from Joe Brooks

EPRI
Electric Power
Research Institute

6 July 1983

Dr. Carl McMillin
Monsanto Research Corp.
1515 Nicholas Road
Dayton, OH 45418

Dear Carl:

As per our telephone conversation, I am sending you a copy of EPRI's statement of work for analysis of dioxins and furans in PCB and PCB contaminated oil. I anticipate that Monsanto would address Tasks 1, 2, 3 and 8. However, if you feel that you can contribute to any of the other tasks, then you may wish to respond there too.

Your response should include completion of EPRI Form 112 for each task and another form for all tasks taken together. In addition, you should indicate whether you think Monsanto can accomplish the work as stated using state-of-the-art technology.

If you feel some tasks cannot be done, please indicate the rationale for your position. Do provide budget and timetables for those tasks that you feel are achievable.

Since EPRI is very anxious to begin this work, I would appreciate a rapid response. Feel free to call me if you have any questions (415) 855-2018.

Sincerely,

Jacques Guertin
Project Manager
Environmental Physics and Chemistry

JG:ms
Enclosures

cc: Gil Addis

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EPRI - RFP2028 PCDF
PROPOSED WORK STATEMENT

1. STATEMENT OF WORK

1.1 Background

Concerns about effects of polychlorinated biphenyls (PCB) on human health and the environment led to a ban on production and to regulation of use and disposal in the United States. PCB, often blended with tri-and tetrachlorobenzenes (T/TCB), were used as dielectric fluids in capacitors, transformers and other electrical equipment. Regulation of PCB continues to have a very direct effect on owners or operators of this equipment. Work at the Electric Power Research Institute (EPRI) is aimed at aiding electric utilities in complying with these regulations. EPRI also is investigating areas which may affect the future perception of the hazards of PCB or alter the requirements for compliance.

Many of the effects associated with PCB now appear to be attributable to either polychlorinated dibenzofurans (PCDF) and/or dibenzodioxins (PCDD). PCDF can be formed from PCB. PCDF and PCDD can be formed from T/TCB. It is possible then that PCDF and PCDD were introduced into electrical apparatus as trace contaminants in PCB during manufacture or installation or that they may be formed during normal or abnormal operation. However, there is little hard information on the occurrence if any of PCDF and PCDD in electrical equipment and no basis for assessing their effects.

The purposes of the program are to identify the levels of PCDF and PCDD in operating electrical equipment and to attempt to relate these levels to the conditions of service of the equipment. To accomplish these purposes, appropriate analytical techniques must be demonstrated and relevant dielectric liquid samples must be selected and analyzed. The results should have maximum reliability and credibility. The overall program thus includes several participants - the University of Umea with C. Rappe as consultant, the Battelle Memorial Institute with Kuhlman Corporation as sub-contractor and the New York State (NYS) Department of Health. Where desirable, equipment trials will be staged in order to provide particular PCB exposure scenarios which may not be readily available from equipment in service.

The final report of EPRI program RP1263-11 will be used as a literature base.

A. Work Tasks

1. Task 1: Preparation of Samples

The purpose of this task is to provide samples of insulating liquids spiked with C^{13} labeled PCDF and PCDD con-

congers. Carbon-13 containing congeners in measurable quantities are absent in normally occurring materials and are clearly distinguishable from normal isotopic materials by mass spectrometry. Spiking samples with labeled congeners provides a unique internal standard for accurate analysis.

Synthesis and spiking will be done by ?_____

One gram sized aliquots of the spiked liquids will be supplied to the round robin participants - Battelle Memorial Institute, NTS and the University of Umea (C. Rappe). Similar aliquots will be retained for use in this program by the spiker. This procedure minimizes the risk of loss of these costly and potentially hazardous materials. A representative of Battelle will participate in the spiking procedure and the associated record keeping.

The work in this task is divided into three parts:

a. Standard Materials

Six congeners of PCDF, prepared from C^{13} precursers, will be provided to spike approximately ninety (90) dielectric fluid samples of one gram size at approximately the 100 ppb level of each congener. The six congeners will be:

1.a.1	2,3,7,8-PCDF
1.a.2	1,2,3,7,8-PCDF
1.a.3	2,3,4,7,8-PCDF
1.a.4[1]	1,2,3,4,7,8-PCDF
1.a.5[1]	1,2,3,7,8,9-PCDF
1.a.6[1]	1,2,3,6,7,8-PCDF

Does the use of any of these pose any separation problems with present state-of-the-art techniques?

b. Baseline Samples

Samples of five liquids will be spiked with measured amounts (100 ppb) of each of the six PCDF and one PCDD congeners listed above (1.a.1-1.a.7). The five liquids will be:

1.b.1	Aroclor 1016
1.b.2	Aroclor 1242

[1] 1.a.4, 1.a.5, and 1.a.6 are prepared as a mixture in the ratio of 1:1.3:0.5.

1.b.3	Aroclor 1260
1.b.4	Tri/tetrachlorobensene mixture (T/TCB)
1.b.5	Aged mineral oil containing 500 ppm PCB and 500 ppm T/TCB

The first four samples will approximate as closely as possible unused liquids formerly employed in the manufacture of electrical apparatus. The oil used in sample 1.b.5 will be obtained from an aged transformer and will be spiked with Aroclor 1254 and T/TCB approximating the condition of unused askarel.

c. In-Service Samples

Samples of up to twenty liquids supplied by Battelle from electrical apparatus in service (see Task 3 for a discussion of the sample selection and acquisition process) will be spiked in the same way as samples 1.b.1-1.b.5. Spiking level will be determined after completion of Tasks 3a and 3b. The spiking is best done for all samples in one, or at most, two batches. This will maximize reproducibility and reliability and minimize demands on an outside expert. (Aliquots of only ten samples will be provided to the University of Umea.)

2. Task 2: Selection of Samples from In-Service Equipment

a. Selection of Generic Candidates

The range of applications of equipment filled with askarel or mineral oil containing PCB will be surveyed by ?____ and their typical and extreme conditions of service will be delineated. From this, approximately twenty (20) generic equipment candidates for sampling and analysis of liquid will be identified. Emphasis in the identification process will be on equipment operating under extreme conditions. However, a broad enough range of samples should be planned to determine conditions under which no PCDF will be found. A chart of pertinent history to be obtained on each sample will be developed together with a listing of desirable added optional information to be reported as available. The sample selection process and the format of the history chart will be confirmed by the EPRI project manager. A milestone report on this work will be written.

b. Acquisition of Samples

Battelle with the help of the EPRI project manager, if needed, will arrange to acquire liquid samples representative of the candidate types identified above in

order to supply the samples to ?__-CRD for spiking - Task 1c.

3. Task 3: Gas Chromatography/Mass Spectroscopy Analysis (GC/MS)

The purpose of this task is to analyze dielectric fluids containing PCB for PCDF and PCDD congeners, specifically those listed in Task 1. ?__ Battelle, and NYS [and, under subcontract, the University of Umea (C. Rappe)], will use the best separation and GC/MS techniques at their disposal and will implement any improvements in methodology appearing during this work.

The work in this task is divided into four sections:

a. GC/MS Cross Check

?___ the University of Umea, Battelle, and NYS will analyze the five samples provided under Task 1b (.1-.5) for PCDF and PCDD. At a minimum, the samples will be analyzed for the six 2,3,7,8,X-PCDF congeners listed in Task 1a and for 2,3,7,8-PCDD. The initial goal for the lower limit of detection will be 1 ppb. Actual detection limits for each of the significant compounds will be estimated. Control samples will be evaluated as necessary. (If necessary, replicate analyses will be performed on selected samples as requested by EPRI - at an additional cost of $2000 per sample.) Other compounds, particularly PCDF and PCDD congeners will be determined when feasible. Any significant unknown or indeterminate peaks found in the gas chromatogram or mass spectrogram will be noted.

b. Assessment of Methodology

?___ (and, via subcontract, C. Rappe), Battelle, and NYS will participate in the preparation of a milestone report summarizing the results of the analytical cross-check, the sources of any initial differences, the resolution of these diffences and any remaining unresolved problems with suggestions for future resolution. The report will be prepared in order to provide a common working basis for the later round robin (Task 3c) and to provide recommendations to EPRI to either continue the program as planned initially or to divert funds for additional verification of analytical methodology. Contaminated aged transformer oil will receive particular attention here because its chemical complexity may not allow analysis of congeners at the ppb or even tens of ppb level. Such a finding could affect the sample selection process (Task 2). This report is not intended for publication

and would not be distributed beyond EPRI and its reviewers and the participants in this task.

c. Round Robin Analysis

?___ Battelle, and NYS will analyze a minimum of twenty (20) samples (University of Umea 10 samples) from Task 1c for the six 2,3,7,8,X-PCDF congeners listed in Task 1a and for 2,3,7,8,-PCDD and, by standard methods, for PCBs. Other compounds will be determined when agreed upon between each contractor and EPRI. Any additional costs for additional analysis will be agreed on before hand. Detection limits sought will be specified based on results in Tasks 3a and 3b.

4. Task 4: Equipment Trials

The purpose of this task is to investigate the conditions under which PCDDs and PCDFs might be formed in utility operations. Battelle will have primary responsibility for supervising this task. Their subcontractor (Kuhlman) will run at least ten trials using 10 kVa single-phase transformers operating under a variety of conditions. Contaminated mineral oil or askarels from the test equipment will be sampled and analysed by the GC/MS technique validated in Task 3 to evaluate factors which can lead to PCDF and PCDD formation. Experimental variables will include such factors as arcing, corona, high temperature aging, and variations in askarel and contaminated oil composition. ?___ will provide consultation on this task.

5. Task 5: Pyrolysis of Aroclors 1016, 1242, 1254, and T/TCB

Three Aroclors will be pyrolysed by NYS using literature-based procedures to explore their relative tendencies to form PCDFs and related materials. It has been suggested that 1016 and 1242 may form much lower amounts of biologically active isomers than the Aroclors which have already been studied. This hypothesis will be tested using analytical procedures developed in Task 3. ?_ will provide consultation on this task.

6. Task 6: Combustion of Aroclors 1016, 1242, 1254, and T/TCB

Three Aroclors will be burned in CL&R's (NYS) model incinerator to explore their relative propensity to form toxic PCDFs. These experiments may shed light on the hazard posed in the combustion of Aroclors 1016 and 1242. ?__ will provide consultation on this task.

7. Task 7: Development and Validation of a Rapid MS/MS Based Screening Technique

NYS will develop a chemically based screening technique to provide a rapid, economical complement to traditional rigorous analytical procedures. The availability of the samples and extracts discussed in Tasks 3.a, 3.c, 5 and 6 provide an unusual opportunity to validate a MS/MS-based screening procedure rapidly and economically. It may be desirable to involve an independent consultant agreeable to both NYS and EPRI for technical guidance.

8. Task 8: Reports

a. Preparation of Monthly Reports

Informal monthly letter reports summarizing technical progress will be prepared during the course of this project.

b. Preparation of Milestone Reports

Milestone reports will be prepared on completion of:

bi. Task 2a ?_____
bii. Task 2b (Battelle)
biii. Task 3b (All)

c. Preparation of Final Reports

Final reports including all work reported in previously mentioned milestone reports will be prepared in publishable form by each of the contractors.

d. Summary Report

"PCDF and PCDD in Electrical Equipment" - Each contractor will participate in preparation of a report summarizing the PCDF and PCDD content found in the equipment sampled. The report would include any conclusions possible about the sources of any PCDF and PCDD found. It would also include any extrapolations possible about the PCDF and PCDD content of electrical equipment in general.

This report as well as the milestone report on Task 3 may be best coordinated by an independent consultant who is not a participant in the analysis of samples. Each contractor will contribute

his expertise in the chemical analysis of dielectric fluids, in the chemistry of PCB, PCDF and PCDD and in the behavior of electrical equipment.