LMT Technology Verification

Workplan

California Department of Toxic Substances Control
Office of Pollution Prevention and
Technology Development

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A. PROJECT ORGANIZATION

TITLE AND APPROVAL SHEET

Department of Toxic Substances Control	U.S. Environmental Protection Agency
Mr. Tony Luan Assignment Manager	Ms. Norma Lewis Technical Project Manager
_____	_____
Mr. John Wesnousky Technical Review Panel	Ms. Lauren Drees Quality Assurance Officer
_____	_____
Dr. G. Wolfgang Fuhs Technical Review Panel	
_____	
Mr. Ed Benelli Technical Project Manager	
_____	

DISTRIBUTION LIST

Department of Toxic Substances Control

Mr. Tony Luan, DTSC/OPPTD (Primary Decision Maker)
Mr. John Wesnousky, DTSC/OPPTD (Technical Review Panel)
Mr. G. Wolfgang Fuhs, Ph.D., DTSC/OPPTD (Technical Review Panel)
Ms. Ruth Chang, Ph.D., DTSC HML (QA/QC Reviewer)
Mr. Ken Beutler, DTSC/OPPTD (Health and Safety Reviewer)
Mr. Milad Iskander, DTSC HML (Lab Service Provider)
Mr. John P. Christopher, Ph.D., DTSC/HERD (Toxicology Reviewer)
Mr. Dick Jones, DTSC/OPPTD (Project Reviewer)
Mr. Pat Bennett, DTSC/OPPTD (Project Reviewer)

U.S. Environmental Protection Agency

Ms. Norma Lewis, U.S. EPA (Technical Project Manager)
Ms. Lauren Drees, U.S. EPA (QA/QC Officer)

LMT, Inc.

Mr. C. Patrick Costin, President, LMT Inc.
Mr. John Moberly, Ph.D., Consultant to LMT Inc.

PROJECT MEMBER DUTIES AND RESPONSIBILITIES

Department of Toxic Substances Control

Assignment Manager - Tony Luan has final DTSC authority and oversight of planning team activities.

Project Manager - Ed Benelli is responsible for overseeing implementation of the Technology Verification Workplan, coordinating project team meetings, ensuring that necessary resources are provided for planning team decisions, and for preparing project reports.

QA/QC, Member - Ruth Chang, Ph.D., is responsible for ensuring the data collection system meets QA/QC requirements.

Industrial Hygienist - Ken Beutler is responsible for approving field worker Hazard Appraisal Recognition Plans.

Laboratory Activities – Milad Iskander is responsible for providing Hazardous Materials Laboratory oversight of analytical data generated during the testing phases.

Toxicologist - John P. Christopher, Ph.D. is responsible for identifying toxic constituents that may pose a threat to public health and the environment.

Project Reviewers - DTSC project team members are responsible for participating in field activities, project meetings and reviewing project reports. Each member of the project team was selected based on his/her knowledge of the scientific and engineering principles involved.

U.S. Environmental Protection Agency

Project Manager - Norma Lewis is responsible for providing U.S. EPA oversight and review of the Technology Verification Workplan, workplan implementation, and data evaluation reports.

QA/QC - Ms. Lauren Drees is responsible for providing U.S. EPA QA/QC review of the data analysis.

B. PROJECT BACKGROUND

Problem Background

Heavy liquids or dense solutions are used to separate materials of lighter or heavier density based on the material's buoyancy. Materials with a density greater than the liquid will 'sink' to the bottom, while materials with densities less than the liquid will 'float' to the surface. Separation of mineral samples has historically been accomplished with halogenated organic liquids, the most common being tetrabromomethane, methylene bromide, tetrachloroethylene, tert-butyl ethylene, and methylene iodide. Other organic liquids are added to adjust the specific gravity for user needs. These organic dilutents include naptha, acetone, and ethanol. Several of these compounds are volatile organic compounds (VOCs) and are recognized to contribute to photochemical smog.

One application of separation processes utilizing heavy liquids is in the determination of coal washability. This test determines the theoretical limits for the removal of mineral impurities from coal by beneficiation processes that rely on specific gravity separations. The data from these kinds of tests are required by individuals and organizations that supply, purchase, and utilize coal. These groups conduct research in the design of coal-processing plants, the efficiency of coal-processing plants, and in the qualities of coal reserves.

Lithium Metatungstate (LMT) is a heavy liquid solution with a specific gravity of 3.0. LMT Inc. holds the patent and manufacturing rights to the compound tungstate-hexalithium, $Li_6(H_2W_{12}O_{40})$. LMT solution (tungstate-hexalithium 79.2%, and water 20.8%) is used in analytical laboratories to separate fractions of a samples such as mineral and coal. The solution can be adjusted to obtain a specific gravity as low as 1.1 by adding deionized water. Samples of mineral or coal added to dilutions of LMT will separate into fractions as a function of their specific gravity compared to that of the LMT dilution. Depending on what density is being analyzed, the LMT solution specific gravity may be re-adjusted to further separate a fraction, thereby narrowing the range to identify only particles of a particular specific density. A concentrated LMT solution can then be recovered for reuse by removing dilution water through evaporation. This regeneration is possible because LMT has a high boiling point.

The laboratory techniques for conducting sink/float analysis with halogenated organic liquids are similar to those used for LMT, but with several important distinctions. The organic liquids have high toxicity ratings, are carcinogens and/or mutagenic, are volatile and flammable, and cannot be easily regenerated. The laboratory must be well ventilated and the liquids should be used under fume hoods with scrubbers. Laboratory workers wear protective clothing and can be required to wear respirators. The volatile nature of the organic chemicals causes chemical losses to the atmosphere. Makeup liquids must be added to solutions to maintain volume and to adjust specific gravity. The rinse solution and tested samples become hazardous waste, especially with coal and other porous samples.

Problem Definition

The use of halogenated organic liquids for the separation of mineral and coal fractions impacts air quality by contributing to photochemical smog; presents a significant source of hazardous waste generation from spent solutions, rinses and discarded samples; and poses a threat to worker health and safety due to high volatility, flammability, and toxicity.

Problem Resolution

LMT Inc. developed Lithium Metatungstate to replace halogenated organic liquids used in analytical procedures for mineral and coal separations. LMT solution is claimed to: 1) provide equivalent analytical results; 2) generate tested samples which do not contain residuals of LMT solution; and 3) exhibit reduced exposure to toxic compounds for workers and the environment when compared to the halogenated liquids. Product literature from LMT Inc. regarding Lithium Metatungstate is contained in Appendix A.

LMT Inc. claims that their product can be directly substituted for the halogenated organic liquids in the industry-accepted analytical procedure, American Society for Testing and Materials (ASTM) Standard D 4371-98. Appendix B contains the ASTM test methods to be used in the LMT Verification Project. Because LMT uses water to adjust the specific gravity, the rinse solutions are not discarded as with the organic liquid rinses; instead, LMT can be regenerated by removing the water. Because the analytical procedure includes steps to wash the product from the tested samples, absorption should be minimal and the samples should retain little residual LMT product. With strict laboratory techniques, rinses can be collected and recycled; only a small amount of the product should be lost due to laboratory spills and the like.

As a function of its high boiling point and low vapor pressure, LMT should not pose an inhalation threat to worker health as the halogenated organic liquids do. LMT is non-flammable, reducing the risk to worker safety. The toxicity of halogenated liquids is well characterized, and the risk from exposure to LMT for workers and the environment is anticipated to be much lower. The Material Safety Data Sheet (MSDS) for LMT is contained in Appendix C.

C. PROJECT DESCRIPTION AND OBJECTIVES

The project objectives are to validate the performance claims proposed by LMT Inc. and conduct a limited investigation of worker and environmental exposure to toxics. These objectives require three areas of inquiry: 1) is the use of Lithium Metatungstate solution indistinguishable when compared to the previously used compounds when the ASTM test is employed in mineral and coal separations; 2) what amount, if any, of the LMT solution remains on the samples after normal testing procedures; and 3) what are the toxic effects of LMT solution?

1. To validate LMT Inc.'s performance claims, ASTM Standard D 4371-98 will be

employed using both LMT solution and halogenated organic liquids. The ASTM test will determine the analytic accuracy of LMT solution when used in specific gravity separations through a comparison of the mean weights for the separated specific gravity fractions. Separation tests will be performed on both mineral and coal samples, and by using both the LMT product and the halogenated organic liquids.

2. The amount of residual LMT solution on the tested samples will be identified by the presence of lithium and tungsten. This will be an indication of the amount of LMT solution lost during normal testing procedures. Samples of the LMT solution used for the procedure, and both untested samples and samples exposed to LMT, will be collected and analyzed for lithium and tungsten content. When compared to the untested samples and the concentrations in LMT solution, residuals of these analytes on the tested samples will yield an indication of the amount of LMT remaining after normal testing procedures.

3. A limited investigation of worker and environmental exposure to toxics will be conducted by evaluating the toxic effects of LMT solution on fresh water test organisms. Toxicity will be established by determining the LC_{50}, the concentration at which 50% of the test organisms experience mortality, and by evaluating the NOAEC, the No-Observable-Adverse Effect Concentration.

In addition to these laboratory procedures, investigations will be conducted through surveys of end-users, reviews by technical specialists, and by observations made during field testing procedures.

A survey of end-users of the LMT product will utilize a written questionnaire which solicits the reasons users switched to the new product, the type of samples and analysis they are performing, their satisfaction with LMT performance, and any additional comments and concerns they may have. An assessment of worker health and safety and toxic environmental effects resulting from exposure to LMT solution will be made by Department industrial hygienists and toxicologists. Analytical data generated from the laboratory procedures as well as from a literature search will be used in this assessment. Observations will be made by the Project Manager and Department staff during field testing on the potential impacts to protection of worker health and safety, and on the potential for losses of the product to the environment.

II. SAMPLE TESTING AND DATA ACQUISITION

Table 1. identifies the laboratory test methods to be used in analyzing LMT during the

Table 1. Summary of Sample Analysis and Test Methods for the LMT Verification Project

Testing Location	Sample	Test to be Conducted	Number of Samples	Test Method	Notes
CT&E, Denver, CO	Mineral	Sieve Analysis / Size Determination	5	ASTM D 4749	Sample size: 100 g. Use sieve size of XX.
		Sample Preparation	5	ASTM D 2013	As required, to prepare samples for weighing after gravity separation procedure.
		Gravity Separation Using LMT solution	5	ASTM D 4371-98	2 fractions collected from density 2.95. Same sieve size to be used throughout.
	Coal	Sieve Analysis / Size Determination	5	ASTM D 4749	Sample size: 2000 lbs. Coarse coal portion 2.36-9.5 mm specified for gravity separation procedure.
		Sample Preparation	5	ASTM D 2013	As required, to prepare samples for weighing after gravity separation procedure.
		Gravity Separation Using LMT solution	5	ASTM D 4371-98	3 fractions collected from coarse coal sample. Fractions separated at densities 1.4 and 1.6.
	LMT Solution	Acute Definitive Aquatic Toxicity	3	EPA/600/ 4-90/027F	Fathead minnows, *Pimephales promelas*, to be specified. Five concentrations of LMT: 0.001, 0.002, 0.005, 0.01, and 0.02 percent, plus one control. Static-renewal test.
CT&E, Henderson, KY	Mineral	Sieve Analysis / Size Determination	5	ASTM D 4749	Sample size: 100 g. Use sieve size of XX.
		Sample Preparation	5	ASTM D 2013	As required, to prepare samples for weighing after gravity separation procedure.
		Gravity Separation Using Halogenated Organic Liquids	5	ASTM D 4371-98	2 fractions collected from density 2.95. Same sieve size to be used throughout.
	Coal	Sieve Analysis / Size Determination	5	ASTM D 4749	Sample size: 2000 lbs. Coarse coal portion 2.36-9.5 mm specified for gravity separation procedure.
		Sample Preparation	5	ASTM D 2013	As required, to prepare samples for weighing after gravity separation procedure.
		Gravity Separation Using Halogenated Organic Liquids	5	ASTM D 4371-98	3 fractions collected from coarse coal sample. Fractions separated at densities 1.4 and 1.6.
DTSC HML, Berkeley, CA	Mineral	Acid Digestion	5	DTSC HML, Berkeley Methods	Sample size: 10-20 g; glass container.
		Lithium, Tungsten Concentrations	5	EPA Method 6020A	EPA Method 6020A, or equivalent, for lithium and tungsten.
	Coal	Acid Digestion of Sample	5	DTSC HML, Berkeley Methods	Sample size: 1 lb.; glass container.
		Lithium, Tungsten Concentrations	5	EPA Method 6020A	EPA Method 6020A, or equivalent, for lithium and tungsten.
	LMT Solution	Lithium, Tungsten Concentrations	3	EPA Method 6020A	EPA Method 6020A, or equivalent, for lithium and tungsten. Sample size: 500 mL; poly container.

gravity separation, residual product, and aquatic toxicity determinations. Each project objective is considered to be of equal importance in the evaluation of LMT, and there are no definitive pass/fail criteria for verifying this product.

A. GRAVITY SEPARATION COMPARISON

Mineral and coal samples will be subjected to five replicate analyses using both LMT solution and the halogenated organic liquids. Each individual mineral and coal sample will be separated into at least three fractions using LMT and the organic liquids. This will require at least one subsequent adjustment of the solution's specific gravity. Additional data will yield the total dry weight of each fraction collected, and a computation of the percent dry weight for each. An average and standard deviation will be calculated for each of the fractions separated during the five replicate tests. Using the two-sample t statistic, the mean values for each fraction will be compared to determine the level of confidence at which the methods agree (Miller, Irwin, et al, *Probability and Statistics for Engineers*, 4th ed., Prentice-Hall Inc., New Jersey, 1990, p. 245).

Preparation of the test samples is recognized as a critical step. Obtaining, or producing homogeneous, representative test samples and maintaining sample integrity during shipment must be ensured. Commercial Testing & Engineering Company (CT&E), with facilities in Denver, CO, and Henderson, KY, has used both LMT and halogenated organic liquids in the determination of coal washability, and will provide the coal test samples and heavy liquids used for the Verification Project. CT&E, Denver will conduct the gravity separation test using LMT solutions, while the Henderson facility will conduct the gravity separation test using halogenated organic liquids. While the particular halogenated organic liquids are not specified in the ASTM procedure, the Henderson facility reports that **IIItert-butyl ethyleneIII**, and methylene iodide are commonly used for these separations.

Representative coal samples will be obtained from a source near the Denver laboratory, and mineral samples will be provided by LMT, Inc. The coal sample will be collected using heavy machinery from an open, operating seam. The selection of sample location, collection procedures, handling, packaging, and transport, will ensure that the sample exhibits typical coal properties such as size distribution and moisture content. The coal sample procedures are anticipated to require that several thousand pounds of sample be collected. Several pounds of mineral sample will be an appropriate collection amount.

Sample preparation, packaging, and shipment methods shall ensure that sample integrity is maintained throughout the course of the investigation. The sample sent to CT&E, Henderson will be shipped in 55-gallon drums, which is the standard procedure. The mineral sample will be wrapped in plastic and shipped by normal methods.

The method to be used for preparing mineral and coal samples for analysis will be ASTM Standard D 2013, Method of Preparing Coal Samples for Analysis. ASTM Standard D

4749, Test Method for Performing Sieve Analysis of Coal and Determining Coal Size, will be employed to select the appropriate mineral and coal particle size fractions.

The method to be used for gravity separations of the mineral and coal samples will be ASTM Standard D 4371-98, Standard Test Method for Determining the Washability Characteristics of Coal. This test method describes procedures for determining the washability characteristics of coarse-coal and fine-coal fractions by gravity separation using heavy liquids, with the fractions being differentiated by upper and lower size limits. The size fraction to be used for mineral and coal testing purposes will be that of coarse-coal. The lower cut-off size for determining coarse-coal will be that portion with particle size diameter smaller than 2.36 mm (No. 8 USA Standard Sieve Series), and the upper limit will be 9.5 mm. In order to reduce variability, CT&E, Denver will prepare both coal samples, while the Henderson facility will prepare the mineral fractions.

The laboratory address and contact for the gravity separation process using LMT solutions is:

Commercial Testing & Engineering Co.
4665 Paris Street, Suite B-200
Denver, CO 80239
Mr. Kevin Palmer
(303) 373-4772

The laboratory address and contact for the gravity separation process using halogenated organic liquids is:

Commercial Testing & Engineering Co.
618 Bob Posey Road
Henderson, KY 42420
Mr. Gale Myers
(270) 827-1187

B. RESIDUAL PRODUCT DETERMINATION

An analysis for residual product on the tested samples will include collection of untested mineral and coal samples, collection of tested samples which have been exposed to the LMT solution, as well as an analysis of LMT solution. One sample each will be collected from the untested mineral and coal, and will be analyzed by ICP/MS for lithium and tungsten; these metals will be the essential indicators of residual product. A sample of the LMT solution used in the separation procedures will be analyzed for the concentrations of these metals, as well. Subsequently, one of each of the three sample fractions which has been separated using LMT will also be analyzed for indications of residual LMT product. These will be collected after rinsing and drying operations, and will be collected from different runs of LMT separations. An acceptable method would be to collect a portion of the heaviest fraction from the first LMT run, and the middle and

lightest fractions from the second run. The average residual remaining on the three size fractions will be reported as the result of residuals testing.

The collected sample portions will consist of approximately one pound of coal each, and will be packaged in one-gallon glass containers for shipment. The mineral samples will require 10-20 grams of sample to be collected from each fraction; these will also be packaged in glass. Matrix spikes will be prepared in the field consisting of a known mass of LMT solution added to a known mass of representative, clean coal and mineral samples; these will be included with the samples for analysis.

Testing of mineral and coal samples for lithium and tungsten concentrations will be performed at the Department of Toxic Substances Control Hazardous Materials Laboratory (DTSC HML, Berkeley, CA). Test samples will be prepared for lithium and tungsten analysis using EPA Method 3052, Microwave Assisted Acid Digestion of Organic Matrices, or equivalent. Residues of lithium and tungsten will be analyzed using EPA Method 7430, or equivalent.

The laboratory address and contact for the lithium and tungsten analysis is:

Hazardous Materials Laboratory
California Department of Toxic Substances Control
2151 Berkeley Way
Berkeley, CA 94704
Mr. Milad Iskander
(510) 540-3314

C. AQUATIC TOXICITY DETERMINATION

The use of fresh water organisms for acute toxicity testing has been referenced in 40 CFR Part 136 and constitutes an approved method suitable for determining the toxicity of specific compounds. Toxicity is measured using a definitive test consisting of five dilutions and a control. In the static-renewal test, the test organisms are exposed to a fresh solution of the same concentration of sample at each prescribed interval of time, by transferring the test organisms from one test chamber to another, or by replacing all or part of the test solution. This approach reduces the possibility of dissolved oxygen depletion, the buildup of metabolic wastes, or the loss of toxicants through volatilization or adsorption, and allows for the test organisms to be fed and therefore maintained in a healthier state.

CT&E, Denver will perform three separate aquatic toxicity tests of LMT utilizing the vertebrate fathead minnows, *Pimephales promelas*. In each test a minimum of ten fathead minnows will be exposed to five concentrations of LMT: 0.001, 0.002, 0.005, 0.01, and 0.02 percent, plus one control. Toxicity will be evaluated by determining the average of each of the three LC_{50} and the NOAEC values.

The acute toxicity test is designed to provide dose-response information expressed as the percent concentration which is lethal to 50% of the exposed test organisms within the prescribed period of time. The LC_{50} will be determined for each replicate set of dilutions by the Probit method. The NOAEC will be calculated using procedures utilized in EPA /600/4-90/027F. (For additional reference see Finney, D.J., *Statistical Method in Biological Assay*, 2nd. ed., Hafner, New York, 1964.) Note that any negative result from an acute toxicity test does not preclude the presence of chronic toxicity.

The aquatic toxicity testing procedure used for the LMT Verification Project is equivalent to both methods cited in the California Code of Regulations, Title 22, Section 66261.24, Determination of Toxicity (*Standard Methods for the Examination of Water and Wastewater*, 16th ed., American Public Health Association, 1985, Washington, DC, and *Static Acute Bioassay Procedures for Hazardous Waste Samples*, California Department of Fish and Game, Water Pollution Control Laboratory, 1988, Rancho Cordova, CA).

The laboratory address and contact for the aquatic toxicity tests:

Commercial Testing & Engineering Co.
4665 Paris Street, Suite B-200
Denver, CO 80239
Mr. Cris Theel
(303) 373-4772

D. END-USER SURVEY QUESTIONNAIRE

The end-user survey questionnaire will include a compilation of responses from end-users of the LMT product. Each of the end-users identified by LMT Inc. will be asked to reply to a written questionnaire which solicits the reasons they switched to the new product, the type of samples and analysis they are performing, their satisfaction with LMT, and any additional comments and concerns they may have. The end-user survey questionnaire for LMT is contained in Appendix D.

The addresses and contacts for LMT, Inc. are:

LMT, Inc.
230 South Rock Boulevard, Suite 21
Reno, NV 89502
Mr. Pat Costin
(702) 857-1993

John Moberly Associates
P.O. Box 2239
Olympic Valley, CA 96146
Mr. John W. Moberly, Ph.D.

(530) 581-0718

III. DATA VALIDATION AND ASSESSMENT

A. DOCUMENTATION AND RECORDS

CT&E laboratories will follow standard company laboratory practices for sample handing and chain-of-custody requirements during the gravity separation comparison. Any and all records documenting sample collection, chain-of-custody, testing procedures, and reporting of data, will be compiled in a laboratory data package. Upon evaluation, the data package will include the following documentation:

- Laboratory Analytical Report
- Laboratory Quality Control Report
- Notes from Laboratory Notebook or Other Field Notes
- Raw Data

The laboratory data package for the gravity separation comparison will be copied and sent to the Project Manager. The original laboratory data package for the gravity separation comparison will be archived at the CT&E laboratories in Denver and Henderson.

DTSC HML will follow standard laboratory practices for sample handing and chain-of-custody requirements during the residual product determination. Upon evaluation, the data package will include the following documentation:

- Analytical Request Form
- Sample Analysis Form
- Laboratory Analytical Report
- Laboratory Quality Control Report
- Notes from Laboratory Notebook
- Raw Data

The laboratory data package for the residual product determination will be copied and sent to the Project Manager. The original laboratory data package residual product determination will be archived at DTSC HML, Berkeley, CA.

CT&E , Denver, will follow standard company laboratory practices for sample handing and chain-of-custody requirements during the aquatic toxicity determination. A laboratory data package will be compiled, and will consist of any and all records documenting the creation of biological test samples, testing procedures, and reporting of data. Upon evaluation, the data package will include the following documentation:

- Laboratory Analytical Report
- Laboratory Quality Control Report
- Notes from Laboratory Notebook
- Raw Data

The laboratory data package for the aquatic toxicity determination will be copied and sent to the Project Manager. The original laboratory data package for the aquatic toxicity determination will be archived at CT&E, Denver.

B. QUALITY CONTROL REQUIREMENTS

CT&E's laboratory quality control practices are presented in their QA/QC manual, which is maintained at their facilities. DTSC HML quality control practices are found in the HML User's Manual, which is maintained at that facility. Additional quality control requirements are contained in both the ASTM and EPA test methods.

Instrumentation used during the separation procedures include perforated-plate sieves, hydrometers, and scales. The specification for perforated-plate sieves is found in ASTM Standard E 323, and the specifications and test methods for inspection and verification of hydrometers are in ASTM Standard E 100 and ASTM Standard E 126, respectively. CT&E follows calibration and verification schedules for their weights, balances, and scales stated in their laboratory quality assurance plan, which is maintained onsite at their facility. CT&E employs an independent subcontractor to calibrate their weights, balances, and scales with certified weights semi-annually. Photocopies will be made of calibration service certificates. Additional calibration and standardization data shall be recorded in a permanent log.

DTSC HML, Berkeley, will follow standard laboratory practices regarding instrument and equipment inspection, maintenance, and calibration requirements. Quality control procedures outlined in the EPA methods and the Hazardous Material Laboratory Manual shall be adhered to in all cases. Specifically, the residual product determination using ICP/MS shall include a provision to analyze blanks and determine percent recoveries from laboratory spikes and matrix spikes prepared in the field. The acceptance range for percent recoveries shall be from 80 to 120 percent.

Acceptance of data generated during the aquatic toxicity determination is predicated on the survivability of test organisms in the dilution water blank, and is detailed in the test method protocol. Specific requirements for this investigation include the test species, the solution concentrations, and the type of solution renewal. Fathead minnows, *Pimephales promelas*, are specified for this procedure. The test organisms will be exposed to five concentrations of LMT: 0.001, 0.002, 0.005, 0.01, and 0.02 percent, plus one control. Test solution renewal will be on a daily basis (static-renewal test type).

Calibration and standardization of instruments used for routine measurements such as pH, dissolved oxygen (DO), temperature, and conductivity, must be performed prior to

use each day according to the instrument manufacturer's procedures. Calibration and standardization data shall be recorded in a permanent log. Any and all wet chemical methods used to determine hardness, alkalinity, and residual chlorine shall be equivalent to those cited in EPA /600/4-90/027F. Standardization shall be conducted prior to use each day, and all data shall be recorded in a permanent log.

C. DATA REVIEW AND VALIDATION

Compliance with the requirements of the Workplan will be assured mainly through onsite observations made during the sample collection and testing events by DTSC's Project Manager and additional staff, as may be necessary. The observations will ensure that the test methods and procedures specified are followed satisfactorily.

CT&E gravity separation results will be used to determine if LMT performance differs significantly from that of the halogenated organic liquids. Performance will be quantified by determining the level of confidence at which the methods agree. DTSC HML test results for residual lithium and tungsten concentrations will be used to determine the amount of LMT remaining on tested samples after normal separation procedures are conducted. Based on the results of the testing, if LMT feels their performance expectations have not been met, a decision will be made to either modify the claims or discontinue the project.

CT&E aquatic toxicity tests will quantify the potential for harm to worker health or the environment. Calculations of the LC_{50} and NOAEC will be reviewed by Department toxicology and industrial hygienist specialists. The degree of indicated threat to worker health or the environment will be discussed with LMT Inc. and a decision will be made to either modify the claims or discontinue the project.

The results of DTSC's Project Team Review of all data, including that from industrial hygienists and toxicologists, will identify health, safety, or environmental issues that may arise from commercial use of LMT solution. All issues will be discussed with LMT Inc., and DTSC's Project Manager and LMT Inc. will discuss measures such as engineering or administrative controls to prevent potential harm to workers health and safety, or the environment.

Following completion of the Workplan, all test data, reviews, and comments will be combined into a Draft Verification Report. DTSC's Project Manager will provide a copy of the Report to the Project Team Members, to the U.S. EPA Project Manager and QA/QC Officer, and to LMT Inc. DTSC's Project Manager will then conduct a project review meeting to discuss any revisions to the Report. If inadequacies in the data are indicated at this time, DTSC's Project Manager will note these and offer:

- recommendations for repeated or additional field tests;
- suggested language reducing the scope of the verification; and/or
- proposed language for negative verification decisions.

Subsequent to these revisions, the Draft Verification Report will then be forwarded to the DTSC Technical Review Panel and U.S. EPA. Upon concurrence of the Technical Review Panel and U.S. EPA, the Final Verification Report will then be published.

Appendix A:

LMT Product Literature

Appendix B:

ASTM Test Methods

Appendix C:

LMT MSDS

Appendix D:

End-User Survey Questionnaire