b)	4.0	yes	103	80	120	yes	276	250.0	150.0	350.0	yes	yes
Lead	5016	1.0	mg/kg	nd (b)	4.0	yes	103	80	120	yes	275	250.0	150.0	350.0	yes	yes
Manganese	4966	0.3	mg/kg	nd (b)	0.9	yes	101	80	120	yes	281	250.0	100.0	450.0	yes	yes
Manganese	5001	0.3	mg/kg	nd(b)	0.9	yes	101	80	120	yes	280	250.0	100.0	450.0	yes	yes
Manganese	5016	0.3	mg/kg	nd (b)	0.9	yes	101	80	120	yes	274	250.0	100.0	450.0	yes	yes
Molybdenum	4958	0.5	mg/kg	nd (b)	1.5	yes	97	80	120	yes	265	250.0	150.0	350.0	yes	yea
Molybdenum	5001	0.5	mg/kg	nd (b)	1.5	yes	97	80	120	yes	257	250.0	150.0	350.0	yes	yes
Molybdenum	5016	0.5	mg/kg	nd(b)	1.5	yes	101	80	120	yes	258	250.0	150.0	350.0	yes	yes
Nickel	4986	0.5	mg/kg	nd(b)	1.5	yes	102	80	120	yes	281	250.0	1\$0.0	350.0	yes	yes

LOQ = Limit of Quantitation = lowest level of the parameter mat can be quantified with confidence * = Unavailabledue to dilution required for analysis

na = Not Applicable

nd = parameter not detected

ns = insufficient Sample Submitted

TR = trace level less than LOQ

Certificate of Quality Control

Date Reported: 1une 2/94

EPL Ref # : 876IÞ6

BROO86

Client PO#:

muj

Contact: Mike Wanat Client : Boojum Research Ltd

Analysis of Soil, expressed on a dry weight basis

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[(ľ	[1	1			
Aez	180	ទប	망내	180	eu	۸ea	150	06	66	Nea	0.8	(q) pu	6җ/6ш	0.4	19U	inyding
Aea	16 0	BN	មហ	19U	60	Nez	150	06	101	Nes	08	(q)pu	6ჯ/6ლ	0.4	ສບ	intqins
Aes	TU	សា	1 VU	1 80	80	yes	150	06	001	sey	0.8	(q)pu	6ჯ/ნდ	0.4	Ψu	nurqu 8
yes	seÁ	320.0	0.021	oosz	510	Nea	150	08	201	Nea	1.2	(q)pu	6ෳ/6ຒ	€0	9109	201Z
Aes	SBÁ	320.0	0.021	oosz	512	Nee	150	08	Z01	yes	5.1	(q)pu	ອີສ/ອີພ	6.0	1005	Sinc
Nez	Sev	320.0	0051	520.0	922	Nea	150	08	105	Nea	5.1	(q)pu	8җ/θω	6.0	4069	Zinc
Xeş 🔰	seA	320.0	0 [.] 0⊆1	520.0	566	Nea	150	08	001	Aea	SI	(q)pu	მჯ/მლ	so	8105	mulberreV
Nez	Aea	320.0	0.021	oosz	566	səx	150	08	100	sey	S'L	(q)pu	ßאូ∕6ພ	S.0	1005	mulbereV
sax	SeA	320.0	0.021	O V	512	yes	150	08	001	sey	S.1	(d)bn	6ෳ/6ѡ	5 .0	9967	mulbered
Nez	səA	320.0	0051	250.0	892	səA	150	08	9 6	ves	8.T	(q)pu	ნჯ/მա	s z	9105	비
seX	Aes	320.0	0051	oosz	560	Хөж	150	08	86	Aea	S 2	(q)pu	6x/6w	5.5	1005	11v
sex) Aec	390.0	0.021	oosz	992	Nez	150	08	86	204	6.T	(d)bn	B ₁ /6w	sz	998+	ntī
yes	an a	6Ô	80	1 80	80	Nez	150	08	Z01	sek	8.1	(q)pu	6x/6w	so	8 0	JAVIS
s a A	181	ອນ	80	180	19U	Aea	150	09	201	Aea	Sit	(q)pu	ິ 6)(6)	S.0	νu	Silver
Aea	180	BN	ខា	บซ	18N	sey	150	08	101	yes.	9° L	(q)pu	6x/6w	so	ອບ	SHARE
Nez	Nez	360.0	0.021	oosz	574	sek	150	08	201	yea	ð.t	(q)pu	6 җ/ɓ ա	so	8105	Michael Michael
Sex	Sek	320.0	0'0 <u>\$</u> 1	00\$Z	922	sek	150	08	201	Aea	<u><u> </u></u>	(q)pu	ნෳ/8ຒ	SO	1005	
eldistressA	Accept	hmil	timiJ	1aguel	flueeff	(tdecaty	timi.)	hmil	hueefi	10eoor	hmil	flusefi	ainU	TO0	(ଚ୍ଞ୍ୟୁପଟ)	Parameter
oc		Noper	TOWER	[Upper	Lower			<u>əddn</u>				SAMPLE ID	
ILENAVO			HINGS 20.4	PW			ARANOOR	H % 5590	2014	K	3	Hd				

LOO = Limit of Quantitration = lovel tree parameter that can be quantitration of the parameter that an to the parameter of th

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= parameter not detected pu

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Client : Boojurn Research Ltd Contact: Mike Wanat

Report Date:	June 2/94
EPL Ref # :	941928

Client PO#: BROO686

Analysis of Soil, expressed on a dry weight basis

Jameter	LOQ	Units	4965	4966 Beoficste	4967	4988	4989
ມໝໍຍູເຫ	10	maka	1410	1390	2170	1660	4720
arium		maka	29.9	3050 30 1	63.0	51 4	4720 148
andra	0.2	mo/ko	30.0 pd		0.00	51.4 nd	nd
is muth	0.5	mg/kg mg/kg	nd		nd	nd	od
	0.5	mo/ka	nd	od od	nd	nd	od
admium	0.5	mo/ko	nd	nd	nd	nd	nd
bronium	0.0	mo/ko	42	31	4.6	33	96
obait	0.0	mo/ka	22	22	19	1.4	35
	0.0	mo/ko	15.4	159	103	26.2	214
20	0.2	ma/ka	2830	2780	3600	4470	217 0000
ad .	0.5 1.0	ma/ka	166	12.0	od od	97.2	212
lancanese	03	mo/ka	189	189	104	340	3530
lolybdenum	0.0		nd	nd	nd	nd	51
ickel	0.5	mo/ka	22	25	1.4	3 7	<u>a 1</u>
ilver	0.5	ma/ka	nd	nd	nd	nd	od
ulphur	4.0	ma/ka	3140	3320	3140	2880	4740
in	25	ma/ka	nd	nd	or 10	bo	nd
anadium	0.5	ma/ka	38	21	53	43	68
inc	0.0	ma/ka	15.8	15.5	90	58.6	23.4
loisture Cantent	0.01	s	na	na	na	na	na
						\wedge	
				Authorizatic	an I D.	NIMU O	

LOO = Limit of Quantitation = lowest level of the parameter that can be quantified with confidence.

na = Not Applicable

nd = parameter not detected ! = LOQ higher than listed due to dilution () Adjusted LOQ

Client : Boojum Research Ltd Contact: Mike Wanat
 Report Date:
 June 2/94

 EPL Ref # :
 941928

Client PO#: BROO686

Analysis of Soil, expressed on a dry weight basis

Parameter	ΩQ	Units	4990	4991	4982	4983	4904
Aluminum	1.0	mg/kg	8540	8720	5300	3750	4280
Barium	0.2	mg/kg	91.2	92.9	113	93.3	106
Beryllium	0.5	mg/kg	nd	nd	nd	nd	nd
Bismuth	2.5	mg/kg	nđ	nd	121	86.9	W.4
Boron	0.5	mg/kg	nd	2.9	6.9	7.6	23.8
Cadmium	0.5	mg/kg	nd	nd	nd	0.6	0.6
Chromium	0.3	mg/kg	33.6	31.2	10.7	7.2	10.4
Cobalt	0.3	mg/kg	6.9	8.2	8.9	7.6	9.5
Copper	0.2	mg/kg	3440	2410	22200	17100	20100
iron	0.3	mg/kg	12800	11000	8430	5750	7220
Lead	1.0	mg/kg	88.7	45.3	365	254	265
Manganese	0.3	mg/kg	337	257	464	349	577
Molybdenum	0.5	mg/kg	nd	nd	3.3	10.1	nd
Nickel	0.5	mg/kg	W.9	64.3	264	244	360
Silver	0.5	mg/kg	3.2	2.1	10.8	6.6	10.2
Sulphur	4.0	mg/kg	1500	1500	1760	1460	1920
Tin	2.5	mg/kg	nd	nd	nd	nd	nd
Vanadium	0.5	mg/kg	27.2	25.8	14.5	10.1	12.7
Zinc	0.3	mg/kg	84.6	62.0	115	93.4	127
Moisture Content	0.01	%	na	na	na	na	na
						n	
			* *			11	

LOQ = Limit of Quantitation = lowest level of the parameter that can be quantified with confidence

na = Not Applicable

nd = parameter not detected ! = LOO higher than listed due to dilution () Adjusted LOO

Client : Boojum Research Ltd Contact: Mike Wanat

Report Date:	June 2/94
EPL Ref # :	941928

Client PO#: BROO686

Analysis of Soil, expressed on a dry weight basis

Parameter	L00	Units	4995	4996	4907	4998	4000
Aluminum	1.0	mghg	6870	506	2750	5850	7940
Barium	0.2	mg/kg	105	55.9	96.9	40.6	79.5
Beryllium	0.5	mghg	nd	nd	nđ	nd	nd
Bismuth	2.5	mg/kg	91.2	nd	82.7	nd	nd
Boron	0.5	mghg	7.0	nd	2.8	nd	nd
Cadmium	0.5	mghg	nd	nd	nd	nđ	nđ
Chromium	0.3	mghg	16.5	1.6	3.6	24.2	32.1
Cobalt	0.3	mghg	12.9	2.9	9.0	6.0	9.6
Copper	0.2	mghg	19000	78.0	15100	747	2000
iron	0.3	mg/kg	12100	799	4660	9350	12600
Lead	1.0	mghg	443	20.9	43.6	14.7	52.9
Manganese	0.3	mg/kg	432	336	235	228	276
Molybdenum	0.5	mghg	2.4	nd	nd	nđ	nd
Nickei	0.5	mg/kg	258	5.4	221	31.6	53.8
Silver	0.5	mghg	8.7	0.5	1.4	nd	2.2
Sulphur	4.0	mg/kg	2160	1530	1300	637	1820
Tin	2.5	mg/kg	nd	nd	nd	nd	nd
Vanadium	0.5	mghg	19.0	27	5.8	16.3	28.0
Zinc	0.3	mg/kg	131	48.0	55.9	41.4	67.4
Moisture Content	0.01	%	ne	na	na	na	na
						0	
				Authorizatio	on I.D.	Alm	

LOQ = Limit of Quantitation = lowest level of the parameter that can be quantified with confidence

na = Not Applicable

nd = parameter not detected != LOO higher than listed due to dilution () Adjusted LOO

Client : Boojum Research Ltd Contact: Mike Wanat

Report Date:	June 2/94
EPL Ref # :	941928

Client PO#: BROO686

Analysis of Soil, expressed on a dry weight basis

	······						
Parameter	.00	Units	5000	5001	5001 Replicate	5002	5003
Aluminum	1.0	mg/kg	3220	8130	8620	5100	4090
Barium	0.2	m g/kg	63.8	n . 4	82.7	81.1	125
Beryllium	0.5	mg/kg	nđ	nd	nd	nd	nd
Bismuth	2.5	mg/kg	nd	nd	nd	nd	nd
Boron	0.5	mg/kg	nd	nd	nd	nd	2.7
Cadmium	0.5	mg/kg	nd	nd	nd	nđ	nd
Chromium	0.3	mg/kg	6.3	33.9	36.6	4.9	10.7
Cobalt	0.3	mg/kg	1.0	10.9	11.4	3.1	5.1
Copper	02	mg/kg	60.2	1220	1300	1300	1280
Iron	0.3	mg/kg	2510	12900	13600	3410	3220
Lead	1.0	mg/kg	13.0	37.3	41.3	21.5	24.3
Manganese	0.3	mg/kg	125	269	284	62.3	504
Molybdenum	0.5	mg/kg	nd	3.1	2.0	nd	9.2
Nickel	0.5	mg/kg	4.1	45.2	50.9	32.7	43.8
Silver	0.5	mg/kg	nd	nd	nd	nd	27
Sulphur	4.0	mg/kg	3870	1800	1930	2860	4550
Tin	2.5	mg/kg	nd	nd	nd	nd	nđ
Vanadium	0.5	mg/kg	5.4	28.8	30.3	4.0	4.6
Zinc	0.3	mg/kg	8.9	75.5	79.4	17.4	21.6
Moisture Content	0.01	%	na	na	na	na	na
						\cap	

LOQ = Umit of Quantitation = lowest level of the parameter that can be quantified with confidence

na = Not Applicable

nd = parameter not detected != LOO higher than listed due to dilution () Adjusted LOO

Client : Boojum Research Ltd Contact: Mike Wanat

Report Date:	June 2/94
EPL Ref # :	941928

Client PO#: BROO686

Analysis of Soil. expressed on a dry weight basis

Parameter	100	Units	5004	5005	5006	5007	5008
Aluminum	1.0	mg/kg	4460	3060	4950	4320	3050
Barium	0.2	mg/kg	116	38.7	46.6	68.8	79.0
Beryllium	0.5	mg/kg	nd	nd	nd	nd	nd
Bismuth	2.5	mg/kg	nd	nd	nd	nd	9.7
Boron	0.5	mg/kg	5.3	1.6	nd	nd	3.5
Cadmium	0.5	mg/kg	nd	nd	nd	nd	nd
Chromium	0.3	mg/kg	7.7	6.2	6.4	4.7	3.9
Cobalt	0.3	mg/kg	9.4	6.0	1.9	4.3	1.3
Copper	0.2	mg/kg	2060	169	46.6	262	2790
Iron	0.3	mg/kg	8990	7810	4070	3090	3980
	1.0	mg/kg	27.5	19.7	8.6	48.5	10.1
	0.3	mg/kg	565	492	w.7	321	103
1	0.5	mg/kg	76	7.6	0.6	8.8	nd
	0.5	mg/kg	62.8	12.9	4.9	16.0	62.3
	0.5	mg/kg	2.7	nd	nd	1.5	3.2
	4.0	mg/kg	4590	4650	2750	4140	3680
	2.5	mg/kg	nd	nd	nd	nd	nd
	0.5	mg/kg	11.9	11.0	11.7	6.6	2.9
	0.3	mg/kg	321	22.7	16.9	35.4	28.2
Moisture Content	0.01	%	na	na	na	na	na
				Authorizatio	no I.D.	N.Mus	

LOO = Limit of Quantitation = lowest level of the parameter that can be quantified with confidence.

na = Not Applicable

nd = parameter not detected ! = LOQ higher than listed due to dilution () Adjusted LOQ

Client : Boojum Research Ltd Contact: Mike Wanat

Report Date:	June 2/94
EPL Ref # :	941928

Client PO#: BROO686

Analysis of Soil. expressed on a dry weight basis

Parameter	LOQ	Units	5009	5010	5011	5012	5013
uminum	1.0	mg/kģ	1810	3370	2030	4030	1790
arium	0.2	mg/kg	44.4	47.7	56.9	55.2	54.4
eryllium	0.5	mg/kg	nd	nd	nd	nd	nd
ismuth	2.5	mg/kg	nd	nd	nd	nd	nd
oron	0.5	mg/kg	6.5	1.4	6.0	nd	6.5
admium	0.5	mg/kg	nd	nd	nd	nd	nd
hromium	0.3	mg/kg	2.0	3.5	3.6	4.0	1.5
obalt	0.3	mg/kg	3.8	3.5	3.5	1.2	4.5
opper	0.2	mg/kg	1490	103	921	53.4	1510
on	0.3	mg/kg	2340	2100	2520	2710	2220
ead	1.0	mg/kg	6.1	13.3	15.2	nd	13.5
langanese	0.3	mg/kg	119	78.6	186	67.2	173
kiybdenum	0.5	mg/kg	10.2	5.5	22.4	nd	36.0
ickel	0.5	mg/kg	51.3	5.3	30.1	2.5	54.7
itver	0.5	mg/kg	nd	nd	2.0	nd	nd
ulphur	4.0	mg/kg	3260	3070	47 m	2700	5590
in	2.5	mg/kg	nd	nd	nd	nd	nd
anadium	0.5	mg/kg	4.0	5.7	3.6	6.5	2.9
inc	0.3	mg/kg	54.6	10.7	27.1	25.9	40.1
loisture Content	0.01	%	na	na	na	na	na
						\int	
				Authorizatio	on I.D.	_ <i>[] \</i> 114 _	

LOQ = Limit of Quantitation = lowest level of the parameter that can be quantified with confidence.

na = Not Applicable

nd = parameter not detected ! = LOQ higher man inect due to dilution () Adjusted LOQ

Client : Boojum Research Ltd Contact: Mike Wanat

Report Date:	June 2/94
EPL Ref # :	941928

Client PO#: BROO686

Analysis of Soil, expressed on a dry weight basis

arametei	LOQ	Units	5014	5015	5016	5018 De alla sta	
						нерисале	
Juminum	1.0	mg/kg	3190	2160	4360	4420	
larium	0.2	mg/kg	€6.4	49.0	50.1	50.6	
Jeryllium .	0.5	mg/kg	nd	nd	nd	nd	
lismuth	2.5	mg/kg	nd	nd	nd	nd	
bron	0.5	mg/kg	nd	nd	1.9	1.4	
admium	0.5	mg,kg	nd	nd	nd	nd	
Shromium	0.3	mg/kg	1.4	2.5	8.9	8.1	
20balt	0.3	mg/kg	0.4	13	3.0	3.4	
pper	0.2	mg/kg	196	256	375	380	
'on	0.3	mg/kg	2070	7300	3810	3850	
.ead	1.0	mg/kg	nd	25.0	20.0	21.8	
Manganese	0.3	mg/kg	98.6	206	96.2	97.3	
Aolybdenum	0.5	mg/kg	5.3	2.6	10.3	10.8	
fickel	0.5	mg/kg	9.3	14.9	14.2	16.3	
Silver .	0.5	mg/kg	nd	nd	nd	nd	
Julphur	4.0	mg/kg	2450	2330	4110	4360	
'in	2.5	mg/kg	nd	nd	nd	nd	
/anadium	0.5	mg/kg	3.3	4.6	8.4	9.2	
Sinc .	0.3	mg/kg	18.9	44.7	28.5	29.4	
Moisture Content	0.01	%	[,] na	na	na	na	
						$ \cap$	
						$N \cdot$	
				Authorizatio		I Same	

LOQ = Limit of Quantitation = lowest level of the parameter that can be quantified with confidence

na = Not Applicable

nd = parameter not detected ! = LOQ higher than listed due to dilution () Adjusted LOQ

SAMPLE DATE	12-Apr-94	12-Apr-94	12-Apr-94	12-Apr-94	12-Apr-94	12-Apr-94	12-Apr-94	12-Apr-
SAMPLE VOLUME ASSAYERS CODE	4986	4967	4958	4989	4990	4991	4992	49
			****				****	****
SAMPUNG LOCATIO	Star Lake	Star Lake	Star Laka	Star Lake	Star Laka	Star Lake	Star Lake	Star Lal
	Control	Control	Control	Control	12 Wetland	12-Wetland	72-Wetland	T2-Wet
	maskeg	maskeg	maskeg	maskeg	T2-1 grab	T2-1 grab	T2-2 grab	12-2 an
	CM-1 core	CM-1 soup	CM-2 com	CM-2 soup	0- 5c m	5-10cm	0-5cm	5-10ci
Processing code	SS	SS	55	55	S \$	SS	55	SS
***********	*****	==±±±	* = = * *	*****	*****		****	****
Terro (C)								
oH								
Cond. (umhowern)								
Eh mV)								
Acidity (mg/l)								
Alkalinity (mg.1)								
Ferric (Fe3+)								
Ferrous (Fe2+)								
** LAB **		• • • • • • • • • • • • • • • • • • • •				-		
Temp (C)								
рН								
Cond. (umhos/cm)								
Eh (mV)								
Acidity (mg/l)								
Alkaiinity (mg/l)								
Female (Fe3+)								
ELEMENTS An	0.5	05	0.5	0.5	32	21	10.5	
A	1410	2170	1880	4720	5640	8720	5330	37
As								
8	0.5	05	0.5	05	0.5	2.9	5.9	7
Ba	38.8	63	51.4	148	91.2	92.9	113	93
Be	0.5	0.5	0.5	05	0.5	05	0.5	0
Bi	2.5	25	2.5	2.5	2.5	2.5	121	36
Ca								
Cal	0.5	0.5	9.5	0.5	0.5	0.5	0.5	0
Ce								
လိ	2.2	1.9	1.4	3.5	8.9	5.2	8.9	7
r. 	4.2	4.6	3.3	9.6	33.6	31 2	10.7	7
Çu -	15.4	19.3	36.2	214	3440	2410	22200	171
26 11-	28.30	3000	447U	3900	12000	11000	5430	5/:
, iĝ								
,, i a								
Mo								
Mn	189	104	340	3530	337	257	464	3
Mo	0.5	0.5	0.5	\$.t	0.5	0.5	3.3	10
Na								
Nb								
Ni	22	1.4	3.7	9.1	54.9	64.3	264	24
P			*					
24 C	16.6	1	92,7	21.2	55.7	45.3	365	2
د ۲	3140	3140	2080	4/40	FOLU	1000	1750	140
Si								
Sn	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2
Sr						-	-	-
T∎								
Th								
. 1								
v	5.8	53	4.3	5.8	27.2	25.6	14.5	10
Ň	÷.÷						•••	
	15.8	9	58.9	23.4	84.6	62	1 15	93
Zn								

SL0494S.WKQ [39.2]									
*********			=====	**==				*====	
SAMPLE DATE	12-Apr-94	12-Apr-94	12-Apr-94	12-Apr-94	12-Apr-94	12-Apr-94	12-Apr-94	12-Apr-94	12-Apr-94
ASSAYERS CODE	4994	4995	4996	4997	4998	4999	5000	5001	\$002
SAMPLING LOCATIO	Star Lake T2-Wetland T2-3 grab 0-5cm	Star Lake 72-Wetland T2-3 grab 5-10cm	Star Lake T2-Wetland T2-4 grab 0-5cm	Star Lake T2-Wetland T2-4 grab 5-10cm	Star Lake T2-Wetland T2-5 grab 0-10cm	Star Lake 72-Wetland 72-8 core	Star Lake T2-Wetland T2-7 core	Star Lake T2-Wetland T2-8 core	Star Lake T2-Wetland T2-9 core
Processing code	55	55	55	55	55	SS	SS	SS	SS
** FIELD ** I Temp. (C) I pH		**				*****		= = = = = = = = =	===#==
Eh (mV) Acidity (mg/l) Alkalinity (mg/l) Ferric (Fe3+) Ferrous (Fe2+)									
** L A B ** Temp. (C) pH Cond. (umhos/cm) Eh (mV) Acidity (mg/l) Alkalinity (mg/l) Ferric (Fe3+) Ferrous (Fe2+)									
	==≠===	로운 또 드 크 크 0 7	=====	======	*****	======	==는크로제 0.5		========
	4280	6870	506	2750	5850	2.2 7940	3220	0. 55 8130	0.5 51.00
I AS	.200	00.0				1010	0220	0100	5100
В	23.8	7	0 5	2.8	0.5	0.5	0.5	0 5	05
l Ba	106	105	55 8	96.9	40.6	79.5	63.8	77 4	81.1
l Be	0.5	0.5	0 5	0.5	0.5	0.5	0.5	0 5	0.5
	84.4	91.2	2 5	82.7	2.5	2.5	2.5	2 5	2.5
Ca Cd ! Ce	0.5	0.5	0.5	0.5	Q.5	0.5	0.5	0 5	0.5
Co Co	9.5	12.9	29	9	6	9.6	1	109	3.1
Cr	10.4	16.5	16	3.6	24.2	32.1	6.3	339	4.9
cu	20100	19000	78	15100	747	2000	60.2	1220	1300
Fe Hg K La	7220	12100	799	4660	9350	12500	2510	12900	3410
Mg M''	577	122	336	235	228	270	125	260	eo a
Mo	0.5		0 5	0.5	0.5	0.5	0.5	31	04.5
Na Nb						0.0	0.0	01	0.0
Ni	360	258	54	221	31.6	53.8	4.1	45 2	32.7
Pb	265	443	20 9	43.6	14.7	52.9	13	37.3	21.5
S	1920	2160	1530	1300	637	1820	3870	1800	2880
Se									
Si s"	2.5	2.5	2 5	2,5	2.5	2,5	2,5	2 5	2.5
Sr Te M Ti U									
V W Y Zn	12.7 127	19 131	2 7 48	5.8 58.9	18.3 41.4	28 a7.4	5.4 6.9	28 8 75 5	4 17 4
Zr									

sı	_0494S.WKQ [39.2]									
		====== 12-Apr-94	= = = = = = 12-Apr-94	= = = = = = 12-Apr-94	= = = = = = 12-Apr-94	= = = = = = 12-Apr-94	= = = = = = 12 ·Apr-94	= = = = = = 12-Apr-94	12- Apr-94	* # * = = = 12-Åpr-94
1	ASSAYERS CODE	5003	5004	5005	5006	5007	5008	50W	5010	5011
 	SAMPLING LOCATIO	Star Lake T2-Wetland T2-10 core	====== Star Lake T2-Wetland T2-11 core	Star Lake T3-Wetland T3-1 soup	= = = = = Star Lake T3-Wetland T3-2 soup	= = = = = = Star Lake T3-Wetland T3-3 core	= = = = = = Star Lake T3-Wetland T3-4 core	Star Lake 73-Wetland T3-5 core	±±≈±±≡ Star Lake T3-Wetland T3-5 soup	≡≡≡≇≋≡ Star Lake T3-Wetland T3-6 core
	Processing code ====================================	\$\$ *===##	SS =====	SS ===###=	\$\$ =====	SS ====*#	SS 	SS = = = = = = =	SS ======	SS ≭#∓=≈=
	** L A 8 ** Temp. (C) pH Cond. (umhos/cm) Eh (mV) Acidity (mg/l) Alkalinity (mg/l) Ferric (Fe3+) Ferrous (Fe2+)									
		===== 27	=======================================	======	===== 05	=======================================	32	===== 0.5	===== 05	= # = = = = 7
	A	4090	4460	3060	4950	4320	3050	1810	3370	2030
!	AS	27	53	1.6	0.5	0.5	35	6.5	1.4	6
Ì	Ba	125	116	38 7	46 6	68 8	79	44 4	47.7	56.9
I	Be	0.5	05	0 5	0 5	0 5	0 5	0 5	0.5	0.5
	Bi	2.5	2 5	2 5	2 5	2 5	97	2 5	2,5	2.5
 	С									
1	Cd	0.5	0.5	05	05	0 5	05	0.5	0.5	0.5
i	Ce									
	00	5.1	94	6	19	43	13	38	3.5	3.5
1	Ur Cu	10.7	2080	02 1 69	46 6	262	3 9 2790	∠ 1490	3.5	3.6 921
i	Fe	3220	8990	7810	4070	30\$0	3980	2340	2100	2520
 	Hg K La Ma									
i	Mn	504	565	482	90 7	321	103	119	78.6	186
1	Мо	9.2	76	78	06	8.8	05	10.2	5.5	22.4
	Na Nb									
	N	43.8	62 8	129	4 9	16	62 3	51.3	5.3	30.1
1	Pb	24.3	27 5	197	86	48 5	101	6.1	13.1	15.2
l	Sb	4550	4590	4650	2750	41440	3680	3260	3070	4760
 ;	Se Se									
8	l s"	2.5	2 5	2 5	2 5	2 5	2 5	2.5	2.5	2,5
1	Sr	,								- · -
		1								
	1 Th	I								
		٨٩	119	11	11 7	6.6	20	Л	£ 7	2 0
	I w	4.0	113	11		0.0	23	4	ə./	3.8
ļ	ļ Ÿ	,								
	l Žr	21.5	32 1	22 7	169	35 4	28 2	54.8	10.7	27.1
·	1 4 1	. <u> </u>								

SL0494S WKQ 139.21					1
			=====	*****	*****
SAMPLE VOLUM	E 12.44µ1.94	12-Apt-34	17.701.94	12-40-34	12-Apr-94
ASSAYERS CODI	E 5012	5013	5014	5015	5016

SAMPLING LOCAT	IO Star Lake	Star Lake	Star Lake	Star Lake	Star Läke
11	13.Welland	T3-Wetland	T3-Wetland	T3-Wetland	T3-Wetland
1	T3-6 soup	T3-7 core	T3-7 soup	T3-8 core	T3-8 soup
I Processing and	~ ~ ~	22	99	ee	ee
	: ##== % *	 =≠====	======	33	
** FIELD **					i
) Temp (C	3				1
l pi	4				1
Cond. (umhos/cm	i) A				l
i En (m.v. I Acidity (m.a.(1) I)				Į.
Alkalinity (mg/	0				
Ferric (Fe3+	•)				1
Ferrous (Fe2+)				Í
	<u></u>		··· ··································		
** LAB **	·,				1
1 (emp (C	년 14				3
Cond. (umbos/cm	• •				1
Eh (m)	0 0				
Acidity (mg/	, Ŋ				I
Alkalinity (mg/	D)				I
I Ferric (Fe3+	•)				ļ
ferrous (Fe2+	·) 		==>	***	
ELEMENTS A	a 05	0.5	0.5	0.5	0.5
	AI 4030	1790	3190	21 60	4360
A A	S				ł
	B 0.5	6.5	0.5	0.5	1.9
1 8	a 55.2	54.4	66 4	49	50.1
l E	ie 0.5	0.5	0.5	05	0.5
1 	51 2.5 C	2.3	2 5	2.0	2.5
i c	a				F
l c	d 0.5	0.5	0.5	0.5	0.5
! c	•				
c	o 12	4 5	0.4	1.3	3.8
	Ar 4	1.5 1510	1.4	2.5	8.9
	u ⊃3.4 ≜ 2710	2220	190 2070	256 7300	3/5
· 「	α <u>2</u> 110	2220	2010	7300	3010
i	ĸ				I
1 L	a				
1 M	g		98 5	206	962
I N	l" 67 2	173		~ ~	
i M N	o 0.5	36	53	26	103
N	b				1
}	Ni 2.5	54.7	9.3	14.9	14.2
ļ	Р				İ
i F	² b 1	13.5	1	26	20
 c	S 2700	5590	2450	2330	4110
	ie				
-	Si				
9	in 2.5	2.5	2.5	2 5	2 5
:	Sr				
1	` e -⊾				
۲ ۲	n Ti				
	 U				
	V 6.5	2.9	3.3	4.6	8,4
-	W				
1	Y				
1 2	In 25.9	40.1	18,9	u.7	28.5
I .	<u>د r</u>				

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Client: Boojum Research Ltd Date Submitted: July 20/94 468 Queen St. E. Suite 400 Date Reported: August L94 Box 19 EPL Ref#: 942490 Toronto, ONT, CANADA M5A 1T7 **BROO691** Client PO#: Attn: Andrew Fyson

Certificate of Analysis

Analysis Performed:	Copper by ICP
Methodology:	1) Analysis of trace copper in water by Inductively Coupled Plasma Spectrophotometry. U.S. EPA Method No. 200.7 (Ministry of Environment ELSCAN) RECEIVEN AUG 15 1994
instrumentation:	1) Thermo Jarrell Ash ICAP 61E Plasma Spectrophotometer
Sample Description:	Water
QA/QC:	Refer to CERTIFICATE OF QUALITY CONTROL report
Results:	Refer to REPORT of ANALYSIS attached

a Cottenden

Certified By Eva Cottenden Service Manager

T. Munshaw, M.Sc., C.Chem Vice President, Laboratory Services

Environment Protec Laboratories Inc.

Certificate of Quality Control

Client : Boojum Research Ltd Contact: Andrew Fyson

Date Reported: EPL Ref # :

August 2/94 942490

Client PO#:

BROO691

Analysis of Water

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				P	ess Bl	r	Prc	ss % i	iover)			M	atrix Spik			Overall
	SAMPLE ID			********	Jpper		*****	DWO	Uppe			ľ	Lower	Upper		QC
Parameter	(spike)	LOQ	Units	Result	Limit	4ccept	Result	Limit	Limit	iccept	Result	Target	Limit	Limit	Accept	Acceptable
Jopper	5108	0.002	mg/L	nd(b)	0.004	yes	101	80	120	yes	0.925	1.0	0.6	1,4	yes	yes
Jopper	5123	0.002	mg/L	nd(b)	0.004	yes	100	80	120	yes	1.16	1.0	0.6	1.4	yes	yes
														γ		
				l												
													/	/		
OQ = Limit of Quantitation = low	est level of the para	ameter ma	at can be o	juantified	with coni	lidence				_			IK.	, 		
	equired for analysis	5						4	Authoriza	ation L.D.			/let	m		

na = Not Applicable nd = parameter not detected

= Insufficient Sample Submitted na

TR = trace level less than LOQ (b) = Analyte results on REPORT of ANALYSIS have been background corrected for the process blank

Environment Protec 1 Laboratories Inc.

Report of Analysis

Client : Boojum Research Ltd Contact: Andrew Fyson

Report Date: August 2/94 EPL Ref # : 942490

BR00691

Client PO#:

Analysis of Water

-	•	-		9	-	2			****	
Parameter	8	Units	72-7 0-5	-	72-2 5-10	12-6 S. 17	T2-116	73-4	J- E.L.	T2-2. 9-3
			BST-CATI	Reolicate	/st cxt	lstext	lsr ext	15+ 6×1	ایت حدا	2nd ext
Copper	0.002	ma/L	0.501	0.501	0.449	1.74	0.160	0.227	0.116	474
									lane	

Page 1 of 3

ğ

I

with confidence.

Client : Boojum Research Ltd Contact: Andrew Fyson

Analysis of Water

 Report Date:
 August 2/94

 EPL Ref # :
 942490

Client PO#: BROO691

arametar	100	Units	5115 "2-2 5 16 2 nd eat	5118 72-2 5-15 2nd ent	5117 r2-11b 2nd + st	5118 73-4 2009 ext	5119 736 2nd c.xt	5120 TZ-1 0-5 Meltionics FA	5121 TZH JHJ Laruhate	5122 -72-1-13-1 Mel+Wr.K+ WA
kopper	0.002	mg/L	379	346	7.37	9.75	1.16	0.221	0.654	0.463
									1/	
							Authorizatio	n I.D.	Mare	

LOO = Limit of Quantitation = lowest level of the parameter that can be quantified with confidence

Environment Protec 1 Laboratories Inc.

Report of Analysis

August 2/94 942490 BROO691 Report Date: EPL Ref # : Client PO#: Client : Boojum Research Ltd Contact: Andrew Fyson

Analysis of Water

LOQ Units 72-1-2-5	0.002 mg/L 0.849 0.857			
arameter	opper			

LOQ = Limit of Quantitation = lowest level of the parameter that can be

October 24, 1994

Environmental Protection Laboratories 6850 Goreway Drive Toronto, Ontario L4V 1P1

Attn.:Jim Bishop Re: Purchase order BRO0704

The enclosed 12samples come from a sequential extraction. 5301-5306 were extracted in 1M NH4-acetate and 5307-5312 in concentrated HNO3. All samples are filtered and 5301-5306 acidified (not necessary for 5307-5312). These samples are for Cu determination by ICP to a detection level of 0.01 mg/L. I appreciate that one or two of the samples are rather small. IF NECESSARY, these may be diluted.

5301-5306, extracted in 1M NH4-acetate 5301 **T2-2** 0-5 cm 5302 T2-2 5-10 cm 5303 T2-6 5-10 cm 5304 T2-11b 5305 T3-4 5306 T3-6 5307-5312, extracted in conc. HN03 5307 T2-2 0-5 cm 5308 T2-2 5-10 cm 5309 T2-6 5-10 cm 5310 T2-11b 5311 T3-4 5312 T3-6

Please let me know if there are any problems.

Yours sincerely,

Andrew Fyson



Client:	ient: Boojum Research Ltd 468 Queen St. E. Suite 400 Box 19 Toronto, ONT, CANADA M5A 1T7				October 25/94 November 3/94 943992 Verbal BR00704
rax:	410-801-0034				
Attn:	Andrew Fyson				
			RE(57
		Cer	tificate of Analysis		
Analysis Perfe	ormed	Copper by ICP			
Methodology:	:	1) Analysis of trace Plasma Spectrop U.S. EPA Metho (Ministry of Envi	e copper in water by Inductively Co photometry. od No. 200.7 ironment ELSCAN)	oupled	
sfrumentatio	on:	1)Thermo Jarrell	Ash ICAP 61E Plasma Spectrophot	tometer	
Sample Descr	iption:	Water			
QA/QC:		Refer to CERTIFI	CATE OF QUALITY CONTROL	report.	
Results:		Refer to REPORT	of ANALYSIS attached		

the it Σ () Certified By J.N. Bishop Vice President, Sales and Service m Certified By

for T. Munshaw, M.Sc., C.Chem Vice President, Laboratory Services
pn Laboratories Inc.	
Environment Prote	

Report of Analysis

Client: Boojum Research Ltd

Report Date:November 3/94EPL Ref # :943992EPL Quote #:VerbalClient PO#:BR00704

Water
of
Analysis

		,
J0000	47.7	
922		/ lime-
52	1.47	
MARC .	0.962	
38		
24		
	50 . 20	
	50.5	
Units	лб/L Шд/L	
ğ	ZmO.u	
Parameter	Apper	

= lowest level of the parameter

= 3 Page 1 of 2

Client : Boojum Research Ltd Contact: Andrew Fyson Analysis of Water Parameter Copper	0.002 0.002	Units mg/L	Repo	rt of An ² ^{29.9}	saio 13.4	83 96.09	3.85	Report Date: EPL Ref # : EPL Quote #: Client PO#:	November 3/94 943992 Verbal BR00704
							Authorization	C	
− Inditational of Contraction - Inditation	Lof the cor	, todt rotomo	and and and and	hiteroo - Heline be				A	

Iowest level of the parameter that can be quantified with confidence. Limit of Quantitation 3



Environment Protection Laboratories Inc.

Certificate of Quality Control

Client Boojum Research Ltd Contact: Andrew Fyson

Date Reported	November 3/94
EPL Ref # :	943992
EPL Quote#:	Verbal
Client PO#:	BR00704

Analysis of Water

				Р	ess B	(Prc	35 % F	overy			Ma	au ix Spik	e		Överali
	SAMPLE ID			· · · · · ·	Upper			ower	Jpper				Lower	Upper		QC
Parameter	(spike)	LOQ	Units	Result	Limit	\ccep	Result	Limit	Limit	Accept	Result	Target	lLimit	Limit	Accept	Acceptable
opper	5301	0.002	mg/L	nd(b)	0.004	yes	97	80	120	yes	*	*	*	*	*	yes
											-					
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					· ·	· /							\Box	•		
100 = 1 imit of Quantitation = lowest	level of the nar	ameter th	at can be d	nuantified	with con	fidence							N.			
* = Unavailable due to dilution requ	uired for analysi	S		1 uanuneu									18/10	nue		

- na = Not Applicable
- = parameter not detected nd
- = Insufficient Sample Submitted ΠS
- TR = trace level less than LOQ

(b) = Analyte results on REPORT of ANALYSIS have been background corrected for the process blank.

APPENDIX 3 ASSAYER'S QA/QC



February 2, 1993

Mr. Paul Douris Boojum Research Ltd. 468 Queen Street East Suite **400**, Toronto, Ontario M5A 1**T7**

Dear Paul:

Further to your request for information on EPL's round robin participation and QA/QC documentation, I am enclosing three separate packages.

1.0 QUALITY ASSURANCE, QUALITY CONTROL, DETECTION LIMITS

This is a precis of our QA Manual, which is at least three inches thick. This precis describes EPL's QA/QC goals and objectives as well as the laboratory applications and (p. 5) a listing of specific, routine QA/QC steps that we perform on every project. EPL is probably unique among labs in going to these lengths, and in actually providing the customer with a full QA/QC report on each project.

2.0 EPL LABORATORY CERTIFICATION

This describes our certification in Canada and the **U.S.**, as well as a listing of the round robins we've participated in. Our performance in **these** round robins is always among the top 1-5 participants.

3.0 MISA ATG # CHART

A listing of EPL's methods, keyed to MISA test group, U.S. EPA method code, and EPL's method detection limits - all of which meet the MISA requirements, as well **as** the other existing regulatory requirements.

If you require further information please give me a call.

Yours very truly, J.N. Bishop Vice President

JNB/no Enclosures

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QUALITY ASSURANCE, QUALITY CONTROL, DETECTION LIMITS, LIMS

QUALITY ASSURANCE PROGRAM

EPL's Quality Assurance Program (QAP) develops information which can be used to provide on indication of the **need** for corrections to the analytical system (QA). The QA Program measures whether or not the lab is in overall control. Quality Control (QC) becomes a subset of the QAP and evaluates the accuracy and precision of analytical data to establish the quality of data.

The following section provides an overview of EPL's Quality Assurance Program.

EPL's QA Manual is available for review upon request. An outline is attached (Appendix 1). The complete document which is several inches thick is available for viewing anytime at the EPL office.

OBJECTIVES AND GOALS

Quality Objectives

The Quality Assurance Program (QAP) assures the accuracy, precision, and reliability of the analytical data produced by EPL. Management, administrative, statistical, investigative, preventive, and corrective techniques are employed to achieve this objective through the following goals.

Quality Goals

- To develop and implement approved methods capable of meeting EPL client **needs** for precision, accuracy, sensitivity, and specificity.
- To ensure that **all** EPL **staff** receive training in quality technology enabling them to *carry out* their QAP responsibilities.
- To establish **and** keep under review a baseline of quality performance against which the effectiveness of quality improvement efforts are measured.
- To monitor the routine operational performance of the laboratory through participation in appropriate interlaboratory testing programs and to provide for corrective actions as necessary.
- To improve and validate laboratory methodologies by participation in method validation studies.

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Quality Tactics

This section lists the tactics EPL follows to achieve the QAP goals.

- Quality activities emphasize the prevention of quality problems rather than detection and correction of problems after they occur.
- Quality cost figures are computed quarterly and reported to the President.
- All employees undergo training programs commensurate with their positions, duties, and responsibilities.
- EPL uses only published and approved methods.
- EPL retains copies of all test and analytical reports in a manner and for a period specified by regulatory or accrediting bodies.
- EPL has a comprehensive calibration program involving all instrumentation used for making analytical determinations.
- EPL uses appropriate, reagents and chemicals, certified when necessary, and appropriate calibrated glassware, certified when necessary.
- EPL establishes and maintains a total interlaboratory quality management system to assure continued precision and accuracy of laboratory results.
- EPL participates in interlaboratory testing programs on its own initiative and as prescribed by accrediting organizations.

Laboratory Facilities

EPL's state-of-the-art laboratory is located at 6850 Goreway Drive, Mississauga, Ontario L4V 1P1. Specific features include:

- A high security building with restricted access to laboratory area.
- Emergency electrical back up to essential services including, fumehoods, storage refrigerators, lab lighting etc.
- Controlled laboratory suitable for trace analysis.
- Centralized services, library with on-line data searching, centralized glassware washing, maintenance, chemical and labware stores.

Sample Management

EPL's operating policies regarding sample management are designed to ensure proper identification and storage, efficient handling and full documentation of Chain of Custody. All data are recorded in EPL's proprietary Laboratory Information System (LIMS).

- Where applicable, EPL provides precleaned containers of the type and with the preservatives specified by MOE 695/88, with full Chain of Custody documentation.
- **Upon** receipt of samples, EPL's Sample Receptionist documents the following information under a unique project number.

Client information:

- client name and contact
- client reference number
- date of submission, chain of custody

Sample information:

- type, amount, # of containers
- preservation type
- condition (warm, chilled, broken, ID uncertain, etc.)
- Unique lab numbers are generated for each sample.
- All of the information is documented **on** a Sample Receipt Record.
- Labels containing the pertinent information are generated for each container received and applied to the samples prior to storage.
- Samples are stored in a locked, segregated, walk-in refrigerator (4 oC)/freezer with emergency power back up and hard copy recording temperature charts.

Workload Management

EPL defines its analytical services from **LIMS** specified Analytical Test Codes. The test codes form the basis of the SOP's which outline the analytes, the sample type, detection limits the reference method and instrument operating methods, calibration standards and QC records. The use of these codes ensures that the requirements of the requested testing is clearly defined and formally documented.

- Projects are defined by assigning the specified test codes to the lab sample within a given project.
- On-line LIMS reports display the real time status of lab workstations.
- EPL follows the **U.S.** EPA's recommended frequency for processing QC samples. These requirements are predefined and enforced by the LIMS system.
- Each analytical run contains as a minimum 1 process **blank** and 1 process recovery spike per **15** samples, as well as 1 replicate **and** 1 matrix spike per client within the **run**.
- Hardcopy worksheets containing **all** of the pertinent information are generated for each run.
- Signed and dated records of each laboratory activity are maintained.
- Lab staff have ready access to LIMS status reports including:
 - work in progress
 - work due dates
 - overdue **work**
 - _ project **status** etc.
- The **LIMS** audit trail documents **all** key events:
 - sampling date
 - date **received**
 - process data
 - _ analysis data
 - report data



QUALITY ASSURANCE/QUALITY CONTROL (QA/QC)

- All analytical services are based upon accepted (MOE, **U.S.**EPA) procedures and are fully validated prior to use.
- Analytical standards are prepared from neat solids or from certified solutions.
- Calibration standards are validated against external reference standards wherever possible.
- Extensive use is made of Standard Reference Material (SRM) for routine procedure evaluation.
- Surrogate standards are used.
- Routine submission of blind samples is standard practice.
- Analytical sequences are predefined and ensure all results are traceable to calibration and QC data.
- Hardcopy reports displaying **all** of the required data are generated for each instrument analysis.
- Analytical results are determined only from instrument responses that fall within the demonstrated calibration range.
- Acceptable QC sample performance must be demonstrated prior to the authorization of data, (data are subjected to **3** levels of QC review: technician, supervisor, and manager).
- On-going method and instrument performance records are maintained for all analysis.
- A QC certificate **is issued** with each project. The QA/QC data reported **is** specific to your project, and it consists of:
 - full spike/recovery determination blanks
 - standard reference material
 - replicate analysis
- Records containing **all** pertinent data are securely archived for seven years.
- The LIMS database is backed up daily.



Interlaboratory Comparison

- EPL is accredited by CAEAL as of June 1991.
- EPL is accredited by New York State, as of January 1992
- EPL welcomes audits and inspections by current and potential clients
- Whenever possible EPL participates in interlaboratory round robin studies. A list of round robins EPL has participated in is attached (Appendix 2).

ANALYTICAL METHODS & INSTRUMENTATION

EPL analytical methods are listed in Appendix 3. We have also included information on standard holding times and preservation methods. Methodologies specific to this contract are referenced on "Attachment A".

EPL METHOD DETECTION LIMITS

EPL follows EPA and Ontario Ministry of the Environment (MOE) analytical methods. Method Detection Limits (MDL's) are established following MOE Analytical Protocols.

Method Detection Limit (MDL) - in a given matrix and with a specific method is defined as the minimum concentration f and analyte that can be identified, qualitatively or quantitatively measured, and reported to be greater than zero at the 99% confidence level.

An MDL is a statistically defined decision point. Measured results falling at or above this point are interpreted to indicate the presence of an analyte in the sample with a specified probability, and assumes that there are no known sources of error in identification or biases in measurement.

It should be noted that when MDL estimates are developed using clean samples (i.e. reagent blanks) they represent an optimum achievable value. MDL's obtained in this fashion are useful for establishing performance criteria and allowing comparison of interlaboratory method capabilities, but are not applicable in defining the quantitation capability for other samples which introduce matrix effects. EPL MDL's have been established for the matrix being analyzed. As such, real sample MDL's *can* be higher than instrument detection limits. MDL's **specific** to this proposal are available upon request.

LIMS

Environment Protection Laboratories have a proprietary PC based LIMS system. MOE and **OGS** systems supplied by BMB Compuscience served as the genesis LIMS. The software

has been customized for EPL's specific needs. The system is capable of both sample/data handling and reporting as well as a data management to log workload, throughput and costing.

All major components of the LIMS have a backup which can be easily installed should the original fail. Data are stored on the host/server PC (33 Compaq 386) hard drives, and it is backed up to tape nightly.

There **are** two levels **of** security on the LIMS system, one at the network operations level requiring that the user know both a user number and a password. As well, the LIMS software has security, restricting access to certain modules and various levels within those modules (i.e. browsing, updating, approvals).

The system has a sophisticated costing and invoicing module. Invoices and Certificates of Analysis **are** generated by the LIMS on completion and approval of sample analysis. Reports of Analysis are available electronically through EPL's bulletin board, in ASCII delimited files if requested.

EPL's instrument data capture module has a QC checking routine which flags QC data that fail out of tolerance. The routine also compares the present QC data to long term trends. The analyst receives **a** QC error report with each **run** of samples which lists all QC exceptions. In addition, the percent spike recovery is calculated and listed. All control charts **are** updated and generated each night by the LIMS. All QC data are stored in databases for long term precision and accuracy tracking.

The data undergoes **three** levels of approval before release to the client. Each approval is time stamped **along** with the LIMS usercode of the individual who approved it.

All written methods are available on the LIMS for referral by the analyst.

INTERLABORATORY COMPARISON STUDIES

EPL routinely participates in government and industry sponsored interlaboratory comparative studies. Appendix 2 lists all the studies EPL has participated in since start up. Two round robins are of special interest - the Canadian Association of Environmental Analytical Laboratories accreditation for metals and anions and the O'Connor Associates **BTEX** evaluation; both demonstrate that **EPL's** data falls in the upper decile. More recent robins such as the **air** filter study from the Association of the Chemical Profession of **Ottario** (ACPO), January 1992, and the Atmospheric Environment Studies 1991 **air** filter study have established EPL as **a** leading air analysis laboratory. EPL has also recently been certified by New **York** State, and their certification involved analyses of interlaboratory check **samples**.



EPL LABORATORY CERTIFICATION

Neither the Government of Canada nor the Provincial Government of Ontario has a formal approval process for laboratories. However, EPL is recognized as a top quality laboratory by senior officials in the Canadian Federal Government and the Ontano Ministry of the *Environment*, and we have been approved by these agencies to perform analytical work for them.

EPL has performed extensive testing for the Province of Ontario, and has taken part in numerous interlaboratory studies for water and other materials. Our laboratory has always performed very well, and on the basis of our data quality we have been contacted by the Ontario Ministry of the Environment to perform environmental analyses.

EPL has also performed testing for several departments of the Federal Government of Canada, including Agriculture Canada and Environment Canada. Before selecting EPL, the Canadian Government examined EPL's data quality and put the lab through an extensive cross-comparison with several U.S. laboratories. The fact that both the Provincial Government of Ontario and !he Canadian Federal Government have approved EPL for their work constitutes de facto acceptance of EPL's capabilities. The Ontario Ministry of !he Environment has even used EPL to act as a referee laboratory to settle questions about data from different provincial government laboratories.

EPL is a member of the International Association of Environmental Testing Laboratories (IAETL), an organization made up of laboratories working on issues such as accreditation.

We are **also** members of the Ontario Bottled Water Association (**OBWA**) and the Canadian Bottled Water Association. EPL was selected by the OBWA as their laboratory of choice for 1991 and 1992.

EPL has been certified by the Canadian Association *of* Environmental Analytical Laboratories (CAEAL). CAEAL is the only organization in Canada that formally certifies analytical laboratories.

In the U.S., EPL has been granted Certification by New York State, through the Department of Health. EPL's certification covers bottled water, effluent, air samples and the range of other environmental analyses. Certification by New York State is regarded as primary certification by a large number of other states.

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Environment Protection Laboratories nc. Round Robin Studies

DATE	SPONSOR	TYPE OF ANALYSIS
Jan 1990	MOE # 90-1	Cyanide / Water
Jan 1990	MOE ENV890543	Metals / Sediment
Aug 1990	MOE # 90-5	BTX and Acrylonitrile
Aug 1990	MOE St. Bruno	Metals / Sediment
Sept 1990	MOE	PAH / Sediment
Oct 1990	MOE # 90-6	Mercury
Nov 1990	MOE # 90040	Lithium / Water
Nov 1990	MOE Sludge	Metals / Sludge
Jan 1991	CCIW # G-1	Chlorinated Hydrocarbons / Sediment
Feb 1991	O'Connor Associates	BTX / Water
Mar 1991	AES	Metals / High Vol Filters
April 1991	CAEAL	Metals / Anions
May 1991	MOE	PCDD/DF / PUFs
May 1991	Proctor & Redfern	PAH / PCDD/DF / Metals
Oct 1991	CAEAL	Metals / Anions
Oct 1991	MOE # 91-3	Phenols by 4AAP
Nov 1991	MOE # 91-4	Oil & Grease
Nov 1991	MOE # 91-5	TOC / DOC in Water
Nov 1991	ACPO	Metals / Anions on Filters
Dec 1991	WTC	Cyanide / Water
Dec 1991	ESSO	BTX / VCM
Dec 1991	CCIW CEPA CP-3	PCDD/DF / Ampules
Jan 1992	ACPO	Metals / Anions on Filters
Feb 1992	WTC	Cyanide / Effluents
Feb 1992	State of New York	Metals / Anions / Pesticides / Volatiles
Feb 1992	Labatts	Metals / BTX
March 1992	CAEAL	Metals
April 1992	ACPO	Metals / Anions
April 1992	State of New York	Metals / Anions / Pesticides / Volatiles
April 1992	O'Connor Associates	BTEX
April 1992	Golder/Shell	BTX / EHC / Lead
July 1992	State of New York	Metals / Anions / Pesticides / Volatiles
Oct 1992	CCIW CEPA CP-4	PCDD/DF / Ampules
Nov 1992	State of New York	Metals / Anions / Pesticides / Volatiles
Nov 1992	CAEAL	Metals

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DAW AXELROD, M. D. COMMISSIONER



Expires 12:01 AH April 1, 1993 ISSUED April 1, 1992 REVISED June 30, 1992

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INTERIM CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

Lab ID No.: 11284

Director: HR. TIM MUNSHAW Lab Name: ENVIRONMENT PROTECTION LABORATORIES INC Address : 6850 GOREWAY DRIVE MISSISSAUGA ONTARIO CAN

is hereby APPROVED as an Environmental Laboratory for the category

ENVIRONMENTAL ANALYSES NON POTABLE WATER

All approved subcategories and/or analytes are listed below:

Brdrocarbon Pestic 4 -000 4 4 -008 4 4 -007 1 pba-88C 1 drim 1 eta-88C 2 atan 2 blordane fotal 1 elta-88C 2 blordane fotal 1 elta-88C 2 blordane fotal 1 elta-88C 2 blordane fotal 1 elta-88C 2 blordane fotal 1 elta-88C 3 blordane fotal 1 elta-88C 3 blordane fotal 1 elta-88C 3 blordane fotal 1 elta-88C 3 blordane fotal 1 eltas 1 sodrim 1 s	ildes : Faste B C C C C C C C C C C C C C C C C C C	vater Kiscellaneous : romide orom, Total yanide, Total Color bemois 11 & Grease Total Recoverable ydrogen Ion (DD) pecific Conductance illica, Dissolved ulfide (as S) unfactant (NDAS) resperature Pryanic Carbon, Total rable Aromatics (ALL)	Mineral : Acidity Alkalinity Chloride Sulfate (as SO() Hardness, fotal Acrolein and Acrylonitrile (ALL) Tastewater Bacteriology (ALL) Chlorinated Hydrocarbons (ALL) Demand (ALL) Tastewater Ketals I (ALL) Mitroaromatics and Isophorone (ALL) Polynoclear Aromatics (ALL) Phthalate Esters (ALL) Purgeable Halocarbons (ALL)	Wastewater Ketals III : Cobait, Total Kolybdenna, Total Tin, Total Tballium, Total Witrosoamines : W-Hitrosodi-m-propylamine Diorins (ALL) Haloetherm (ALL) Baloetherm (ALL) Wastewater Ketals II (ALL) Nutrient (ALL) Polychlorinated Biphenyls (ALL) Priority Pollutant Phenols (ALL) Residue (ALL)
			Lawrence .	& Sturmen
Serial No.:	12864		Larrence S. Starman H.D., Ph HIGT PRIMITY (MILL)	.D., Acting Director, MARY MISPHITHIRIPRICALING

Wadswarth Center for Laboratories and Research

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Expires 12:01 AH April 1, ISSUED April 1, 1992 REVISED June 30, 1992

INTERIM CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Low of New York State

Lab ID No.: 11284

Director: MR. TIM MUNSHAW Lab Name: ENVIRONMENT PROTECTION LABORATORIES INC Address = 6850 GOREWAY DRIVE MISSISSAUGA ONTARIO CAN

is hereby APPROVED as an Environmental Laboratory for the category

ENVIRONMENTAL ANALYSES / POTABLE WATER

All approved subcategories and/or analytes are listed below:

Driaking Vater Ion-Metals : Altalinity Chloride Color Iluoride, fotal Mitrate (as N) Hydrogen Ion (pH) Solids, fotal Dissolved Sulfate (as SO4) D.V. Organobalide Pesticides : Endrin Lindane Methorychlor foraphene D.V. Chlorinated Acids : 2,4-D 2,4,5-TP (Silver) Drinking Yater Metals I (ALL) Volatile Balocarbons (ALL) Drinking rater Bacteriology : Standard Plate Count Drinking Water Tribalomethane (ALL) Volatile Aromatics (ALL)

Larrene & Aturn

nı hıkımalanı Wadsworth Center for Laboratories and Research

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Serial No.: 12865

DAVID AXELROD, M. D. COMMISSIONER



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INTERIM CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

fasued in accordance with and pursuant to section 502 Public Health Law of New York State

Lab ID No.: 11284

Director: HR, TIM MUNSHAW Lab Name: ENVIRONMENT PROTECTION LABORATORIES INC Address : 6850 WREWAY DRIVE MISSISSAUGA ONTARIO CAN

is hereby APPROVED as an Environmental Laboratory for the category

ENVIRONMENTAL ANALYSES/AIR AND EMISSIONS

All approved subcategories and/or analytes are listed below:

I (ALL)

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DOH-3317 (11190)

Serial No.: 12866

NEW YORK STATE DEPARTMENT OF HEALTH

DAVID AXIILROD, M. D. COMMISSIONER



Expires 12:01 AH ADFil 1. ISSUED April 1, 1992 REVISED June 30, 1992

INTERIM CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

Lab ID NO.: 11284

Director: MR. TIM MUNSHAW Lab Name: ENVIRONMENT PROTECTION LABORATORIES INC Address : 6850 GOREWAY DRIVE MISSISSAUGA ONTARIO CAN

is hereby APPROVED as an Environmental Laboratory for the category

ENVIRONMENTAL ANALYSES/SOLID AND HAZARDOUS WASTE

All approved subcategories and/or analytes are listed below:

Miscellaneous : Cyanide, Total Hydrogen Ion (pH) Sulfide (as S) Priority Pollutant Phenols (ALL)

Characteristic festing : foricity - Ketals Only Ketals I (ALL) Polynuclear Aron. Hydrocarbon (ALL) Polychlorinated Bipbenyls (ALL) Purgeable Aronatics (ALL)

Acroleia and Acrylogitrile (ALL) Chlorinated Hydrocarbons (ALL) Metals II (ALL) Purgeable Halocarbons (ALL)

Chlor. Erdrocarbon Pesticides (ALL) Haloethers (ALL) Mitroaromatics Isophorone (ALL) Phthalate Esters (ALL)

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Larrence S. Sturian, H.D., Ph.D., Acting Director mershapping minimumentur Munimum Proventional Wadsworth Center for Laboratories and Research xvii

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DOH-3317 (11190)

Serial No.: 12867



CAEA LABORATORY CERTIFICATIO & PROGRA REGISTRATION STATUS

LABORATORY:	ENVIRONMEN PROTECTION LABS
REGISTRATION NO:	1380
GISTRATION DATE:	JUNE 7.1991

	PARAMETER	METHOD	AUDIT DATE	PE SCORE	STATUS
ATRIX: E	RESH WATER				
1	CHLORIDE	ION CHROMATOGRAPHY	9 I.03.01	100	Y
د . د	CALCIUM. DISSOLVED	ICP	91.03.01	100	Y
	CADMIUM. DISSOLVED	ICP	91.03.01	100	Y
	COBALT. DISSOLVED	ICP	91.03.0I	100	Y.
	CHROMIUM - DISSOLVED	1024	91.03.01	100	Y
	COPPER · DISSOLVED	ICP	91.03.01	100	Y
	TRON - DISSOLVED	ICP	91.03.01	100	Y
	MAGNESIUM. DISSOLVED	icp	91.03.01	95	Y
	MANGANESE - DISSOLVED	ICP	91.03.01	la,	Y
	NICKEL - DISSOLVED	ICP	91.03.01	100	Y
	LEAD - DISSOLMD	ICP	91.03.01	100	Y
	VANADIUM - DISSOLVED	icp .	91.03.01	100	Y
	- ZINC-DISSOLVED	ICP	91.03.01	100	Y
	POTASSIUM	FLAME PHOTOMETRIC	91.03.01	80	Y
	SODIUM	FLAME PHOTOMETRIC	91.03.01	100	Y
	NITRATE	ION CHROMATOGRAPHY	91.03.01	90	Y
	NITRATE + NITRITE	ION CHROMATOGRAPHY	91.03.01	100	Y
	SULPHATE	ION CHROMATOGRAPHY	91.03.01	100	Y

PROVISIONAL REPORT

APPENDIX 4 ELECTRON MICROSCOPY APPENDIX 4 ELECTRON MICROSCOPY



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