

US EPA ARCHIVE DOCUMENT

FINAL
Technology Evaluation
Workplan
(ABB)

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TABLE OF CONTENTS

A. PROJECT MANAGEMENT1
 1. TITLE AND APPROVAL SHEET1
 2. DISTRIBUTION LIST2
 3. PROJECT ORGANIZATION3

B. PROJECT OVERVIEW5
 1. PROBLEM DEFINITION/BACKGROUND5
 2. PROJECT OBJECTIVES7
 3. PROJECT TASK DESCRIPTION8

C. MEASUREMENT / DATA ACQUISITION16
 1. SAMPLING PROCESS DESIGN16
 2. SAMPLING METHOD REQUIREMENTS16
 3. SAMPLE CUSTODY REQUIREMENTS17
 4. QUALITY CONTROL REQUIREMENTS18
 5. INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE
 REQUIREMENTS24
 6. INSTRUMENT CALIBRATION AND FREQUENCY24
 7. DATA MANAGEMENT24

D. ASSESSMENT / OVERSIGHT24

E. DATA VALIDATION AND USABILITY25
 1. DATA REVIEW, VALIDATION, AND VERIFICATION REQUIREMENTS25
 2. VALIDATION AND VERIFICATION METHODS25
 3. RECONCILIATION WITH DATA QUALITY OBJECTIVES25

REFERENCES26

Appendix A, Performance Test Methods and Laboratory Standard Operating Procedures (SOPs)

Appendix B, DTSC HML Data Quality Control Practices

Appendix C, Selected Performance Values for Exxon Univolt 61 or N61

LIST OF TABLES

Table 1, Summary of 1992 PCB Waste Generation - Electric Utility6
Table 2, Verification/Certification Claims for BIOTEMP®7
Table 3, Proposed Performance Verification/Certification Tests and Criteria12
Table 4, Laboratory Facilities and Points of Contact17
Table 5, Field Monitoring, Sampling, and Analytical Methods20

LIST OF FIGURES

Figure 1, Project Organization Chart4
Figure 2, Technology Verification/Certification Schedule15

A. PROJECT MANAGEMENT

1. TITLE AND APPROVAL SHEET

Department of Toxic Substances Control

Mr. Tony Luan, P.E.
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Ms. Suzanne Davis
Technical Project Manager

US Environmental Protection Agency

Ms. Norma Lewis
Technical Project Manager

Dr. Ruth Chang
DTSC Quality Assurance Officer

2. DISTRIBUTION LIST

Mr. Tony Luan, DTSC/OPPTD (Primary Decision Maker)
Dr. Ruth Chang, DTSC HML (QA/QC Reviewer)
Mr. Dick Jones, DTSC/OPPTD (Project Reviewer)
Mr. John Wesnousky, DTSC/OPPTD (Project Reviewer)
Ms. Norma Lewis, U.S. EPA (Technical Project Manager)
Ms. Laura Drees, U.S. EPA (QA/QC Officer)
Mr. Jim Baker, ABB T&D Power, Inc.

3. PROJECT ORGANIZATION (see Figure 1)

Department of Toxic Substances Control

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

Project Manager - Suzanne Davis is responsible for overseeing implementation of the Technology Evaluation 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 is responsible for ensuring the data collection system meets QA/QC requirements.

Planning Team Members - All team members are responsible for participating in plan preparation activities, project meetings and reviewing project reports. Each member of the project team was selected based on experience, responsibility, or authority.

U.S. Environmental Protection Agency

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

QA/QC - Lauren Drees is responsible for providing U.S. EPA QA/QC review of the Technology Evaluation Workplan and data analysis.

Project Reviewers - U.S. EPA project team members are responsible for reviewing DTSC project team activities and reports.

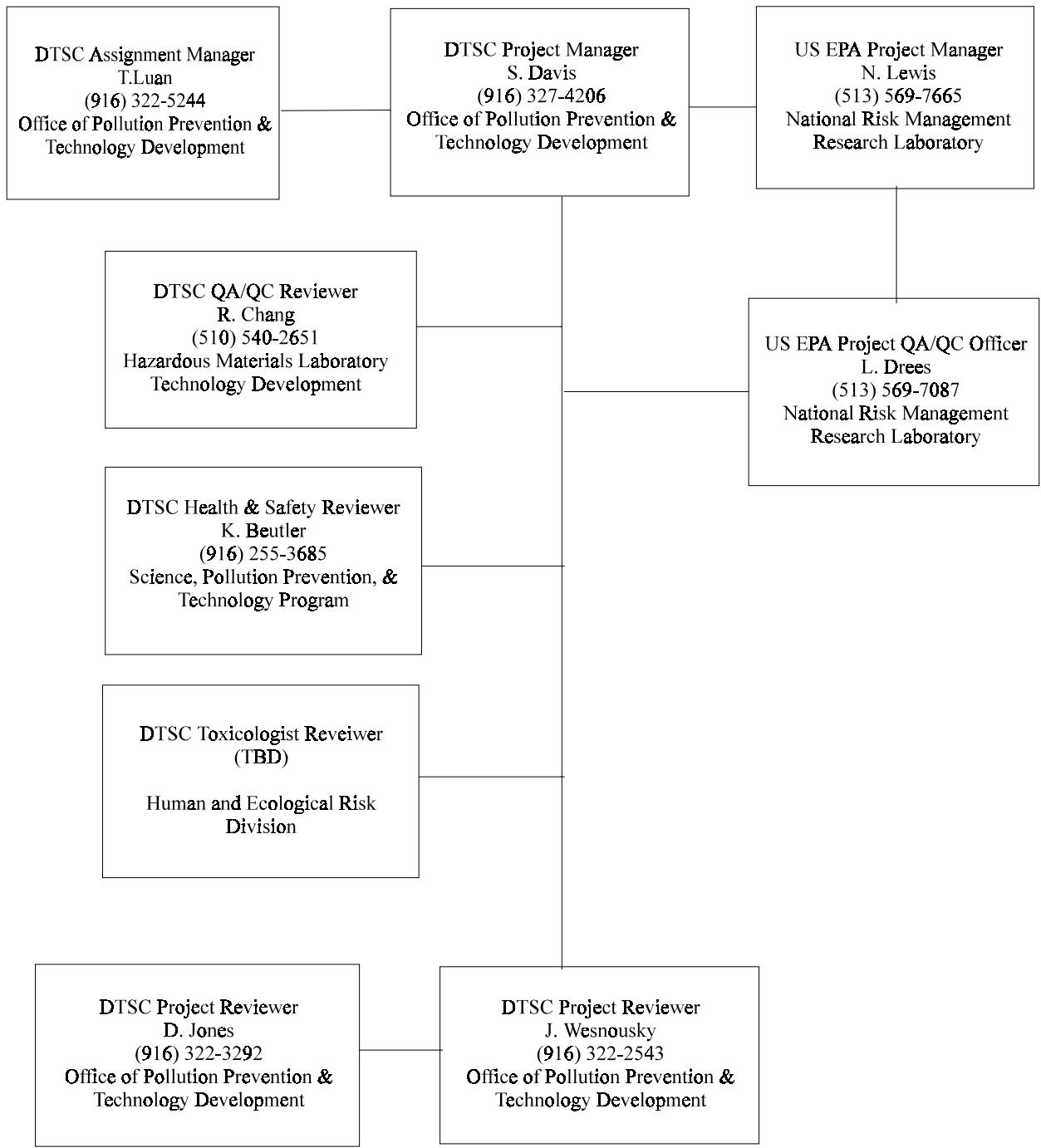


Figure 1. Project Organization Chart

B. PROJECT OVERVIEW

1. PROBLEM DEFINITION/BACKGROUND

Problem Background

Electric power utilities use electrical transformers for a variety of applications, including power distribution. The transformers generate significant amounts of heat, and must contain cooling/insulating (dielectric) mediums to prevent gas formation, electrical shorts, fire or explosion, and transformer damage. Most transformers currently use some type of mineral oil as the cooling fluid; however HTHs and synthetics (less-flammable fluids) are used in transformers that must operate in safety-related applications (near or inside buildings). Recently, transformer fluid vendors have developed vegetable seed oil-based dielectric fluids. These fluids have been certified as meeting “less-flammable” safety-related requirements by organizations such as Underwriters Labs or Factory Mutual.

Typically, liquid-containing distribution class transformers store from 30 to 1000 gallons of oil. Spills from transformers are potentially an environmental concern because even small amounts of oil can contaminate bodies of water, possibly deplete oxygen, coat plant and animal life, be toxic or form toxic products, affect breeding, produce rancid odors, or foul shorelines or other habitats. Effects on soils are not as well characterized.

Polychlorinated Biphenyls (PCBs) are still in use but no longer produced because of their high toxicity - they are regulated under the federal Toxic Substances Control Act (TSCA). Dielectric fluids and electric equipment with dielectric fluids treated under TSCA are not regulated under the federal Resource Conservation and Recovery Act or RCRA (40CFR 261.8). Non-PCB transformer fluids do not meet the requirements for regulation as hazardous waste under RCRA; however, mineral oils that have been in service for approximately 10 years have exceeded California’s acute toxicity levels for copper due to leaching from the transformer coils.

Under the Clean Water Act (CWA), EPA’s Spill Prevention, Control, and Countermeasure (SPCC) requirements apply to facilities that are non-transportation related or fixed, and could reasonably be expected to discharge any type of oil into or upon the navigable waters of the United States. The CWA has been interpreted to cover all surface waters of the United States, including intermittently dry creeks through which water may flow and ultimately end up in public waters. To be regulated, the facilities must also have an aboveground oil storage capacity of more than 660 gallons in a single container or a total aboveground capacity of more than 1320 gallons, or a total underground capacity of more than 42,000 gallons.

Table 1 illustrates the types and amounts of waste oil change-outs, spills, and associated clean-up costs that a small to medium-sized electrical utility transmission system monitoring and maintenance facility experienced in 1992. This facility, which is only one of several operated by the electrical utility, generated 155 tons of spilled oil and contaminated soil, most of which was caused by accidents involving utility poles and transformers.

TABLE 1. SUMMARY OF 1992 PCB WASTE GENERATION - ELECTRIC UTILITY

Waste Generated	Annual Quantity Generated (tons)	Annual Costs (\$)
Oil Spill and Leak Residue	155	46,000
Waste Oil from Electrical Transformers	126	100,000
Wastes Containing PCB	28	50,000

Source of Waste: Primarily damage to transformers

Source of Waste: Draining of oil prior to reconditioning or decommissioning transformers

Source of Waste: Primarily damage to transformers and PCB recovery

Source: U.S. EPA, Risk Reduction Engineering Laboratory, EPA/600/S-92/063 - October 1992

Problem Definition

Used mineral oil from transformers may be hazardous due to PCB contamination, and in California, from copper leaching from the transformer coils. (U.S. EPA does not regulate copper under RCRA.) Often, municipal landfills do not allow disposal of mineral oil or other free liquids, or soil containing mineral oil from transformer spills because of concerns that the soil may be hazardous. Dielectric fluid manufacturers have been searching for fluids that have the same desired or better performance qualities as mineral oil, but are less toxic and biodegrade quickly in the advent of a spill. Vendors have recently developed vegetable seed oil-based dielectric fluids that they believe meet the desired characteristics.

Problem Resolution

Renewable sources of oil from vegetable seeds often have higher viscosity indices, lower evaporation losses, and higher flash and fire points than petroleum-based oils. However, their thermal, oxidative, and hydrolytic stabilities may be lower than petroleum-based oils due to the carbon-carbon double bond of the triglycerides contained in the vegetable oils. High oleic vegetable seed-based oils with antioxidant additives have been developed to replace mineral oil in electrical power distribution transformers. Vendors claim that the oils meet or exceed industry-established performance requirements, are less toxic, and quickly biodegrade. Stakeholders such as utility companies and regulators need verified results of the above claims. Standard laboratory tests of transformer fluid performance have been developed and performed. Standard acute toxicity tests have been performed. Some biodegradation tests have been developed and performed; however, there are concerns that such tests are not necessarily appropriate for vegetable oils, nor for spills onto soil. However, tests specific to soils and appropriate for measuring vegetable oil biodegradation need to be developed and performed. These concerns are outlined later in the workplan in the biodegradation section on pg 9.

ABB is an international corporation and major supplier of transformers and other electrical related equipment. ABB has developed and brought to market a vegetable seed oil-based dielectric fluid called BIOTEMP[®]. Approximately 20 transformers have been supplied with BIOTEMP[®] fluid. Of these 20 transformers, 10 transformers are in-service. To verify the claims listed in Table 2 below, DTSC will conduct the following tests on virgin BIOTEMP[®] fluid; performance tests per ASTM Oil Specification Test Methods D3487 and D5222, acute toxicity tests, and aquatic biodegradability tests. Sampling, analyzing, and testing oil from units that have been in service will also be conducted as part of this Verification/Certification. Existing data will be reviewed for data quality and used (if determined adequate) to compare estimated cost impacts associated with BIOTEMP[®] and mineral oil.

2. PROJECT OBJECTIVES

The project objectives are to verify the technology claims agreed upon by ABB and the agencies which are listed in Table 2 below. Other objectives not included in the Verification/Certification claims are: (1) the worker health and safety aspects of BIOTEMP[®], (2) comparison of the chemical composition of virgin BIOTEMP[®] fluid to the applicant’s formulation specifications, (3) speciation of chemical composition, and (4) estimation of costs based on the expected life of BIOTEMP[®] as compared to those of mineral oil.

Table 2. Verification/Certification Claims for BIOTEMP[®]

Number	Claim
1	<u>General Performance</u> : In the following composition ratio(s) (98.5% vegetable oil, 1.5% additives), meets criteria for oxidative, thermal, and chemical stability, as measured by Oil Qualification Tests - ASTM D3487 (Mineral Oil) and ASTM D5222 (High Temperature Hydrocarbons).
2	<u>Aquatic Biodegradability</u> : BIOTEMP [®] biodegrades 97% in 21 days, based on the average of several performance tests as measured by the Coordinating European Council (CEC) Test Method CEC-L-33-A-93. ³
3	<u>Flammability</u> : Has a Flash Point of at least 300°C, and a minimum Fire Point of 300°C, based on the average of several performance tests by independent labs performing ASTM D92 (Cleveland Open Cup). ⁴
4	<u>Acute Toxicity</u> : Results for virgin product tested by U.S. EPA/600/4-90/027F Test for Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms does not meet the toxicity characteristic criteria in Code of California Regulations, Title 22, Section 66261.24(a)(6).

Both the manufacturer and utilities have recommended that the agencies focus on environmental performance measures, such as toxicity and biodegradation, because (1) large amounts of laboratory performance data generated under industry standards are available, (2) DTSC’s expertise is in the environmental performance areas, and (3) project restraints on time and resources are limited. In addition, the cost impact on transformers using vegetable oil-based dielectric fluids are of interest to users. The cost estimates will be based on limited, existing data for BIOTEMP[®] provided by ABB. Additional chemical and environmental composition data will also be obtained as part of this workplan.

3. PROJECT TASK DESCRIPTION

To validate ABB’s claims, four broad sets of measurements are required which are performance testing, chemical composition, aquatic biodegradation testing, and acute toxicity. Information from three of these measurements (chemical composition, acute toxicity, and aquatic biodegradation) are needed by the agencies to evaluate environmental impacts.

The following sections present the analytical methods to be used for verifying each claim listed in Table 2 and other Verification/Certification tests determining the product properties and chemical composition.

Verification/Certification Claim #1 - General Performance

Performance Tests - Numerous ASTM and ANSI methods are available in the general areas of oxidative, thermal, and chemical stability; physical and electrical properties, and extended life analysis. The industry has developed a standard suite of dielectric fluid performance tests through the ANSI consensus-based standards development process. No standard suite of tests exist for vegetable oil-based dielectric fluids; however, two ASTM suites of standard tests have been developed: ASTM D3487 (mineral oils) and ASTM D5222 (High Temperature Hydrocarbons, or HTHs). The most significant difference in the two suites are the specifications for flammability and fire points. Typically, the specifications for HTHs are used when the transformer may be used in areas where people may be present, such as buildings. End-users and the vendors have identified the subset of performance tests as those appropriate for determining if there has been a loss of performance or fluid quality over time in use. Care must be taken when using these tests to measure vegetable oil-based fluids' quality or performance because not all tests or criteria are appropriate for vegetable oils.

Table 3 lists the proposed Verification/Certification tests which are also selected purchase specifications. To minimize the effects of sampling for in-service fluids, performance tests requiring a large volume of fluid such as dielectric breakdown (impulse) by ASTM D3300, dielectric breakdown (gap) by ASTM D1816, and oxidation stability (rotary bomb) by ASTM D2112 will not be conducted. Oxidation stability by ASTM D2440 will be limited to a single tube for either 72 or 164 hours for the in-service fluid. Table 5 lists the performance tests to be conducted on both the virgin and in-service fluids. A reference oil, Exxon Univolt 61 or N61, will be also tested for the same performance parameters as the virgin fluid as part of this evaluation's QA/QC measures.

Existing data for four samples of virgin fluid have been reviewed and tentatively rejected due to non-independence: sampling was performed by the vendor. Existing performance data submitted to date has consisted of; three samples tested for thermal aging and oxidation stability using ASTM Method D2112, electrical stress by ATM Method D2200, gas production due to electrical arcing, and chemical structure by infrared spectras; and one sample tested for dielectric strength by ASTM Method D877, neutralization number by ASTM Method D974, moisture content by ASTM Method D1533, viscosity by ASTM Method D445, color by ASTM Method D1500, and flash and fire point by ASTM Method D97. This existing data will be accepted for use in the project if the virgin fluid data generated as part of this workplan are similar. If existing data are not available or do not pass the QA/QC review, then it shall be limited to use in the vendor section of the report.

Verification/Certification Claim #2 - Aquatic Biodegradability

Biodegradation - numerous lab test methods exist for aquatic biodegradation, less for soil biodegradation. Some methods measure biodegradation by amount of organic carbon fully mineralized to CO₂, some by measuring dissolved oxygen, and some by identifying and quantifying intermediate degradation products. However, the few soil biodegradation field test protocols available are expensive and time-consuming. The U.S. EPA's Oil Spill Program experts are not satisfied with the available field test protocols and are currently developing a new soil biodegradation protocol for vegetable oils. Due to the lack of basic research on the degradation products for vegetable oil and proven test guidelines for measuring the biodegradation of vegetable oil in soil, the soil biodegradation Verification/Certification is not included in this Verification/Certification.

Proposed Aquatic Biodegradation Test Method -

The Coordinating European Council (CEC) has developed a test which was originally intended to measure the biodegradability of hydrocarbons, specifically two-stroke motor oils, in water: CEC-L-33-A-93. This method compares the biodegradation potential of the test oil against the standard oils specified in the test method. The test oil and the standard oil are placed in separate flasks containing an inoculum/mineral substrate mixture. The extract solutions from these flasks are collected on the zero-day and after the 21-day incubation period. The extract solution is analyzed by infrared spectroscopy (IR) measuring the maximum absorption of the oil at the C-H stretch of the CH₂-CH₃ band at the wavelength of 2930 cm⁻¹ " 10cm⁻¹. The biodegradability is expressed as a percent difference in the residual oil contents between the poisoned flasks and the respective test flasks.

Verification/Certification Claim #3 - Flammability

Flammability - the flash point and fire point for virgin and in-service BIOTEMP fluid will be determined using ASTM Method D92, Cleveland Open Cup test. Table 3 lists the purchase specification and mineral oil criteria for each method. Table 5 provides specific information on the number of samples and frequency which the samples will be collected.

Verification/Certification Claim #4 - Acute Toxicity

Acute Toxicity (Fish Bioassay) - to verify acute toxicity data, compare toxicity to existing regulatory action levels, and partially address concerns about spills near receiving waters. The EPA method listed below was selected to verify the acute toxicity claim listed in Table 2. A screening and definitive test will be conducted for a 96-hour test period using the test organism, *oncorhynchus mykiss*. Samples will be prepared by the "Static Acute Bioassay Procedures for Hazardous Waste Samples" developed by the California Department of Fish and Game, Water Pollution Control Laboratory in the Code of California Regulations, Title 22, Section 66261.24(a)(6). Results will be compared to the toxicity characteristic listed in the Code of California Regulations, Title 22, Section 66261.24(a)(6). The virgin oil is considered to exhibit a toxic characteristic if the LC50 is less than 500 milligrams per liter when measured in soft water (total hardness 40 to 48 milligrams per liter of calcium carbonate).

Test Method: *Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms*, EPA/600/4-90/027F, August 1993.

Test Organism: *Oncorhynchus mykiss* (rainbow trout)

Other Verification/Certification Tests:

Chemical composition - to verify composition of fluids, to verify the formulator meeting selected purchase specifications, and to establish a baseline for measuring potential metals leaching and oil degradation under electrical loading and over time. Where appropriate, testing method(s) will be the same as those used by the applicant to allow correlation of newly generated data with that of existing applicant-generated data.

To create a chemical "fingerprint" for BIOTEMP fluid and verify the chemical composition, samples will be collected and analyzed for specific vegetable oil characteristics by the Association of Analytical Chemists (AOAC) methods listed below, for semivolatile organics by EPA Method 8270, and for trace metals analysis by EPA Method for SW6010 (see Table 5). Additional samples for volatile organics by EPA Method 8260B may be collected by DTSC to verify the chemical composition if deemed necessary.

Vegetable oils are principally composed of fatty acids such as triglycerides. Chemical analysis of such oils is usually achieved through solvent extraction, derivatization from Fatty Acid Methyl Esters (FAMES), and identification and quantification on a gas chromatograph-mass spectrometer. Identification can be further specified by the number of carbon atoms and location of double bonds by indicating the location of the first double bond relative to the methyl end of the molecule. In addition to the oils, the antioxidants will also be identified and quantified through chromatography (High Pressure Liquid Chromatography, or HPLC).

- C AOAC Official Method 981.11, Oils and Fats, Preparation of Sample (98);
- C AOAC Official Method 972.28, Total Fatty Acids in Oils & Fats, Hexane Distillation (97);
- C AOAC Official Method 963.22, Methyl Esters of Fatty Acids in Oils and Fats, Gas Chromatographic Method (98);
- C AOAC Official Method 983.15, Phenolic Antioxidants in Oils, Fats, and Butter Oil, Liquid Chromatographic Method (94); and
- C AOAC Official Method 977.17, Polymers and Oxidation Products of Heated Vegetable Oils, Gas Chromatographic Method for Non-Elution Materials (95).

Table 3. PROPOSED PERFORMANCE VERIFICATION/CERTIFICATION TESTS AND CRITERIA

Test Method	D3487 Specifications (Mineral Oils)		D5222 Specifications (High Fire Point Oils)	
	Mineral Oil Specifications	ABB Specifications for BIOTEMP®	HTH Typical Values	ABB Specifications for BIOTEMP®
PHYSICAL TESTS				
Flash Point, min, EC, D 92 (Cleveland Open Cup)	145	>300*	N/A	N/A
Fire Point, min, EC, D 92 (Cleveland Open Cup)	N/A	N/A	304-310	> 300
Pour Point, max, EC, D 97	-40	-15 to -25*	-24	-15 to -25*
Viscosity, max, cSt, D 445, 100EC	3	10*	11.5-14.5 ^a	10*
40EC	12	45*	100-140 ^a	45*
0EC	76	300*	1800-2200 ^a	300*
ELECTRICAL TESTS				
Dielectric Breakdown, D 877, kV, min	30	30*	42	30*
Dielectric Breakdown, D 1816, kV, 1.02 mm gap, min	28	28*	30	28*
2.03 mm gap, min	56	65*	61	65*
Dielectric Breakdown, impulse, D 3300, min, kV, Needle Negative	145	100*	N/A	N/A
Needle Positive		90	N/A	N/A
Dissipation Factor (or power factor), max, D 924, 25EC	0.05	0.05*	0.01	0.05*
100EC	0.30	2.00*	0.30	2.00*
CHEMICAL TESTS				
Oxidation stability, sludge, acid number, inhibited, D 2440	passes test	passes test	passes test	passes test
Oxidation stability, rotary bomb, min, minutes, inhibited, D 2112	195	200	800-1000 ^a	200
			Water, max, ppm, D 1533	35 ^b

Test Method	D3487 Specifications (Mineral Oils)		D5222 Specifications (High Fire Point Oils)	
	Mineral Oil Specifications	ABB Specifications for BIOTEMP [®]	HTH Typical Values	ABB Specifications for BIOTEMP [®]
<p>*Purchase specification value. ^aNot critical if the oil meets this criteria. ^bAlthough the water content value for BIOTEMP is higher than the water content values listed for the mineral oil and HTH fluid, this higher water content will not reduce the dielectric strength to unacceptable levels or result in the formation of a separate water (ice) layer. Therefore, water content results will be compared to its purchase specification value instead of the ASTM D3487 or D5222 values.</p>				

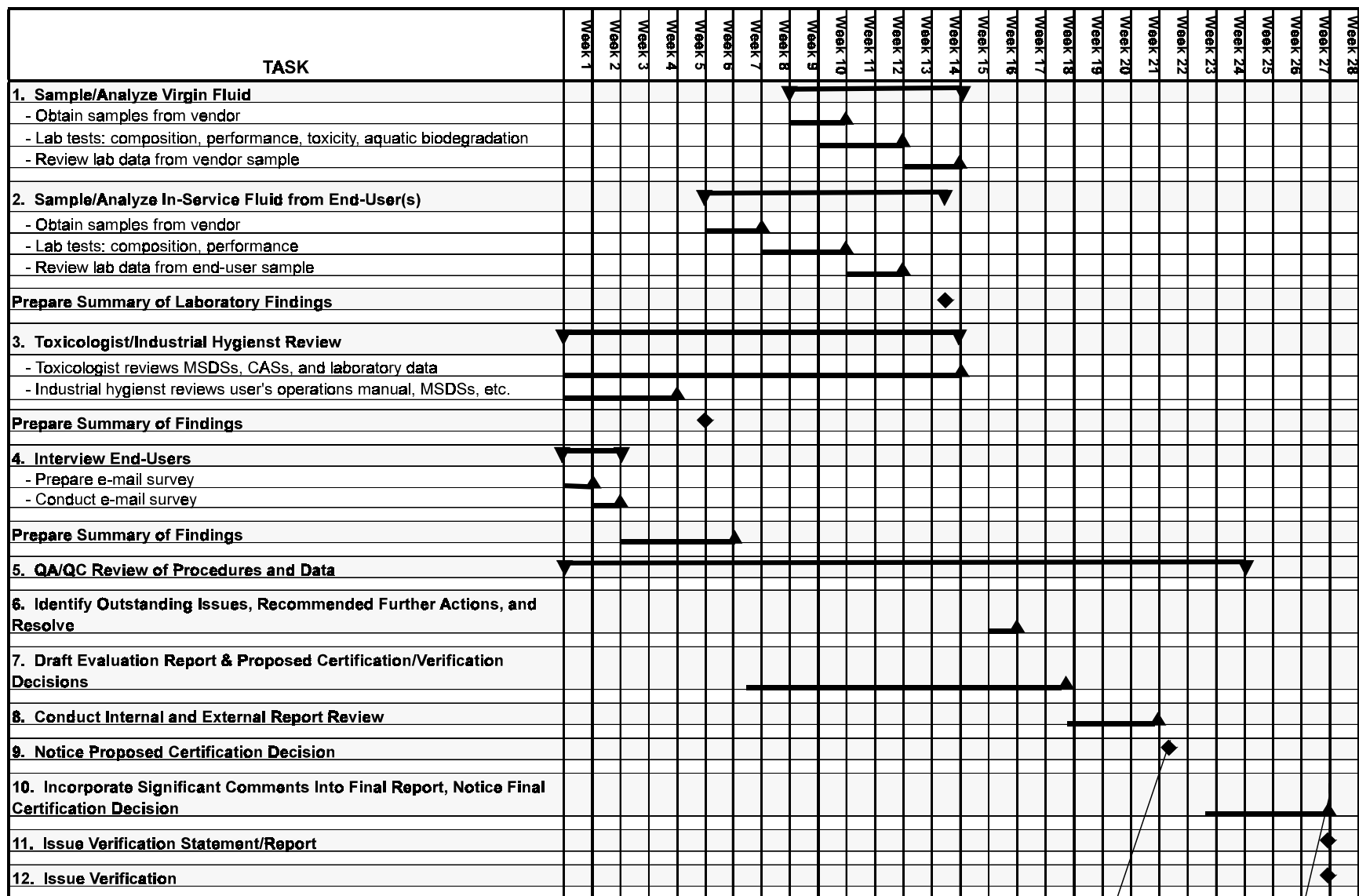
Toxicologist/Industrial Hygienist Review

Another activity is to evaluate the worker health and safety aspects of BIOTEMP[®]. A DTSC Toxicologist will review existing data consisting of material safety data sheets (MSDS), chemical abstract studies (CAS), and existing chemical composition, acute toxicity and biodegradation data provided to DTSC by the applicant. Existing data will be reviewed using the same QA/QC requirements described in Sections C4 through C6. Existing data for five samples of virgin fluid have been reviewed and tentatively rejected due to non-independence: sampling was performed by the vendor. The existing data submitted to date has consisted of; two samples tested for acute toxicity using the same EPA method and approach described for Verification/Certification Claim #4; and three samples tested for biodegradability by CEC-L-33-A-93. This existing data will be accepted for use in the project if the virgin fluid data generated as part of this workplan are similar. If existing data are not available or do not pass the QA/QC review, then it shall be limited to use in the vendor section of the report. A summary of the toxicologists findings will be prepared for the DTSC Project Manager and incorporated into the Verification/Certification report.

A DTSC Industrial Hygienist will review proposed sampling procedures, MSDSs, and other safety related information to evaluate and approve hazard appraisal recognition plan (HARP) forms submitted by DTSC staff for site visits.

A proposed schedule outlining the activities required to evaluate ABB’s technology is shown in Figure 2. The main activities are chemical composition, toxicity, performance and aquatic biodegradation testing. Following these tests, the project team members will meet, discuss data results, and/or identify additional data needs. DTSC’s Project Manager will prepare evaluation summaries following each major activity in the workplan, and a Verification/Certification statement and report will be prepared. Section C7 identifies the supporting documentation and records required for the Project Team’s evaluation.

Figure 2. ABB VERIFICATION/CERTIFICATION SCHEDULE



C. MEASUREMENT / DATA ACQUISITION

1. SAMPLING PROCESS DESIGN

Samples of virgin fluid will be taken at the dielectric fluid formulating facility and analyzed to confirm the fluid meets the Verification/Certification claims listed Table 2, and verify the chemical composition. Results from this evaluation will also establish a baseline for identifying changes in composition or toxicity of in-service fluid, and comparing existing and future data. The results will also be used to compare the selected performance parameters of the BIOTEMP[®] dielectric fluid to the criteria listed in Table 3.

Samples of in-service fluid will be taken from transformers that have been in use for at least one year in a regular sampling/testing environment. The main purpose for sampling and analyzing the in-service fluid is to determine changes in composition or metal concentrations over time and to verify the chemical composition of the in-service fluid. The second purpose is to measure any potential changes in key fluid performance parameters.

Table 5 lists the number of samples to be collected for performance testing, acute toxicity, aquatic biodegradation, and chemical composition. The proposed schedule for sampling, analyses, performance tests, and peer review is shown in Figure 2. Section C2 lists the specific sampling requirements.

2. SAMPLING METHOD REQUIREMENTS

Virgin Fluid: Virgin dielectric fluid samples will be collected by a representative from the dielectric fluid manufacturing facility under DTSC oversight. The samples of virgin product will be representative of commercially available lots from the manufacturer as retail outlets do not supply this product. At least four samples from three different lots shall be obtained. A total of 12 samples will be collected but only four samples will be analyzed per the methods listed in Table 5. Disposable sampling equipment shall be used to reduce the potential of cross contamination between samples.

To ensure independent and representative samples are collected, DTSC personnel will oversee the sample collection from the production area. Samples will be assigned a field sample identification number determined prior to sampling. Proper chain of custody and storage procedures will be followed. The labs will send the data directly to DTSC. The vendor may take split lots to run tests at their own facility if desired.

In-Service Fluid: Sampling of in-service fluid will be conducted by end-user representatives under DTSC oversight and in conjunction with the normal on-going sampling program. At least 4 transformers will be sampled. Samples of in-service fluid will be limited to one sample per transformer to minimize the amount of fluid removed from each transformer and the impact to the ongoing test program. Each in-service fluid sample will be analyzed by the chemical composition methods listed in Table 5. Performance test results from the end-user's on-going sampling program will be used which consists of flash point, fire point, conductivity, power factor, and moisture content. New Tygon tubing connectors will be used at each transformer fluid sampling port to reduce the potential of cross contamination between samples.

Performance testing will be conducted by Doble Engineering Company, an independent testing facility, qualified in performance tests of dielectric fluids used in power distribution transformers. Acute toxicity testing will be conducted by Associated Labs. The DTSC HML laboratory will perform the EPA Method 8270/3580A and 6010/3031 analysis. The aquatic biodegradation will be conducted by Powertech Labs, Inc.. The AOAC chemical composition analyses will be performed by Krueger Food Laboratories.

**Table 4
Laboratory Facilities and Points of Contact**

Laboratory Name	Address	Contact Person	Analyses to be Performed
Doble Engineering Company	85 Walnut Street Watertown, MA 02472	Eileen Finnan	ASTM Methods
Associated Labs	806 North Batavia Orange, CA 92688	Pam Schiro	Acute Toxicity (Fish Bioassay)
Powertech Labs, Inc.	12388 88th Avenue Surrey, B.C. Canada V3W7R7		CEC-L-33-A-93/94
DTSC HML	700 Heinz Street, Suite 150 Berkeley, CA 94170	Pam Schiro	EPA Method 8270/3580A and 6010/3031
Krueger Food Laboratories	24 Blackstone Street Cambridge, MA 02139-3170	Dana Krueger	AOAC Methods

3. SAMPLE CUSTODY REQUIREMENTS

DTSC has established procedures for maintaining the control and integrity of samples from collection, preservation, transportation, testing, storage, and disposal. DTSC’s representative will label samples, maintain field notes, complete the chain of custody (COC) or HML’s sample analysis request (SAR) forms, and package and ship samples to the appropriate laboratories. DTSC’s representative will also sign and date each COC or SAR, and retain a copy in the project files. Upon receipt of the samples, the laboratory will sign and date the COC or SAR and contact the DTSC project manager about broken containers, and discrepancies on the COC form. DTSC’s laboratory sample control procedures are identified in **Appendix B**. These procedures or equivalent sample control procedures will be followed.

4. QUALITY CONTROL REQUIREMENTS

4.1 General Quality Control Requirements

Quality Control requirements shall follow the principles and guidelines of ANSI/ASQC E4-1994 (Ref 2). The testing facility shall report the statistics for the full group of fluids.

Performance testing will be conducted on one sample of Exxon Univolt 61 or N61, the reference mineral oil. Results for this mineral oil will be compared to published performance specifications for this oil and used to evaluate the laboratory’s performance for ASTM methods. The published performance specifications are provided in Appendix C.

4.2 Sample Collection

One equipment blank will be collected and analyzed for semivolatile organics by EPA method 8270 and trace metals by EPA method 6010. The equipment blank will consist of DI water rinseate for the Tygon tubing connection used to collect in-service transformer oil samples. For the virgin fluid, the equipment blank will consist of DI water rinseate from the sampling equipment.

One field blank will be collected per sampling event and sent to the DTSC laboratory. The field blank will consist of DI water (HPLC grade or pesticide grade) used to fill an empty sample container in the field. One sample will be analyzed per sampling event by EPA method 8270.

Four duplicates will be collected for the virgin BIOTEMP fluid but only one sample will be analyzed per the methods listed in Table 5. No duplicates will be collected for the in-service fluid samples to minimize the impact on the transformer sampled.

Samples shipped to the DTSC HML laboratory will be iced and kept at a temperature below 4EC. Samples shipped to the other laboratories will be stored within ice chest to keep samples out of direct sun. Sample containers will be supplied and prepared by laboratories per the particular method requirements.

4.3 Method Requirements

4.3.1 Performance Method

Results for the ASTM performance tests are acceptable if they meet the QA/QC requirements outlined in precision sections for each test method. When the method does not provide criteria, then the results will be reviewed qualitatively. If the test criteria are not met, then the laboratory will notify DTSC's manager on the corrective actions taken or if retesting is required.

4.3.2 Aquatic Biodegradability

Results for the aquatic biodegradability tests are acceptable if they meet the QA/QC requirements outlined in Section 7.6, Restrictions and Section 7.8, Precision of CEC Method CEC-L-33-A-93/94. If the criteria of the tests are not met, then the laboratory will notify DTSC's manager on the corrective actions corrective actions taken or if retesting is required.

4.3.3 Acute Toxicity

Results for the acute toxicity tests are acceptable if they meet the QA/QC requirements outlined in Section 9.16 and Table 14 of the method. If the criteria of the tests are not met, then the laboratory will notify DTSC's manager on the corrective actions corrective actions taken or if retesting is required.

4.3.4 Chemical Composition

Results for the AOAC tests are acceptable if they meet the QA/QC requirements outlined in each individual method. If the criteria of the tests are not met, then the laboratory will notify DTSC's manager on the corrective actions taken or if retesting is required. For AOAC Method 983.15, the method performance criteria is provided in Table 983.15A for seven different antioxidants. Due to this ingredient's proprietary nature, the exact antioxidant will not be listed in this workplan. However, the laboratory will be contacted and provided this information prior to sampling.

For EPA Methods 8270/3580A and 6010/3031, QA/QC methods outlined in the method and DTSC's HML User manual QC section. Matrix spike samples shall be made at ten times the detection limit. Matrix spikes and duplicates will be accepted per the criteria listed in the method and in Table 4.4-2 in the HML User's Manual.

Table 5 - Field Monitoring, Sampling, and Analytical Methods

Sample	Type	Parameter(s)	Frequency	Location	Method(s)	Container Type	Preservative(s)	Holding Time		
Performance (see Footnote 2 & 3)										
Virgin Fluid	(Physical)	Flash Point* Fire Point* Pour Point* Viscosity (100, 40, and 0 degrees C)	Collect four samples per lot (total of 12 samples) Analyze only four samples (one sample per lot and one duplicate)	Dielectric fluid formulation facility	ASTM D92 ASTM D92 ASTM D97 ASTM D445	2.5 L amber P* 25 ml amber P	None	None		
	(Electrical)	Dielectric Breakdown* Dielectric Breakdown (gap) Dielectric Breakdown (Impulse) Dissipation Factor (25 & 100)*			ASTM D877 ASTM D1816 ASTM D3300 ASTM D924	450 ml amber P 2.0 L amber P				
	(Chemical)	Oxidation stability, sludge, acid number, inhibited Oxidation stability, rotary bomb, min, minutes, inhibited Water Content*			ASTM D2440 ASTM D2112 ASTM D1533	150 ml amber P 125 ml amber P				
	Dielectric Fluid Chemical Composition	Methyl Esters			AOAC Official Method 963.22	60 ml amber P			None	None
		Oils and Fats			AOAC Official Method 981.11	60 ml amber P				
		Total Fatty Acids			AOAC Official Method 972.28	60 ml amber P				

Sample	Type	Parameter(s)	Frequency	Location	Method(s)	Container Type	Preservative(s)	Holding Time
		Phenolic Antioxidants			AOAC Official Method 983.15	60 ml amber P		
		Polymers and Oxidation Products			AOAC Official Method 977.17	60 ml amber P		
Virgin Fluid (cont.)	Dielectric Fluid Chemical Composition (cont.)	Semivolatiles organics (screening)	Collect four samples per lot (total of 12 samples) Analyze only four samples (one sample per lot and one duplicate)	Dielectric fluid formulation facility	EPA Method 8270/3580A	250 ml amber G	< 4EC	7 days to extract; 40 days to analyze after extraction
		Trace metals (As, Ba, Be, Cd, Co, Cr, Cu, Mo, Ni, Pb, Se, Tl, V, Zn)			EPA Method 6010/3031	100 ml amber P	< 4EC	6 months
	Acute Toxicity	LC50 LOEC NOEC	Collect two samples per lot (total of 6 samples) Analyze only three samples		see footnote 1	100 ml, amber G	None	None
	Aquatic Biodegradation		Collect two samples per lot (total of 6 samples) Analyze only three samples		CEC-L-33-A-93	150 ml, amber G		
Reference Oil (Exxon Univolt 61 or N61)	(Physical)	Flash Point* Fire Point* Pour Point* Viscosity (100, 40, and 0 degrees C)	Collect and analyze one sample total	Dielectric fluid formulation facility	ASTM D92 ASTM D92 ASTM D97 ASTM D445	2.5 L amber P* 25 ml amber P	None	None

Sample	Type	Parameter(s)	Frequency	Location	Method(s)	Container Type	Preservative(s)	Holding Time
	(Electrical)	Dielectric Breakdown* Dielectric Breakdown (gap) Dielectric Breakdown (Impulse) Dissipation Factor (25 & 100)*			ASTM D877 ASTM D1816 ASTM D3300 ASTM D924	450 ml amber P 2.0 L amber P		
Reference Oil (Exxon Univolt 61 or N61) (cont.)	(Chemical)	Oxidation stability, sludge, acid number, inhibited Oxidation stability, rotary bomb, min, minutes, inhibited Water Content*	Collect and analyze one sample total	Dielectric fluid formulation facility	ASTM D2440 ASTM D2112 ASTM D1533	150 ml amber P 125 ml amber P	None	None
In-Service Fluid	(Physical)	Flash Point Fire Point	Collect one sample per transformer (Minimum of four samples) End-user's results to be used	Transformer Fluid Sample Port	ASTM D92 ASTM D92	250 ml amber P	None	None
	(Electrical)	Conductivity Dissipation Factor			ASTM D4308 ASTM D924			
	(Chemical)	Water Content		ASTM D1533				
	Dielectric Fluid Chemical Composition	Methyl Esters		AOAC Official Method 963.22	60 ml amber P	None	None	
		Oils and Fats		AOAC Official Method 981.11	60 ml amber P			
		Total Fatty Acids		AOAC Official Method 972.28	60 ml amber P			

Sample	Type	Parameter(s)	Frequency	Location	Method(s)	Container Type	Preservative(s)	Holding Time
		Phenolic Antioxidants			AOAC Official Method 983.15	60 ml amber P		
		Polymers and Oxidation Products			AOAC Official Method 977.17	60 ml amber P		
		Semivolatile organics (screening)			EPA Method 8270/3580A	250 ml amber G	< 4EC	7 days to extract; 40 days to analyze after extraction
		Trace metals (As, Ba, Be, Cd, Co, Cr, Cu, Mo, Ni, Pb, Se, Tl, V, Zn)			EPA Method 6010/3031	100 ml amber P	< 4EC	6 months
Field Blank	Dielectric Fluid Chemical Composition	Semivolatile organics (screening)	Collect and analyze one per sampling event	Field	EPA Method 8270/3520	1.0 L amber G	< 4EC	7 days to extract; 40 days to analyze after extraction
Equipment Blank	Dielectric Fluid Chemical Composition	Semivolatile organics (screening)	Collect one per lot sampled for virgin fluid and one per transformer sampled	Field	EPA Method 8270/3520	1.0 L amber G	< 4EC	7 days to extract; 40 days to analyze after extraction
		Trace metals (As, Ba, Be, Cd, Co, Cr, Cu, Mo, Ni, Pb, Se, Tl, V, Zn)	Analyze one sample for both in-service and virgin samples (Total of 2 samples per analysis)		EPA Method 6010/5030	1.0 L amber P	< 4EC	6 months
Matrix Spike	Dielectric Fluid Chemical Composition	Semivolatile organics (screening)	Collect and analyze one sample for virgin fluid and in-service fluid (Total of 2 samples per analysis)	Laboratory	EPA Method 8270/3580A	250 ml amber G	< 4EC	7 days to extract; 40 days to analyze after extraction
		Trace metals (As, Ba, Be, Cd, Co, Cr, Cu, Mo, Ni, Pb, Se, Tl, V, Zn)			EPA Method 6010/3031	100 ml amber P	< 4EC	6 months

Sample	Type	Parameter(s)	Frequency	Location	Method(s)	Container Type	Preservative(s)	Holding Time
<p>G - Glass LOEC - Lowest observed effect concentration NOEC - No observed effect concentration P - High Density Polyethylene *The 2.3 L sample size will cover the analysis marked by an asterisks. All other analysis will require the collection of an additional sample for the amount listed for that analysis. Footnotes: 1. Acute toxicity (fish bioassay) analyses will follow "Method for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms", EPA/600/4-90/027F, August 1993. 2. Per specification, all oil samples are to be collected per ASTM 923. 3. Glass sample containers should be sealed with either aluminum foil or some similar material to seal the sample properly and preclude the entrance of moisture or interaction between the oil and any gasketing material.</p>								

5. INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE REQUIREMENTS

Performance testing equipment and analytical laboratory equipment shall conform to specifications outlined in the individual laboratory's standard operating procedures (SOPs), the individual test methods and ASTM Method D117 (Appendix A).

6. INSTRUMENT CALIBRATION AND FREQUENCY

For all testing performed as part of this workplan, the testing facility shall adhere to the instrumentation requirements specified in the individual laboratory's SOPs and the individual test methods (Appendix A).

7. DATA MANAGEMENT

All records used in sample collection, transportation, chain-of-custody, testing and sample control, and final reporting will be filed with DTSC's Project Manager (see Appendix B for sample and data control). Data and reports submitted directly to DTSC's Project Manager shall be concurrently submitted to ABB. Data packages submitted by the laboratories will contain completed chain-of-custody forms, a narrative describing the sample condition and sample storage conditions, analytical results, and QC results including corrective actions and unusual testing conditions if any.

Reports generated by DTSC's Project Manager will be maintained in the project files in both hard and electronic format. Electronic reports will be copied to diskette for back-up and filed with DTSC's Assignment Manager.

All reports and data generated from this project will be centrally filed with OPPTD for a minimum of 3 years following Verification/Certification. At this time, DTSC's Assignment Manager will have the authority to transport the report and supporting data to State Archives.

D. ASSESSMENT / OVERSIGHT

During implementation of the workplan, DTSC's Project Manager will provide a weekly update to DTSC's Assignment Manager. The U.S. EPA Project Manager and ABB will be updated biweekly on the project's status by e-mail. DTSC's Project Manager will frequently interface with the Project Team's QA/QC member on data collection procedures, data quality, and data analysis.

Following each major task, DTSC's Project Manager will prepare a "Summary of Findings". A copy of the Summary will be forwarded to each Project Team Member, Assignment Manager, and U.S. EPA Project Manager and Quality Assurance (QA) Officer for review and comments. Following completion of the Workplan, the "Summary of Findings" will be combined into a Draft Evaluation Report. DTSC's Project Manager will provide a copy of the Draft Evaluation Report to the Project Team members, U.S. EPA Project Manager and QA Officer, and ABB. DTSC's Project Manager will then conduct a project review meeting to discuss the final results of the project and draft a Verification/Certification decision.

E. DATA VALIDATION AND USABILITY

1. DATA REVIEW, VALIDATION, AND VERIFICATION REQUIREMENTS

The sampling design is outlined in Section C1 and C2. No deviations from the sampling design are anticipated. However, if a deviation does occur, then the conditions and reasons for the deviation will be recorded in the field log. A copy of the field log will be provided to each project team reviewer and ABB at the end of sampling. If any of the following conditions occur, then the testing results may be deemed void:

- fluid samples were collected or prepared in a non-independent manner;
- laboratory QA/QC procedures were not followed;

- incorrect or improperly labeled samples;
- samples which may have been tampered with;
- samples which do not meet holding or preservation requirements

2. VALIDATION AND VERIFICATION METHODS

The project team will verify and validate the procedures and data generated by the testing facility (Appendix B discusses some key indicators that DTSC considers when determining validity of data), or by the independent certified analytical laboratory.

3. RECONCILIATION WITH DATA QUALITY OBJECTIVES

If inadequacies in the data are noted at the time the Draft Evaluation Report is distributed, the Draft Evaluation Report will note these inadequacies and offer: 1) recommendations for additional tests; or 2) suggested language reducing the scope of the Verification/Certification.

REFERENCES

1. *EPA Guidance for Quality Assurance Project Plans*, EPA QA/G-5, EPA/600/R-98/018, U.S. EPA, Office of Research and Development, Wash. D.C. 20460, February 1998.
2. *Specifications and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs*, ANSI/ASQC E4-1994, American Society for Quality Control, 611 E. Wisconsin Ave., Milwaukee, Wisconsin 53202.
3. *Oil Decomposition Tests, Project 9285-44*, Powertech Labs, March 1997.
4. *Part I Application, California Environmental Protection Agency, Hazardous Waste Environmental Technology Certification Program for BIOTEMP[®] Electrical Insulating Fluid*, ABB Power T&D Company Inc., Dielectric Fluids Division, September 23, 1999.
5. *Toxicity Evaluation of BIOTEMP[®] to Oncorhynchus mykiss*, prepared for ABB Power T&D Company, Inc., Parametrix, Inc., December 1998.

Appendix A

Performance Test Methods and Laboratory Standard Operating Procedures (SOPs)

Appendix B

DTSC HML Data Quality Control Practices

Appendix C

Selected Performance Values for Exxon Univolt 61 or N61