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Guidance Document for Conducting Toxicity Reduction Evaluation (TRE) Investigations for Canadian Metal Mining Effluents

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The draft Metal Mining Effluent Regulation (MMER) requires that all Canadian metal mines produce effluent that is non-acutely lethal (#50% mortality in 100% effluent) to rainbow trout when tested in accordance with Environment Canada's Reference Method EPS 1/RM/13. Mine operations will also be required to monitor the acute lethality of effluent to *Daphnia magna* in accordance with Environment Canada's Reference Method EPS 1/RM/14. In the event of an effluent toxicity failure, the Toxicity Reduction Evaluation (TRE) developed by the U.S. EPA is a commonly used step-wise approach designed to assist industrial dischargers in eliminating final effluent acute lethality. A TRE is a site-specific study designed to identify the substances responsible for acute lethality, isolate the source, evaluate the effectiveness of control options, and confirm the reduction in acute lethality of the final effluent. Toxicity Identification Evaluations (TIEs) are a set of procedures that identify the specific substance responsible for acute lethality, and can form a subset of tools used in a TRE. The purpose of this Guidance Document is to provide TRE guidance specifically focused on challenges faced by the Canadian metal mining sector in order to assist mining facilities in meeting the acute lethality requirements for both rainbow trout and *Daphnia magna*. This Guidance Document is intended to provide mine managers with an effective tool for implementing an appropriate strategy for resolving acute lethality issues, provide laboratories with a useful guide for conducting TRE studies with metal mining effluents, and ultimately, increase the likelihood of achieving and maintaining a consistently non-acutely lethal metal-mining effluent. It is not intended to replace the existing U.S. EPA documents, but rather to provide supplementary guidance specific for application with Canadian regulatory species and metal-mining effluents. Key components of the metal-mining TRE document will be presented.

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A Combined TIE/TTE Approach to Identification of Effective Treatments for Reduction or Elimination of Mine Effluent Lethality

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Environment Canada funded a study to demonstrate the use of toxicity identification evaluation (TIE) and toxicity treatment evaluation (TTE) on representative "challenged" mine effluents, as tools that may assist such mines to achieve compliance with Metal Mining Effluent Regulations (MMER). Two "challenged" mines (A and B) were identified which agreed to provide effluent for the project to evaluate and develop a combined TIE/TTE approach. The TIE is taken to the point of suggesting the nature of the main toxicant(s). This serves to focus the TTE investigations on treatments that are most likely to be effective. Thus, treatments can be investigated, and an effective treatment can be found, without definitive toxicant identification and confirmation. Treatment selection based on TIE increases the likelihood of success.

For Mine A, a gold mine, the primary toxicants identified by TIE were ammonia and metals (likely copper and zinc), with cyanate and/or nitrite also possibly contributing. The TTE showed that filtration through granulated activated carbon (GAC) reduced effluent acute lethality to *D. magna* but not to fathead minnow (used as a surrogate for rainbow trout), while filtration through GAC and zeolite eliminated toxicity to both species. GAC alone removed most candidate toxicants but only half the ammonia and nitrite, while GAC plus zeolite removed all the suspected toxicants. The various treatments were repeated using regulatory rainbow trout testing which confirmed the elimination of toxicant by activated carbon and zeolite. Activated carbon plus air stripping and activated carbon plus hydrogen peroxide addition reduced toxicity but did not eliminate it.

For Mine B, a nickel-cobalt mine, the toxicants suggested by TIE included total dissolved solids (TDS) (major ions such as potassium and bicarbonate), a metal toxic to *D. magna* at low pH (possibly copper), and some additional toxic agent affecting rainbow trout (possibly thiosulphate). A TTE study was not completed as mine staff reported that they were undertaking changes to the treatment process, designed to achieve zero mill discharge. Initial TIE results indicated that filtration through zeolite reduced the effluent acute toxicity, and also reduced TDS and copper concentrations.

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The Use of 100% Process Water Recycle to Achieve a Non Toxic Effluent at the Raglan Div. of Falconbridge Limited

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The Raglan Mill had been in production for less than a year when the initial testwork on the effluent revealed that the effluent was defined as 'toxic' under regulations. Testwork was begun to address the fundamental reasons for the toxicity and a systematic approach was initiated to identify causes.

Elimination of the problem was not going to be easy once the main conclusion identified high dissolved solids as the main culprit. In particular normally benign soda ash reagent additions used in milling was identified as the main contributor to the problem. Significantly the effluent respected all other requirements under the regulations.

Throughout the process, Raglan acted in conjunction with the regulators and others to address the problem during the course of a year of testwork. After numerous reviews, risk assessment, the future of the regulatory framework, and considering our commitment to Sustainable Development, it was decided to push for a less conventional approach. Following analysis of all requirements and safety considerations, it was decided to pursue a concept to recycle 100% of the mill process water. This would have numerous positive benefits to the operation, including reducing fresh water consumption, lowering costs and meeting regulatory requirements.

The system is currently scheduled to be commissioned in mid October of 2001, with a one year start up phase envisioned. The cost of the project is estimated at over $3 million dollars (CAN).

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Toxicity Identification Evaluation of Effluent from a Mine

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The toxicity of discharges from mining operations continue to be of concern to the regulatory community and mine operators. Toxicity in discharges may be caused by a variety of factors, including metals, ammonia, pH, process chemicals and total dissolved solids. In this study, a toxicity identification evaluation (TIE) was performed on samples of a discharge from a gold and silver mine which exhibited toxicity in 7-day partial lifecycle tests using the freshwater cladoceran *Ceriodaphnia dubia*. The results indicated that an organic anion was responsible for toxicity and that phosphorus concentrations in the treatments were correlated with toxicity. Collectively, the data suggested that a phosphine-based collector (Aerophine 3418A Promoter) used in the metals flotation process was the most likely cause of the observed toxicity. Consequently, the chemical was evaluated for toxicity and its response to the TIE procedures which were effective at reducing toxicity in the discharge sample. These results, and those of a confirmatory spiking study, consistently suggested that Aerophine was the cause toxicity. Efforts at the mine to reduce the residual concentration of this process chemical resulted in reduced toxicity of the discharge.

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Application of Source Investigations and Toxicity Treatability Evaluations for Toxicity Reduction of a Final Effluent at a Canadian Metal Refinery.

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The final treated effluent from a Canadian metal refinery has periodically been in non-compliance with the regulatory toxicity limits (#50% mortality in 100% effluent) for rainbow trout and *Daphnia magna*. Sodium, carbonate, atypical ion balance and copper were suspected as possible causes of *Daphnia magna* mortality, while copper and sodium were possible causes of trout mortality. Because the substances responsible for final effluent toxicity could not be confirmed using standard TIE methods, an alternative approach was undertaken, which included i) characterization of upstream sources of toxicity, and ii) evaluation of treatment technologies and management options for toxicant removal. A mass balance approach identified three streams (out of 22) representing the largest contributors, in terms of toxicity and chemical load, to the ETP. Removal of the most toxic stream (Stream #1) from the process resulted in a decrease in Cu and was beneficial to trout survival. For *Daphnia*, it was suspected that TDS from Streams #2 and #3 contributed to toxicity even in the absence of elevated Cu from Stream #1. Subsequent bench-scale testing focused on TDS components in Streams #2 and #3, and included: i) K and Ca Ion Exchange, ii) Zeolite, iii) Evaporation, and iv) Selective Precipitation. Only evaporation of Stream #3 eliminated toxicity to *Daphnia*. Elimination of toxicity following removal of both Streams #1 and #3, provided evidence to support the hypothesis that copper (from Stream #1) and elevated TDS (from Stream #3) were the main causes of *Daphnia* mortality. Stream #1 was successfully removed from the process with the added benefit of metals recovery back into the refining process. Further testing is underway to determine a conductivity threshold for *Daphnia* in the absence of toxicity due to copper. Following removal of Stream #1 and control of TDS, the effluent has been non lethal to trout and *Daphnia*.

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Toxicity Characterization of Water Treatment Effluent, Rouyn-Noranda Area, Quebec

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Treated water discharged from the Inmet Mining Corporation Lac Dufault Division Norbec site, 5 km north of Rouyn-Noranda, Quebec, shows sporadic acute toxicity to *Daphnia magna*. This presentation describes the results of a Toxicity Identification Evaluation (TIE) commissioned by Inmet to understand the cause(s) of the toxicity.

Acid drainage originating from two closed tailings impoundments is treated by one of two systems; a simple lime addition system and a high density sludge (HDS) lime neutralization plant commissioned in 1998. The HDS plant performance consistently meets or exceeds the performance of similar WTPs in Canada, as described by Best Available Technology Economically Achievable (BATEA). *Daphnia magna* acute toxicity tests conducted with three water samples taken in May, 2001 indicated that effluents from the simple lime system and the final effluent point were non-toxic. However acute toxicity was observed with the HDS effluent - EC50 values were 60 and 67 percent using the APHA/AWWA/WEF Standard Methods and Quebec Provincial methods, respectively. A TIE was subsequently performed on the HDS effluent.

Toxicity attributable to total dissolved solids, organic constituents, oxidizers and some metals was eliminated based on the TIE study results. Zn was implicated for the observed toxicity; however, there was no clear correlation between Zn concentrations and the transient toxicity. It was also noted that sample toxicity substantially decreased with time, as did sample pH. This observation may explain the transient toxicity and the inability to correlate effluent toxicity with Zn concentrations, since laboratory studies have demonstrated that Zn toxicity is reduced as pH decreases. The fact that the Norbec HDS WTP meets or exceeds BATEA performance but may still produce effluent with transient *Daphnia magna* toxicity has important implications for industry and regulators alike.

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Dome Mine Copper Toxicity Control with EDTA and Ammonia Toxicity Control with Post pH Adjustment

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The 91 year old Dome Mine in South Porcupine, Ontario, currently processes 5 million tons of ore and yields 320,000 ounces of gold per year. Cyanide tailings from a conventional carbon in pulp leaching process are impounded in a large (325 ha) tailings area. Effluent is discharged seasonally during the summer and early fall months to maintain a site water balance while taking advantage of natural degradation processes.

An Effluent Treatment Plant (ETP) was constructed in 1995 to treat metals and suspended solids. In 1997, an INCO SO_2/Air cyanide destruction module was added to treat cyanide. The ETP was designed to remove effluent toxicity according to pilot scale tests. In 1998, treated effluent failed *Daphnia* toxicity tests required by the Ontario MISA Regulation, despite effluent chemical parameters being well below permit limits. A Toxicity Identification and Reduction Evaluation identified ammonia as the toxicant to Rainbow Trout, and free copper as the toxicant to *Daphnia magna*.

During the 1999 ETP operating season the addition of EDTA (ethylene diamine tetra acetic acid) commenced. EDTA complexes remaining free copper as an end of pipe treatment in much the same way residual cyanides or natural organic compounds do; both are at very low levels in the Dome effluent. Effluent now passes all *Daphnia magna* toxicity tests and downstream monitoring has detected no environmental effects. EDTA is slow to biodegrade in the environment and potential alternatives Humic Acid and Sodium Thiosulphate were evaluated with limited success. Continued optimization of the ETP has resulted in a drop in copper loadings and EDTA usage. The risk for EDTA to re-mobilize metals from downstream sediments has been controlled with a reduced dosage rate.

Ammonia reductions have focussed on best management practices with the goal to reduce the risk of ammonia toxicity to Rainbow Trout and nutrient loadings to the South Porcupine River. Careful post pH adjustment of the final effluent controls un-ionized ammonia toxicity. Disposal of high ammonium nitrate wastewaters into the tailing pond by the explosive handling contractor (ORICA) was halted and is now treated by evaporators. The result has been no Rainbow Trout toxicity failures since 1998.

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Influence of Water Quality on the Acute Lethality of Ammonia

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COGEMA Resources Inc. McClean Lake Operation uranium ore milling process uses ammonia to precipitate ammonium diuranate, which is subsequently transformed to a final product known as yellowcake (U_3O_8). Excess ammonium sulphate solution is transferred to the ammonium sulphate crystallization plant where ammonium sulphate crystals are produced. This product is sold in bulk to the fertilizer blending industry. The plant is highly effective at removing ammonia, however upset conditions can result in elevated ammonia concentrations in the treated mill effluent. As part of the first year of operation, acute rainbow trout toxicity testing of effluent was undertaken on a monthly basis. Observations of mortality in several tests at less than 100% effluent appeared to be associated with elevated ammonia, however clear dose response relationships were not evident.

The influence of pH and temperature on ammonia speciation are relatively well established, and the influence of ammonia speciation on toxicity has been quantitatively defined. Less often considered is the effect of ionic strength on ammonia speciation and toxicity. Although the effects of ionic strength on ammonia speciation in freshwaters is relatively small, it can be an important consideration in effluent toxicity testing, when the ionic strength of treated effluent can be substantially elevated. Consideration of the effects of pH, temperature and ionic strength on ammonia speciation were necessary to resolve the dose-response relationships of the effluent toxicity tests.

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Development of a Guidance Document for Acute Lethality Testing of Metal Mining Effluents

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The new Metal Mining Effluent Regulations (MMERs), when promulgated, will include a provision for a non-acutely-lethal effluent. Toxicity tests will be conducted according to Environment Canada's Biological Test Methods for rainbow trout (EPS1/RM/13) and *Daphnia magna* (EPS 1/RM/14). Data reliability will be critical in maintaining confidence in the assessment of regulatory compliance. There are a number of factors that can impact upon the reliability of acute lethality toxicity test data, which are addressed to various degrees by the Environment Canada test methods and the Canadian Association of Environmental Analytical Laboratories (CAEAL)/Standards Council of Canada (SCC) laboratory accreditation program. However, additional guidance may assist both generators and end users of the data. One of the TIME network's prioritized activities is the development of a Guidance Document that addresses the key aspects of acute lethality testing and provides guidance aimed at maximizing data reliability. The Guidance Document will assist mine personnel in the collection and submission of samples, in the evaluation of toxicity test reports, and will enhance the efforts of laboratories to produce reliable data. Key components of the Acute Lethality Guidance Document will be presented, specifically, results of the literature and data review phases of the study, factors influencing data reliability with recommended guidance, the role of accreditation in laboratory QA, and guidance to industry, government and laboratory personnel.

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Rainbow Trout Liver Hepatocyte Test as a Potential Alternative to the Acute Toxicity Method For Screening Effluents

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In this test, freshly prepared hepatocytes from three juvenile rainbow trout are exposed to liquid sample for 48h at 15ºC. After the exposure period, hepatocytes are collected for cell viability evaluations. Cell viability is determined either by the propidium iodide (PI) exclusion technique or the neutral red (NR) uptake inhibition technique. The PI method can be adapted to a microplate or cuvet fluorometer and the NR uptake test can be determined either spectrophotometrically or fluorometrically. In a recent study, trout primary hepatocytes were exposed to various industrial effluents for 48 hours. Fingerling trout were also exposed to various effluents for 96 hours using Environment Canada's rainbow trout acute lethality test. The results showed that 33 out of 42 effluents were concordant in that the hepatocyte test was able to detect absence and presence of toxicity in effluents. This data suggests that there is 80% concordant between the rainbow trout acute lethality and hepatocyte tests and therefore, high potential for the hepatocyte test to be an excellent effluent screening technique. In the non-concordant effluents, most samples indicated that the hepatocyte test identified toxicity although the trout acute lethality test did not respond. Unfortunately, no effluent split-sample testing has been conducted with metal mining effluents. Although a number of procedural items will still have to be clarified and further testing of additional effluent types is required, a recent independent assessment of the research behind the development of the hepatocyte test concluded that the standardization process could be started.

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Literature Review of Chemistry and Toxicity of Mercury, Cadmium, Selenium and Antimony in Metal Mining Effluents

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CANMET, the Mining Association of Canada (MAC), and Environment Canada funded a review of minor elemental constituents that may be of environmental concern in the context of metal mining effluents. The objective of this work was to conduct a critical review of the literature for mercury, cadmium, selenium and antimony with respect to their chemical behaviour under different process and environmental conditions, their potential contribution to mine effluent toxicity, and applicable treatment technologies to reduce or eliminate toxicity due to these four metals. The mineral associations of each metal, and the mine types likely to release them, were discussed. Typical concentrations in mine effluents were compared to acutely toxic levels, with emphasis on levels toxic to *Daphnia magna* and rainbow trout. Based on this review, and considering the usual chemical characteristics of mine effluents, the likely need for treatment was discussed for each metal. Factors that may influence treatment success were discussed, and typical removal efficiencies were given, for the different treatment technologies that could be used to reduce the effluent concentrations of these metals.

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Creating a Model for Predicting Metal Toxicity to Invertebrates

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Standards for the protection of the aquatic environment are generally limited to total metal concentrations even though they do not reflect metal bioavailability. To address differences in metal bioavailability, research is underway to create computer models that can predict toxicity of metals under various physical/chemical conditions. As part of the national MITE-RN (Metals in the Environment Research Network) program, our study attempts to predict toxicity of metals to *Hyalella azteca* by taking a similar approach to that used in the development of the biotic ligand model for fish. Short-term experiments were conducted in various media to estimate binding constants for nickel and major ions to *Hyalella azteca*. These binding constants can then be added to chemical speciation models to create a bio-chemical model, capable of predicting short-term toxicity of nickel in waters of different physical/chemical characteristics. Details of the study and results will be presented.

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