FINAL

Technology Evaluation Workplan (Cooper)

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A. PROJECT MANAGEMENT

1. TITLE AND APPROVAL SHEET

Department of Toxic Substances Control

Mr. Tony Luan, P.E.
ETV Pilot Program Manager

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Mr. John Wesnousky, P.E.
Technical Review Panel

____

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)
Ms. Norma Lewis, US EPA (Technical Project Manager)
Ms. Lauren Drees, US EPA (QA/QC Officer)
Mr. John Luksich, Cooper Power Systems
Dr. Scott Johnson, CH2MHill

3. PROJECT/TASK ORGANIZATION (see Figure 1)

Department of Toxic Substances Control

ETV Pilot Program 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.

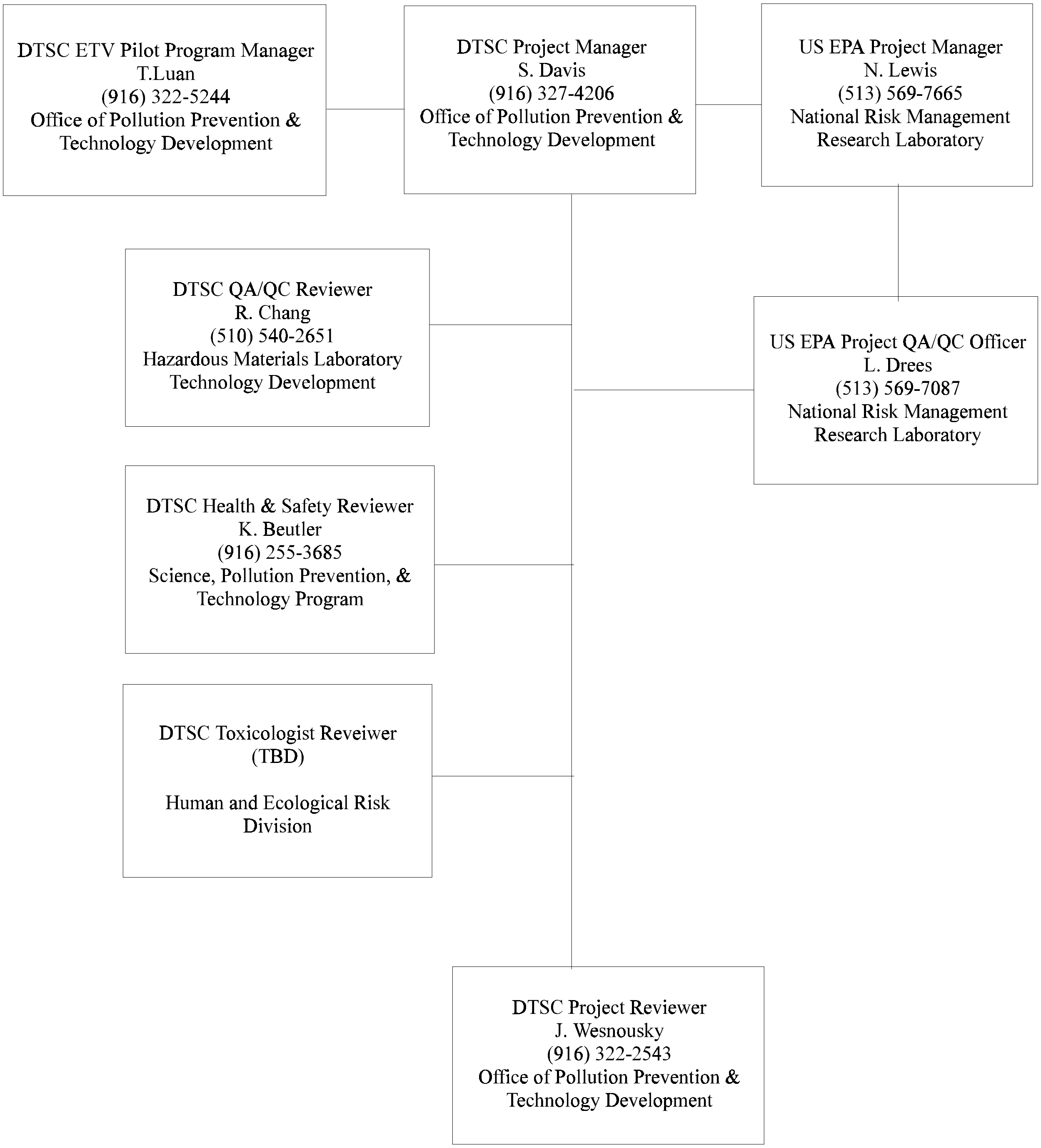


Figure 1. Project Organization Chart

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. The medium can be a solid, or liquids such as mineral oil, high molecular weight hydrocarbons (HMWHs), or synthetic oils such as silicone. Most transformers currently use some type of mineral oil as the cooling fluid; however HMWHs 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 generally do not meet the requirements for regulation as hazardous waste under RCRA; however, mineral oils that have been in service for approximately 10 years may 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
Source of Waste: Primarily damage to transformers		
Waste Oil from Electrical Transformers	126	100,000
Source of Waste: Draining of oil prior to reconditioning or decommissioning transformers		
Wastes Containing PCB	28	50,000
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 accept mineral oil or other free liquids for disposal, or allow disposal of 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, biodegrade quickly in the advent of a spill, and produced from a renewable resource. 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. 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 independent, 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 page 9.

Cooper Power Systems (Cooper) is a corporation with North American electrical power equipment dielectric fluids development and sales offices located in Waukesha, Wisconsin. Cooper has developed and brought to market a vegetable seed oil-based dielectric fluid called Envirotemp® FR3™. Approximately 120 transformers currently use Envirotemp® FR3™. To verify the claims listed in Table 2 below, DTSC will conduct tests on virgin Envirotemp® FR3™ including select performance tests per ASTM Oil Specification 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 Envirotemp® FR3™ and mineral oil.

2. PROJECT OBJECTIVES

The project objectives are to verify the applicant's technology performance claims which are listed in Table 2 below. Other objectives not included in the Verification/Certification claims are: (1)assessment of worker health and safety aspects of Envirotemp® FR3™, (2) comparison of the chemical composition of virgin Envirotemp® FR3™ fluid to the applicant's formulation specifications, and (3) estimation of costs based on the expected life of Envirotemp® FR3™ as compared to those of mineral oil.

Table 2. Verification/Certification Claims for Envirotemp® FR3™

Number	Claim
1	General Performance: In the following composition ratio(s) (>98.5% vegetable oil, <1.5% additives), meets criteria for dielectric fluid performances characterized by ASTM D3487, *Standard Specification for Mineral Insulating Oil*, and ASTM D5222, *Standard Guide for High Fire Point Fluids of Petroleum Origin*, and others[3].
2	Aquatic Biodegradability: Envirotemp® FR3™ biodegrades 99% based on the average of several performance tests, as measured by OPPTS 835.3110, *Ready Biodegradability*[4].
3	Flammability: Has a Flash Point of at least 300°C, and Fire Point of 350°C, based on the average of several performance tests by independent labs performing ASTM D92 (Cleveland Open Cup)[3].
4	Acute Toxicity: 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 do not meet the toxicity characteristic criteria in Code of California Regulations, Title 22, Section 66261.24(a)(6).

Both the manufacturer and utilities, who are the largest likely customer group, have recommended that the verification effort focus on environmental performance measures, such as toxicity and biodegradation, because (1) large amounts of general laboratory performance data generated under industry standards are available, (2) DTSC's expertise is in the environmental performance areas, and (3) time and resources are limited. In addition, the cost impact of using transformers with vegetable oil-based dielectric fluids are of interest to users. The cost will be based on existing life expectancy data for Envirotemp® FR3™ provided by Cooper. Additional chemical and environmental composition data will also be obtained as part of this workplan.

3. PROJECT TASK DESCRIPTION

To validate Cooper'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 chemical composition.

Verification/Certification Claim #1 - General Performance

Performance Tests - Numerous ASTM and ANSI methods are available in the general areas of thermal and chemical stability; physical and electrical properties, and extended life analysis. The industry has developed specifications for dielectric fluid performance tests through the ANSI consensus-based standards development process. No specifications exist for vegetable oil-based dielectric fluids; however three ASTM specifications and two International Electrochemical Commission (IEC) specifications have been developed for other dielectric fluids: ASTM D3487 (mineral oils), ASTM D5222 (HMWHs), ASTM 4652 (Silicone Fluids), IEC 1099 (Virgin Synthetic Organic Esters) and IEC 1203 (In-Service Synthetic Organic Esters). The dielectric strength value listed in these five specifications are similar and are the basic property used to evaluate a dielectric fluid's performance. Viscosity and pour point values are used in designing new transformers while the neutralization number, dissipation factor, interfacial tension, and water content values are used to monitor the fluid's quality while in use. The viscosity, pour point, water content, flammability and fire point values differ between the five specifications due to the chemical nature of each dielectric fluid type. Typically, the less-flammable fluids are used when the transformer may be used in areas where fire safety is required, such as inside buildings. 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. Only the ASTM methods associated with ASTM specifications D3487 and D5222 will be performed. Envirotemp® FR3™ will not be tested for oxidation stability by ASTM Method D2440 since this fluid doesn't produce a carbon sludge when oxidized. Instead, ASTM Method D971 (interfacial tension), and ASTM Method 974 (neutralization number) will be tested for virgin and in-service Envirotemp® FR3™ fluid to monitor changes in fluid quality. 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, and dielectric breakdown (gap) by ASTM D1816 will not be conducted. Table 5 lists the performance tests to be conducted on both the virgin and in-service fluids. A typical mineral insulating oil, such as 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. This typical mineral insulating oil will be referred to as the reference mineral oil in this workplan.

Verification/Certification Claim #2 - Aquatic Biodegradability

Biodegradation - numerous lab test methods exist for aquatic biodegradation, less for soil. Some measure biodegradation by amount of organic carbon fully mineralized to CO_2, 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 -

OPPTS 835.3110 Ready Biodegradability,

Six analytical methods are listed in U.S. EPA OPPTS 835.3110 Ready Biodegradability. Based on the low solubility of oil in water, test methods which measure dissolved oxygen were determined inappropriate and not included in this discussion. Two methods listed in OPPTS 835.3110 measure oxygen consumption (Manometric Respirometry) or carbon dioxide evolution (CO_2 Evolution). A third possible method is a modified MITI (Japanese) method that also uses respirometry, looks at possible biodegradation compounds, and compares the O_2

consumption to a theoretical consumption (meaning the compound must already be known). However, neither solvents nor emulsifying agents can be employed under the MITI method; therefore, this method was not considered for use.

The CO_2 evolution method will be used as part of this Verification/Certification because it appears to be the least complicated of the available methods. The CO_2 evolution method estimates the degree of biodegradation based on the amount of CO_2 produced and expresses this results as a percentage of the theoretical CO_2 which can be produced. The theoretical CO_2 amount is calculated from the number of carbons for the test solution. In the past, Cooper has performed aquatic biodegradability tests using OPPTS 835.3100, Aerobic Aquatic Biodegradation. This method also estimates the amount of CO_2 produced by the test solution by calculating the difference in the CO_2 evolution produced in the test flask and the control flask. OPPTS 835.3110 has been chosen because it has a more stringent criteria for a valid test compared to OPPTS 835.3100.

Verification/Certification Claim #3 - Flammability

Flammability - to determine the flash point and fire point for virgin and in-service Envirotemp® FR3™ 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, partially address concerns about spills near receiving waters, and as part of waste classification. 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 criteria 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 Envirotemp® FR3™ 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).

- AOAC Official Method 981.11, Oils and Fats, Preparation of Sample (98);
- AOAC Official Method 972.28, Total Fatty Acids in Oils & Fats, Hexane Distillation (97);
- AOAC Official Method 963.22, Methyl Esters of Fatty Acids in Oils and Fats, Gas Chromatographic Method (98);
- AOAC Official Method 983.15, Phenolic Antioxidants in Oils, Fats, and Butter Oil, Liquid Chromatographic Method (94); and
- 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 HMWH Specifications (High Fire Point Oils)	D4652 Specifications (Silicone Oil)	IEC Specifications (Synthetic Oil)[2]		Average Envirotemp® FR3™ Performance Specifications[*]
				IEC 1099 (Virgin)	IEC 1203 (In-service)	
PHYSICAL TESTS						
Flash Point, min, EC, D 92 (Cleveland Open Cup)	$145	N/A	300	$250	N/A	330
Fire Point, min, EC, D 92 (Cleveland Open Cup)	N/A	304-310	340	>300	>300	359
Pour Point, max, EC, D 97	-40[b]	-24[b]	-50	-45[b]	-24[b]	-21[b]
Viscosity, max, cSt, D 445, 100EC	3[b]	11.5-14.5[b]	15-17	NA[b]	NA[b]	7.6[b]
40EC	12[b]	100-140[b]	35-39	35[b]	11.5-14.5[b]	32.7[b]
0EC	76[b]	1800-2200[b]	81-92	3,000[3,b]	100-140[3,b]	N/A
ELECTRICAL TESTS						
Dielectric Breakdown, D 877, kV, min	30[a]	42[a]	35	45[a]	>30[a]	50[a]
Dielectric Breakdown, D 1816, kV, 1.02 mm gap, min	28[a]	30[a]	--	N/A[a]	N/A[a]	67[a]
2.03 mm gap, min	56[a]	61[a]	--	N/A[a]	N/A[a]	67[a]
Dissipation Factor, max, D 924, 60 Hz and 25EC (or power factor)	# 0.05[c]	0.01[c]	0.01	# 2.5[4,c]	# 0.8[4,c]	0.05[c]
max, D 924, 60 Hz and 100EC	# 0.30[c]	0.30[c]	--	# 2.5[5,c]	N/A[5,c]	--

CHEMICAL TESTS						
Neutralization number, mg KOH/g, D974	# 0.03	# 0.01	0.01 (max)	# 0.03	# 2.0	0.02
Interfacial tension, 25EC, dyne/cm, D971	$40	45	--	N/A	N/A	29
Water, max, ppm, D 1533	# 35	25	50	# 200	# 400	9

[*]Average performance parameters for batches produced in 2001.
[2]These values are provided as reference. Envirotemp® FR3™ will be tested for select parameters associated with ASTM specifications D3487 and D5222.
[3]These values are associated with a viscosity at -20EC.
[4]These values are associated with IEC Method 247 performed at 50 Hz and at ambient temperature.
[5]These values are associated with IEC Method 247 performed at 50 Hz and 90 EC.
aBasic property to evaluate dielectric fluid performance.
[b]Design parameter for new transformer. This value will vary due to the chemical nature of the dielectric fluid type.
[c]Maintenance parameter used to monitor the fluid quality over time. This value will vary due to the chemical nature of the dielectric fluid type.

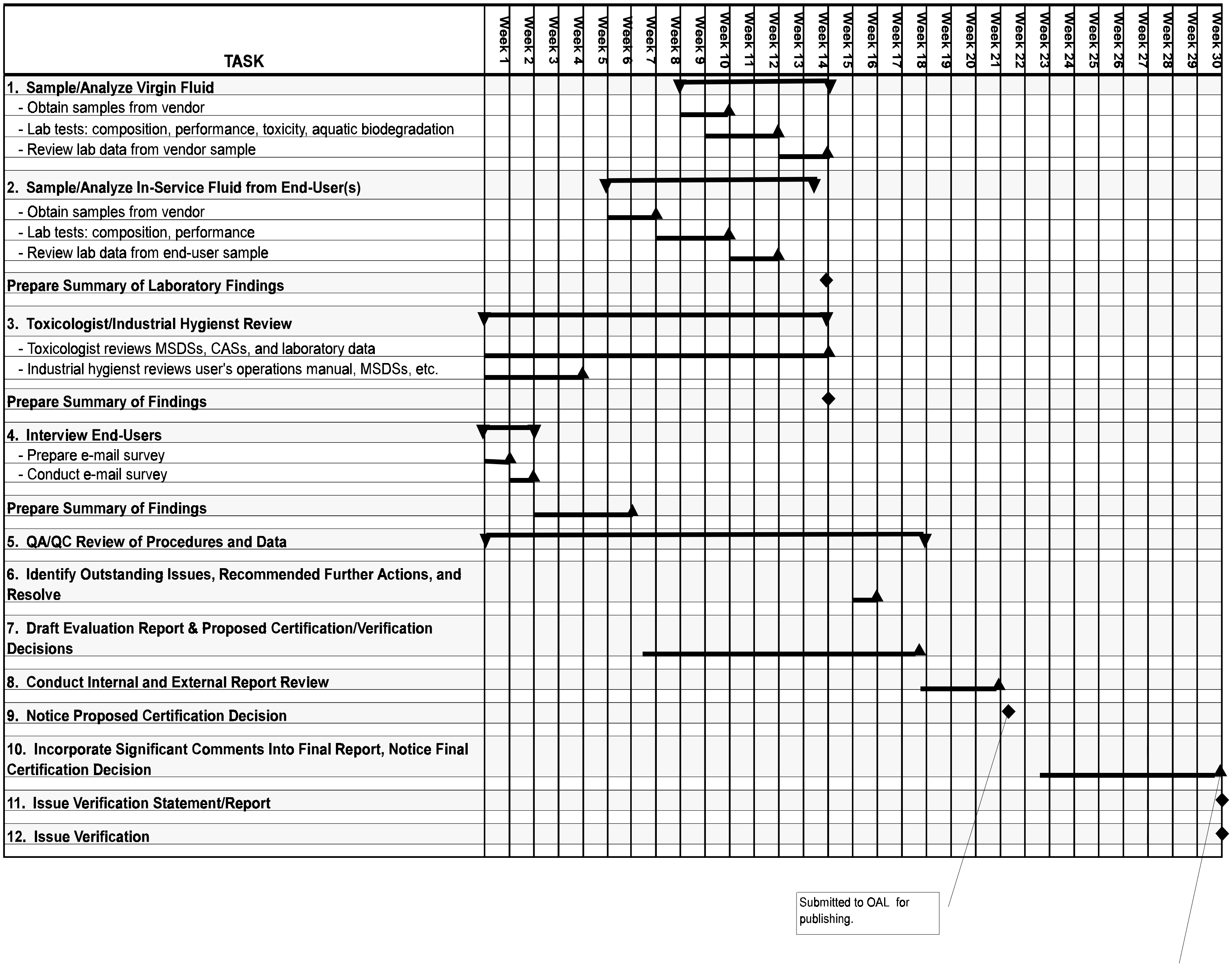
Toxicologist/Industrial Hygienst Review

Another activity is to evaluate the worker health and safety aspects of Envirotemp® FR3™. A DTSC Toxicologist will review of 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 38 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; aquatic biodegradation results for nine samples by EPA Method 835.3100 and six samples by CEC-L-33-A-93; acute toxicity results for two samples using screening and definitive testing by the Organization for Economic Cooperation and Development (OECD) GL 203 and Environment Canada Test Methods; results for two samples by EPA Method 8015 Modified, 8270, 8260, 6010, and wet chemistry analysis; and chemical composition analysis for nineteen samples of vegetable oil by AOCS Method Ce 2-66 and gel permeation chromatography. 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 health assessment report procedure (HARP) forms submitted by DTSC staff for site visits.

A proposed schedule outlining the activities required to evaluate Cooper's technology is shown in Figure 2. After Tasks 1 through 5 are completed, 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. COOPER 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 Envirotemp® FR3™ 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 increase in metal concentrations over time and to verify the chemical composition of the in-service fluid. The second purpose is to measure any 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, dissipation 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, experienced in performance tests of dielectric fluids used in power distribution transformers. Acute toxicity testing will be conducted by Associated Laboratories. The DTSC HML laboratory will perform the EPA Method 8270/3580A and 6010/3031 analysis. The aquatic biodegradation will be conducted by Global Tox International Consultants, Inc.. The AOAC and AOCS chemical composition analyses will be performed by Silliker.

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	Lance Lewand	ASTM Methods
Associated Laboratories	806 North Batavia Orange, CA 92688	Pam Schiro	Acute Toxicity (Fish Bioassay)

Global Tox International Consultants, Inc.	367 Woodlawn Road W Guelph, Ontario NIH 7K9 Canada		OPPTS 835.3110
DTSC HML	700 Heinz Street, Suite 150 Berkeley, CA 94170	Pam Schiro	EPA Method 8270/3580A and 6010/3031
Silliker	3688 Kinsman Blvd Madison, WI 53704		AOAC and AOCS Methods

3. SAMPLE CUSTODY REQUIREMENTS

DTSC and U.S. EPA have established procedures for maintaining the control and integrity of samples during collection, preservation, transportation, testing, storage, and disposal. DTSC's representative will label samples, maintain field notes, complete the chain of custody (COC) forms, and package and ship samples to the appropriate laboratories. DTSC's representative will sign and date each COC, and retain a copy in the project files. Upon receipt of the samples, the laboratory will sign and date the COC 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 QA/QC results.

Performance testing will be conducted on one sample of Exxon Univolt 61 or N61, the reference mineral oil. This reference oil will be analyzed by Thomas Edison Laboratories and Doble Engineering. Results from both laboratories will be compared per the precision sections outlined in each ASTM method to evaluate Doble Engineering's performance. Since Doble Engineering conducts performance testing for most transformer fluids on the market and generates the published performance specifications, a comparison of Doble's results to the published standards would not be an appropriate QC performance.

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 Envirotemp® FR3™ 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 criteria of the tests are not met, then the laboratory will notify DTSC's manager on the corrective actions that need to be taken.

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 that need to be taken.

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 that need to be taken.

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 that need to be taken. For AOAC Method 983.15, the method performance criteria is proved 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, results are acceptable if they meet the QA/QC requirements 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)	Neutralization number, mg KOH/g Interfacial tension Water Content*			ASTM D974 ASTM D971 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 Total Fatty Acids			AOAC Official Method 981.11 AOAC Official Method 972.28	60 ml amber P 60 ml amber P		
		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
Virgin Fluid (cont.)	Dielectric Fluid Chemical Composition (cont.)	Trace metals (As, Ba, Be, Cd, Co, Cr, Cu, Mo, Ni, Pb, Se, Tl, V, Zn)	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 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

Sample	Type	Parameter(s)	Frequency	Location	Method(s)	Container Type	Preservative(s)	Holding Time
	Aquatic Biodegradation		Collect two samples per lot (total of 6 samples) Analyze only three samples		OPPTS 835.3100	150 ml, amber G		
Reference Oil (Exxon Univolt 61 or N61)	(Physical)	Flash Point*	Collect and analyze one sample total	Dielectric fluid formulation facility	ASTM D92	2.5 L amber P*	None	None
		Fire Point*			ASTM D92			
		Pour Point*			ASTM D97			
		Viscosity (100, 40, and 0 degrees C)			ASTM D445	25 ml amber P		
	(Electrical)	Dielectric Breakdown*			ASTM D877			
		Dielectric Breakdown (gap)			ASTM D1816	450 ml amber P		
		Dielectric Breakdown (Impulse)			ASTM D3300	2.0 L amber P		
		Dissipation Factor (25 & 100)*			ASTM D924			
Reference Oil (Exxon Univolt 61 or N61)	(Chemical)	Neutralization number, mg KOH/g	Collect and analyze one sample total	Dielectric fluid formulation facility	ASTM D974	150 ml amber P		
		Interfacial tension			ASTM D971	125 ml amber P		
		Water Content*			ASTM D1533			
In-Service Fluid	(Physical)	Flash Point	Collect one sample per transformer (total of 4 samples) End-user's results to be used	Transformer Fluid Sample Port	ASTM D92	250 ml amber P	None	None
		Fire Point			ASTM D92			
	(Electrical)	Conductivity			ASTM D4308			
		Dissipation Factor			ASTM D924			
	(Chemical)	Neutralization number			ASTM 974			
		Interfacial tension			ASTM 971			
		Water Content			ASTM D1533			
	Dielectric Fluid Chemical Composition	Methyl Esters	Collect and analyze one per Transformer	Transformer Fluid Sampling Port	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		
		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

Sample	Type	Parameter(s)	Frequency	Location	Method(s)	Container Type	Preservative(s)	Holding Time
		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 Analyze one sample for both in-service and virgin samples (Total of 2 samples)	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)			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

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.

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. *Part I Application, California Environmental Protection Agency, Hazardous Waste Environmental Technology Certification Program for Envirotemp® FR3™ Transformer Fluid*, Cooper Power T&D Systems, no date.

4. *The Biodegradation of Envirotemp® FR3™, Univolt-60 and R-Temp Transformer Fluids*, Internal memo, 4/23/99.

5. *Analytical Report: Acute Trout Toxicity Testing for Two Envirotemp® FR3™ Formulations*, Final Report, Global Tox International Consultants, Inc., December 1, 1999.

Appendix A

Performance Test Methods and Laboratory Standard Operating Procedures (SOPs)

Appendix B

DTSC HML Data Quality Control Practices