RETROFILL REDUCES LIABILITIES RESULTING FROM A NETWORK TRANSFORMER FAILURE

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ABSTRACT

Knoxville Utilities Board's active PCB removal program has indigenous side effects that have resulted in saving costs that otherwise may have been catastrophic.

1. Transformers that had, heretofore, un-noticed latent problems such as broken bushings, failing contacts, reduced BIL, etc. were discovered and repaired.

2. The initial removal of the Askarel, Inerteen, or other highly PCB concentrated fluids immediately lowered the liability of a PCB fire or spill.

3. A transformer evaluation program allowed the complete removal of certain underloaded transformers.

With the retrofill program in full swing, a transformer failure in the high-voltage switch compartment resulted in a PCB spill of 21 gallons of Unison's TF-X fluid. There was a resultant brief explosion and expulsion of a smoke cloud. The now, 23,000 ppm fluid, produced no measurable PCB contamination external of the underground transformer vault from the smoke cloud. Clean up was restricted to only the liquid itself that escaped the switch compartment.

MONS 020234

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INTRODUCTION

There was a time when the Power Operations Supervisor could rest reasonably comfortably at night knowing that all was being done that is technically possible to assure the power system was safe, was operating in a safe manner, and was not virulent or unfavorably impacting the environment. Along with the Technological Age has come the burden of assuring that what man creates must not uncreate man. The dismal stories of PCB transformer failures has done little to appease the electric utilities' conscience and state of well being.

Hiroshima was the United States nemesis on Japan, however, Japan's Yusho rice oil incident on Kyushu Island has probably returned the favor. Since that incident the United States has become engulfed in Federal Regulations and controls that have reached colossal proportions involving a molecule termed PCB for short. From that infamous occurrence in 1968 till the present, Federal Regulation piled on Federal Regulation to "assure" the electric utility industry clean up it's act.

Employee apprehension, public opinion, the news media, and actually just being a good neighbor added to the pressure of dealing with PCB's. Some individuals react with horror if threatened with the presence of this material. And, undoubtedly the news media has made some very good mileage out of making the worst out of, many times, very marginal situations. In any case the pressure still persists and most utilities are reacting.

Scare stories - which became actualities with several transformer failures and fires around the country pre-empted any risk management studies and provided all necessary motivation to stamp out the PCB. If the electric utility was not convinced by July of 1985, the Federal Government issued the Final Rule of FR # 40 CFR PART 761. This Rule, for all practical purposes, eliminated PCB Network transformers from existence.

For those utilities who had their head in the sand, it was then time to rise up and do something about the PCB dilemma. RETRO-FILL, REPLACE, or RETIRE?

SYSTEM DESCRIPTION AND DETERMINATIONS

Knoxville Utilities Board (KUB) has actively pursued the removal of PCB's since the mid 1970's. It became our goal to remove all PCB's by a combination of replacement and/or reclassification which ever proved to be most economical.

KUB's downtown network consisted of 112 transformers in service with a storeroom stock of 7 units. The units ranged in sizes from 167 kva to 2500 kva and were either on a 208 volt continuous network system or on 480 volt spot network systems. All these units were of Inerteen, Pyranol, or Chlorextol Insulating fluids. None of these askarels were less than 348,000 ppm PCB ranging up to 850,000 ppm with the low range unit being a 1966 500 kva G. E. transformer and the high range unit being a 1970 500 kva W. E. transformer. The oldest network unit was manufactured in 1939.

Each unit was evaluated individually. Basically, units older than 25 years would be replaced and units less than 25 years old would be retro-filled and reclassified. (See Appendix A) However, as late as March, 1985 we still had not discovered a company which would guarantee a reclassification process that would leave a unit with less than 50 ppm.

Unison Inc. announced in the summer of 1985 that they had successfully reclassified PCB transformers belonging to Union Carbide. By November of that year we had contracted with Unison to reclass seven of our newest transformers which were connected in two 480v spot networks serving critical loads: A high rise bank building and the City-County building, both buildings very expensive.

This decision was prefaced by two other major considerations:

1. What degree of liability is reduced with retro-fill? (Appendix B)[1]
2. What impact does transformer failure rate affect the decision? (Appendix C)[2]

During the fall of 1985 we continued to evaluate for the best methods of PCB replacement. Studies were made on total replacement vs. total retro-fill. The final outcome was a combination approach. (See Appendix A) However, with competitive bidding on the retro-fill processes, the much lower than expected costs, encouraged us to include more units into the retro-fill program and less in the replacement. This decision paid dividends in several ways: 1) Much quicker approach to eliminating the liability of PCB's. 2) The retro-fill process required us to replace gasketing materials and as a result we discovered transformer problems that spelled transformer failure sometime in the not-to-distant future. 3) A transformer failure that did occur after the first retro-fill was not nearly traumatic as it could have been. This is the crux of this presentation.

MONS 020236

TRANSFORMER FAILURE

Before you ever have a PCB transformer failure is the time to make all the necessary precautions and plans on how to deal with the failure. Among the items to be considered are:

1. Are all EPA Requirements satisfied?[3]

2. Is your emergency response plan adequate?

3. Are your personnel trained?

4. Is there a procedural understanding with the local law enforcement, the Fire Department, the News Media, the citizens, and all the regulating bodies?[4]

5. Do you immediately know who to call to: report the problem, aide in containment, test, and clean up?[5]

If you can answer "Yes!" to these questions without hesitation, then you are somewhat ready for that call saying you have had a transformer explosion.

On February 19, 1987 at 9:52 A.M. KUB received a call from the Fire Department that there was an explosion in the underground vault adjacent to their Fire Hall and Control Center in downtown Knoxville.

The first crew responded to the scene of the explosion within 2 minutes after the initial call. This crew was in the process of getting the next transformer ready for retrofill and happened to be in the vicinity of the failure. Since the explosion was adjacent to the Fire Department they were the ones who called us to put out the fire! Fortunately the protective relaying responded quicker than either of us and cleared the circuit upon the initial explosion, so, only one cloud of smoke was all that was involved as far as the fire was concerned. The Fire Department would have reacted to contain the fire if fire and smoke had continued.

The first crew on the scene did make a quick assay of the vault and noted the resulting spill appeared to be contained. This crew was not prepared to take any further action except to secure the area. The spill control team arrived moments later. During the next few minutes many things began to happen:

-Communication to the Fire Department and the Police Department that we would be securing the area involved. This included blocking the street, sidewalk, grassy side area, and a portion of the Fire Hall.

-All vehicles in this area were impounded and the complete area roped off.
-Our Risk Management Group was called in to handle public contact, such as dealing with the owners of the vehicles.
-The Spill Control Team began thair work.
-EPA Hotline was notified.
-Test and Sampling Laboratory was summoned.
-A news releasa was prepared and issued.
-Clean-up crews were being outfitted.

The spill control team upon arriving suited up fully including the wearing of protective clothing and breathing equipment which supplied air. Their initial work consisted of putting down vinyl and cardboard sheets and applying absorbants to pravent any further spread of spill and to capture the spilled liquid. Normal precautions were taken at the work site to eliminate environment contamination by the worker's operations.

Intarnational Technology Corp. was summoned to maka PCB tests on all the parked vehicles, on specific locations on the sidewalk, grounds, and the Fire Department Building. These tests were done on an emergency basis with the results back in hand before nightfall. The results of these tests resolved some of our anxieties as all teats exterior to the vault showed very low and acceptable PCB levels with a maximum of <1.0 ugram / 100 sq. cm.

CONCLUSION

The fluid in the vault, however, tested 23,000 ppm which was down from the initial contamination before retrofill of 760,000 ppm. In extrapolating the test results, according to research done by Unison in co-operation with Battelle Columbus Laboratory and a resulting paper presented in a 1985 Doble Confarence "Simulated Building Fire Study Using Retrofilled Transformer Dielectric Coolant" by W. H. Martin, the PCB products (contaminants) that were produced were 1/35 to what they would have been had the original askarel been in the transformer. Had this been the case it is highly likely there would have been contamination external the vault. The result would have baen major clean-up of the street, the sidewalk, grounds, and building. To what proportion would be somewhat difficult to ascertain. But, it is evident it would have been a far more extensive procedure than the internal clean-up of the vault, which, in it's self was costly. The Laboratory fees, alone, exceeded $20,000. Clean-up costs were approximately $30,000.

Considering these events and the transformer failure, it is my recommendation that if one plans on having an Askeral transformer failure, it is best to have it with reduced PCB's.

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1. "Simulated Building Fire Study Using Retrofilled Transformer Dielectric Coolant" 1985 Doble Engineering Conference presented by W. H. Martin.

2. "Transformer Life Expectancy" by Dr. Steven C. Vick, Union Carbide Corporation, Tarrytown, New York 10591

3. "Polychlorinated Biphenyls (PCBs) Manufacturing, Processing, Distribution in Commerce, and Use Prohibitions" Federal Register No. 40 CFR PART 761.

4. "Guidance Document for TITLE III of the Superfund Amendment and Reauthorization Act of 1986" produced by the Tennessee Emergency Management Agency.

5. PCB Equipment, Operations & Management Reference Manual, Washington, D. C.: NRECA Research Project 82-7, March 1983. Prepared by: SCS Engineers, Inc.

UNIT EVALUATION: REPLACE vs. RECLASSIFICATION

Askarel Network Transformers: Unit ages from 1939 - 1975

208v Units	480v Units
3 - 167 kva	10 - 500 kva
36 - 500 kva	6 - 750 kva
2 - 600 kva	10 - 1000 kva
30 - 750 kva	11 - 1500 kva
2 - 1000 kva	9 - 2500 kva

1984 ESTIMATES FOR REPLACEMENT:

$30/kva for replacement Included:
- Purchase Transformer
- Cost to Remove Old & Install New
- Cost to Incinerate PCBs
- Cost to Landfill
- Cost to Transport

1985 ACTUAL QUOTES WERE Approx. $50/kva for REPLACEMENT.
or (102,701 kva)($50) = $5,135,000.

1984 ESTIMATES FOR RECLASSIFICATION:

$70/gal for Retrofill Included:
- Retrofill Process (Includes Destruction of PCBs & Transportation Costs)
- Cost of Support Personnel
- Cost of Final Fluid (May be Oil, Silicone, etc)

1985 ACTUAL QUOTES WERE Approx. $58/gal (Askarel fluid)
or (37,981 gal)($58) = $2,202,900

FINAL DETERMINATIONS:

- REPLACE 17- 208V UNITS W/ 16 NEW (Resized) 208v UNITS.
- RETROFILL 102- 208V & 480V UNITS.

(APPENDIX A)

MONS 020240

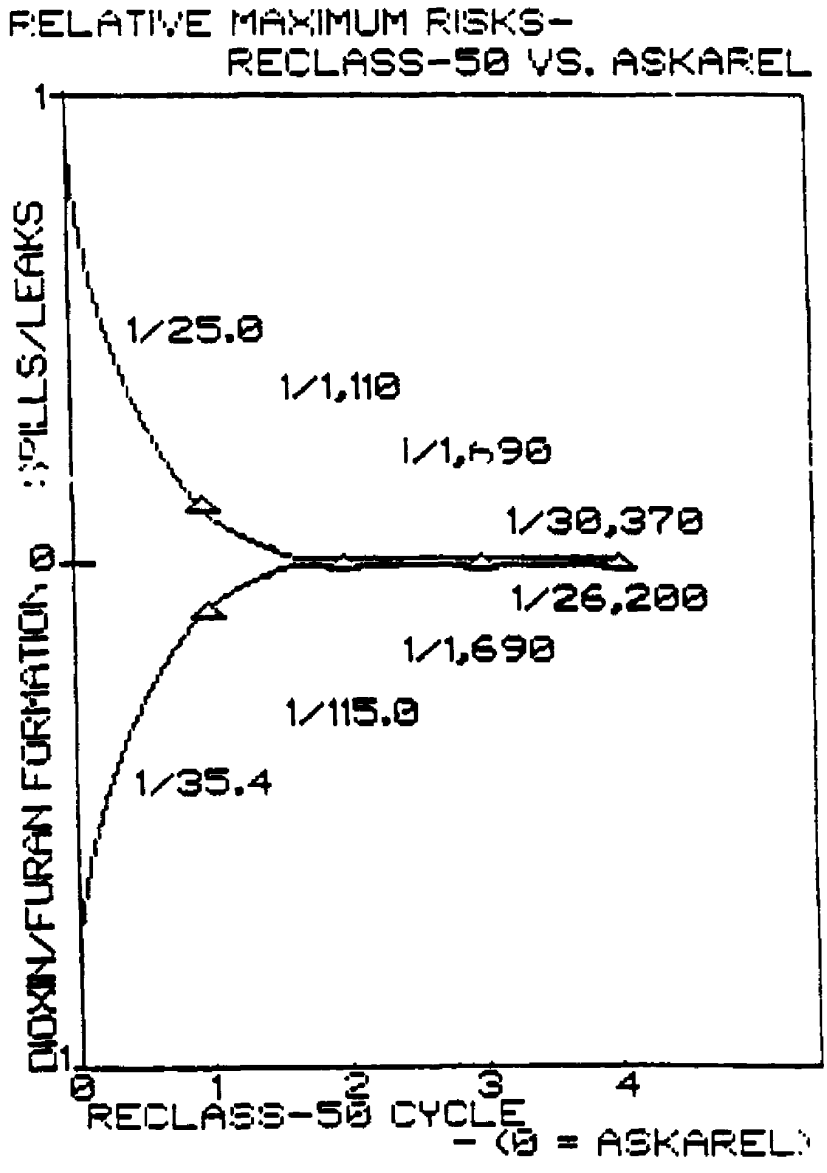
RELATIVE MAXIMUM RISKS-
RECLASS-50 VS. ASKAREL
1
SPILLS/LEAKS
0
DIOXIN/FURAN FORMATION
1
1/25.0
1/1,110
1/1,690
1/30,370
1/26,200
1/1,690
1/115.0
1/35.4
0
1
2
3
4
RECLASS-50 CYCLE
(0 = ASKAREL)

Table 1 shows the comparison between the IEEE 1973-1974 and the 1979 survey results for the total failure rate for all liquid filled power transformers.

TABLE 1

SURVEY YEAR	SAMPLE SIZE (unit-year)	NO. OF FAILURES	FAILURE RATE (failure/unit-year)
1973-74	15210	63	0.0041
1979	17996	111	0.0062

In this table, the failure rate was obtained by dividing the number of failures by the number of units times their service life. It is clear from this data that the failure rate increased by 50% in just six years.

TABLE 2

UNIT AGE	FAILURE RATE (failure/unit-year)
1-10	0.0072
11-25	0.0053
>25	0.0060

Table 2 confirms that the new transformers do indeed suffer from high "infant mortality", middle aged transformers display greatest reliability and only a slight indication of decreased reliability is seen with the oldest age group. This is a phenomenon which has been called the "Bathtub curve" (figure 1)[4]

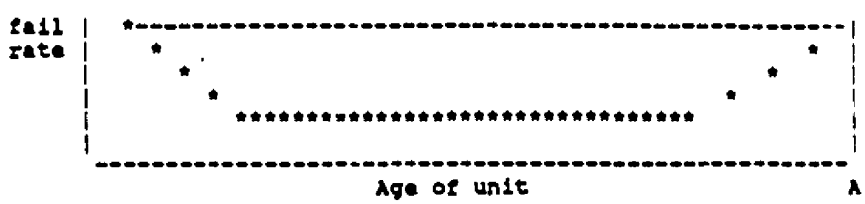

Figure 1

Figure one shows graphically that the decision point for scheduled replacement of transformers to minimize forced outages is point A - the point where the failure rate for an existing in-place transformer equals the failure rate of a new replacement unit. Replacement prior to this point will actually result in a higher overall failure rate for any given system. Since the failure rate for any given transformer is unknown, statistical data must be relied upon to determine the exact shape of the "Bathtub" curve.

(APPENDIX C)[2]

[redacted]

[redacted]

[redacted]

Clor-N-Oil Test Kit as a Risk Management Tool - An Update

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ABSTRACT

The Clor-N-Oil™ PCB Screening Kit can be considered an effective risk management tool only if the probability of a false negative is sufficiently small. Utah Power & Light Company has used the Clor-N-Oil™ Test Kit on over 123,750 transformers and has classified them as negatives or positives. A random sample of the Clor-N-Oil™ negatives (those testing less than 50 ppm PCB) were tested by gas chromatograph (GC) methods to evaluate the probability of a false negative. Approximately 2% of those testing negative with the Clor-N-Oil™ kit tested 50 ppm or greater by GC methods. Therefore, use of the Clor-N-Oil™ kit as a screening tool to reduce the risk or probability of a PCB spill is effective.

RESULTS

Utah Power & Light has Clor-N-Oil™ tested approximately 123,750 transformers, with 13% testing positive (over 50 ppm PCBs) and 87% testing negative (under 50 ppm PCBs). In order for the kit to be an effective risk management tool, the rate of false negatives (Clor-N-Oil™ tested under 50 ppm PCBs while GC testing over 50 ppm PCBs) must be low. A random sample of 937 Clor-N-Oil™ negatives were GC tested. Of the 937, 2.13% were false negatives.

MONS 020244

TABLE 1

CLASS INTERVAL (PPM)

	-1	1-5	6-15	16-25	26-46	47-99	100-475	476-999	1000+
Total	761	93	48	5	10	6	8	2	4
Percent	81.22	9.93	5.12	0.53	1.07	0.64	0.85	0.21	0.43

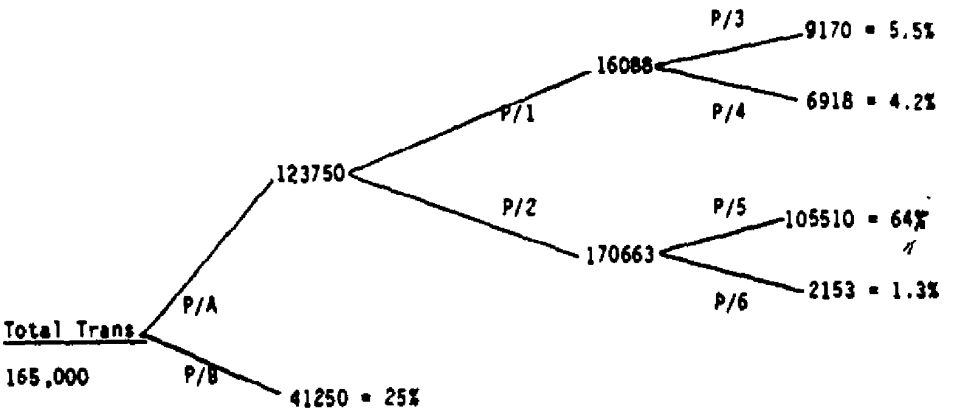
Less than 46 ppm: 97.87%

Greater than 46 ppm: 2.13%

Mean = 19.94 Mode = .01 Median = 1.5

A 2.13% rate of false negatives means that 2153 transformers contaminated with PCBs have been left in service and have the potential to leak or rupture, thus putting UP&L in violation for improper disposal. Disposal, cleanup, environmental costs and health effects increase dramatically if PCBs are present. Therefore, what is the probability of possessing a contaminated transformer, given it has been Clor-N-Oil™ tested and found to be less than 50 ppm (negative)?

MONS 020245



P/A =	Probability of transformer being Clor-N-Oil™ tested	.75
P/B =	Probability of transformer being manufacturer-certified as non-PCB	.25
P/1 =	Probability of a Clor-N-Oil™ Positive	.13
P/2 =	Probability of a Clor-N-Oil™ Negative	.87
P/3 =	Probability of a False Positive	.57
P/4 =	Probability of a True Positive	.43
P/5 =	Probability of a True Negative	.98
P/6 =	Probability of a False Negative	.02

Probability of possessing a contaminated transformer, given it is a Clor-N-Oil™ negative 1.3%

FIGURE 1

MONS 020246

UP&L expects .05% of all transformers to fail per year. That corresponds to 83 failures. What is the probability of a contaminated transformer failing, given it is a Clor-N-Oil™ negative?

$$165000(.0005)(.75)(.87)(.02) = 1.1$$

What is the probability of a contaminated transformer failing if the Clor-N-Oil™ kit is not used?

$$165000(.0005)(.055) = 4.5 \quad \text{(5.5\% is the total number of positives)}$$

Of the 20 false negative samples, 13 were Clor-N-Oil™ tested again at a central facility. Six were positive and seven were once again negative. All 13 samples were GC tested again. Those which Clor-N-Oil™ tested positive (6) had a GC value ranging from 53 to 265 ppm PCB. The discrepancy from negative to positive is probably due to sampling error or sample traceability. The seven samples that were Clor-N-Oil™ negatives both times each had a GC value of less than 1 ppm PCB when GC tested a second and third time. Therefore, the Clor-N-Oil™ test kit correctly predicted the GC result. All 20 samples were first Clor-N-Oil™ tested in the field rather than tested at a central facility. The lack of quality control over Clor-N-Oil™ testing in the field increases the probability of an inaccurate Clor-N-Oil™ result.

CONCLUSIONS

The Clor-N-Oil™ kit can reduce risk by as much as 76%, is simple to use, and inexpensive. By Clor-N-Oil™ testing rather than GC testing transformers, UP&L has saved approximately $1,650,000. Any equipment that ruptures or spills oil will be GC tested, but UP&L believes only 1 in 83 will be over 50 ppm PCB. By not using the kit, 4.5 out of 83 would be over 50 ppm PCB. UP&L believes that using the Clor-N-Oil™ kit will save on disposal and cleanup costs and reduce both environmental and health effects associated with PCBs.

COMPARISON OF ESTIMATED AND ACTUAL PCB VAPOR EXPOSURE DURING A SOIL EXCAVATION PROJECT

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A risk assessment was conducted for a chemical processing plant which had used the PCBs Aroclor 1248 and 1254 in its heat exchange fluids. Concentrations of vaporized PCBs were measured at levels one to two orders of magnitude greater than had been calculated in the risk assessment. Further, the PCBs detected consisted of the lighter Aroclor 1232 or a related compound. Because dust was shown to be adequately controlled, the discrepancy between projected and actual vapor-phase PCBs could only be explained by the unforseen formation of lighter, more volatile PCB species from partial evaporation of Aroclor 1248 and 1254, or failure to detect Aroclor 1232 in the original soil samples. Partial photolysis of the heavier PCBs could also contribute to the formation of the lower-chlorinated PCBs detected in air.

Background

The subject hazardous waste facility was a chemical processing plant which utilized heat-exchange fluids containing PCB 1248 and 1254. Following demolition of the plant, it was determined that the original PCB products or residuals had apparently leaked into surface soils throughout the 3-acre site. Soil was sampled based on a grid pattern that divided the site into roughly equivalent sections. Sampling was continuous, from surface to 6-foot depths, and analysis continued until the vertical extent of contamination was resolved. Additional surface samples were collected and analyzed to delineate the areal extent of contamination. A total of 321 samples were analyzed.

Soil Analyses

Soil samples were analyzed at a laboratory certified by the State of California for hazardous-waste analyses. The contents of each sampling sleeve was homogenized and analyzed according to EPA Method No. 8080. Up to 11 of the Aroclor peaks were used to quantitate the sample. The results of this sampling program revealed that site soils contained variable concentrations of PCBs. The average PCB level in the most heavily contaminated soils located within 12 inches of the surface was 326 ppm, with a range of <1 ppm to 4,400 ppm. The chromatograph from soil samples most closely approximated PCB 1254 (see Figure 1).

MONS 020248

Exposure Guidelines

For worker exposure to PCBs, the California OSHA 8-hour Time Weighted Average (TWA) Permissible Exposure Level (PEL) as well as the American Conference of Governmental Industrial Hygienists' TWA Threshold Limit Value (TLV) are the same: 1.0 mg/m^3 for PCBs containing 42% chlorine by weight (PCB 1242), and 0.5 mg/m^3 for PCBs containing 54% chlorine (PCB 1254). The National Institutes for Occupational Safety and Health, citing the potential for carcinogenic effects from PCBs, has established a recommended exposure guideline of 1 ug/m^3 (0.001 mg/m^3) as a 10-hour TWA for any PCB exposure.

A risk assessment was performed to evaluate community health hazards arising from direct and indirect human exposure to the site. Because there are no standards for allowable levels of PCBs in outdoor air, the risk assessment proposed a safe exposure guideline of 0.101 ug/m^3, for off-site community exposures, based on a calculated allowable daily intake of 2.5 ug/day for children. This guideline was extrapolated from a mortality study (1) and was designed to maintain a lifetime risk of cancer at less than 10^{-5}.

Exposure Estimation

PCBs in soils may reside in the vapor, liquid, or adsorbed phase. Direct evaporation from surface soils, diffusion of vapors from underlying strata, and wind-born dust may result in airborne exposures. Many physical and chemical properties of the soil and the contaminant, as well as external environmental factors, affect the rate of evaporation. Examples of physical properties of soils include tortuosity, porosity, and exposed surface area. Chemical properties include moisture content, organic carbon content, vapor pressure, and solubility. Important environmental factors include insolation, temperature, and wind speed (2).

In determining vapor emissions, rate-limiting parameters such as organic carbon adsorption and effective pore space are ignored when developing worst-case exposure assessments. The authors of the risk assessment applied Henry's Law in estimating vapor exposure levels in the immediate site vicinity.

According to Henry's Law:

$Cv = Hc\ Cl$

$Hc = 1.73 \times 10^{-7}$ @ 20°C (3)

$Cl = 326 \times 10^{-6}$ (g PCBs/g soil-water) X (MW water/MW PCBs)

$Cv = 2.3 \times 10^{-12}$ mole fractions

Using the above values and Henry's Law, one calculates that the near-surface contamination will contribute 0.022 ug PCBs/m^3 of air. (Daytime temperatures at this site averaged 20-25°C).

MONS 020249

Air Sampling

An air-sampling station was positioned at each of the four perimeters of the site. Samples were drawn at a fixed rate (3.5-4.0 liters per minute) by a vacuum pump through a 37-mm glass-fiber filter, followed by 400/200 mg Florisil tubes. Pumps were calibrated daily using a bubble meter.

Sample-collection procedures were based on established NIOSH protocols for PCB air sampling (Method 5503) with modification of the tube and filter size to increase the flow rates and thereby reduce detection limits. This method has been validated (4).

A total of 72 area samples and 11 employee breathing-zone samples were collected. The front section of the Florisil sampling sorbent and the filter were extracted together and analyzed according to NIOSH Method 5503. Aroclor standards and hexane blanks were analyzed periodically during sample tests as required by the method's quality-control protocols. Similar quantitation and identification methods were repeated from the soil testing.

Dust Monitoring

Dust was monitored with a GCA Miniram direct-reading aerosol monitor (detection limt of 0.001 mg/m^3) at upwind and downwind positions throughout the site. If the wind was negligible, readings were taken at each of the four perimeters. Readings were taken approximately every 45 minutes throughout the shift. Downwind readings were compared to upwind readings to determine site-related dust concentrations.

Wind speed and direction were also measured, with a wind vane and anemometer placed on a six-meter pole at the perimeter of the site.

Results

Analytical reuslts of the PCB air samples showed significantly higher levels of PCB vapors than had been estimated in the risk assessment. Downwind concentrations within the site boundaries averaged .563 ug/m^3, with a median concentration of .768 ug/m^3. Employee exposure levels averaged 1.845 ug/m^3 with a median of 2.180 ug/m^3. In addition, results were being reported as PCB 1232, based on the chromatographic comparison to standards, rather than the expected PCBs 1248 and 1254 (see Figure 2). Throughout the project, dust readings on the aerosol monitor were insignificant (<0.01 mg/m^3). The emissions were therefore believed to be associated with PCB vapor. This assumption was supported by analyzing selected filter and tube sections separately. Three of the four samples analyzed contained PCBs in the tube section only.

MONS 020250

Discussion

Risk analysis and estimation are valuable methods of assessing health hazards at hazardous-waste sites. This study demonstrates the limitations associated with estimating risk based on soil analysis data: such risk projections may not describe accurately the dynamic field conditions affecting overall airborne exposure levels. On-site air sampling is therefore a critical adjunct to the risk assessment process for PCB remediation.

Three possible reasons for detecting only PCB 1232 in ambient air at a site which was believed to contain only PCB 1248 and 1254 are 1) partial vaporization of the PCB mixtures; 2) masking of the PCB 1232 in the soil sample chromatographs and 3) photodecomposition of the heavier congeners during soil excavation.

Partial evaporation of the PCB 1248 and 1254 mixtures could also result in vapors characterized only as PCB 1232. The more volatile Cl_1 to Cl_4 biphenyls which constitute 96% of PCB 1232 are also present in PCB 1254 and PCB 1248 at approximate concentrations of 60% and 20%, respectively (5).

Masking may occur when the concentration of the undetected species is much lower than the quantitated species. Soils containing high levels of PCB 1248 and 1254 and low levels of PCB 1232 could produce vapor emissions identifiable only as PCB 1232, since its vapor pressure is 10 to 100 times greater than PCB 1254 and 1248.

The need to identify adequately all of the PCB congeners present in soils at a given site is illustrated in studies performed by Farmer, et al (6), which showed that extremely low soil concentrations of a semivolatile pollutant could produce maximum saturation vapor density at the soil/air interface.

Photolysis produces quaterphenyls, biphenyls and, in the case of hexachlorobiphenyl, chlorinated dibenzofurans (5). These studies dealt with photolysis in water columns where breakdown rates were less than 5% per year. Photolysis rates in exposed, aerated soils could be much higher. The health implications of exposure to photolysis break-down products warrants careful examination of these mechanisms.

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MONS 020252

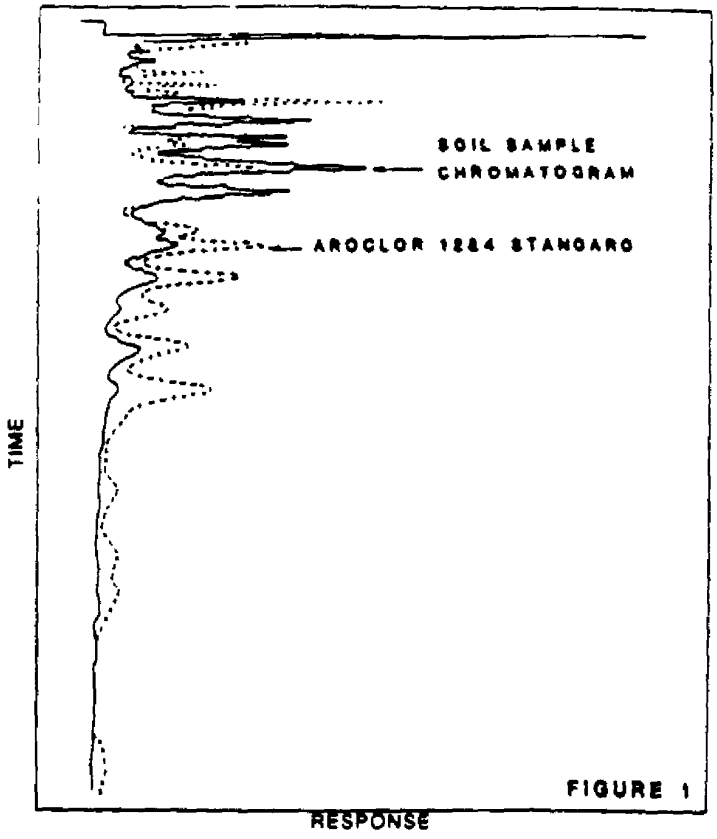

FIGURE 1

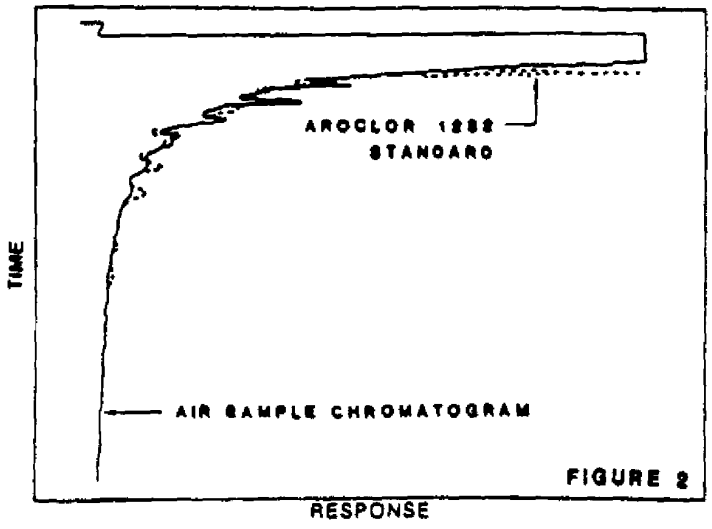

FIGURE 2

STATISTICALLY VALID SAMPLING STRATEGIES FOR PCB CONTAMINATION

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ABSTRACT

The sampling data taken during an assessment of buildings allegedly contaminated by polychlorinated biphenyls (PCBs) are often required to meet many different intended ends. Because of this, the design of sampling strategies for building contamination assessment is a complex task. Through the use of appropriate statistical techniques, the optimal sampling plan may be developed to meet a set of site-specific objectives.

INTRODUCTION

The common use of PCBs in electrical, hydraulic and heat transfer systems has created substantial contamination problems. Because the remediation or disposal of PCB-contaminated materials can be very costly, the source, extent and hazard presented by contamination must be carefully ascertained through extensive sampling and analysis. Generally, sampling strategies are implemented for one or more of the following reasons:

- Preliminary site investigation
- Determination of spatial distribution of contamination
- Identification of sources of contamination
- Risk assessment
- Decontamination support/confirmatory sampling
- Closure sampling.

Sampling buildings for PCB contamination is a complex problem requiring a site-specific sampling strategy predicated on the objectives of the investigation. For example, the type of sampling strategy that is consistent with the objectives of a preliminary site investigation may be inappropriate for those of a risk assessment.

The overall aim of any sampling strategy is to provide the most cost-effective approach to meeting the objectives of the investigation while maintaining a high degree of confidence, precision, and accuracy. A well-planned, statistically valid sampling strategy can help to achieve these goals. Although statistical design is not the only strategy available, without it the risks of failing to detect contamination cannot be quantified, and the costs of sampling and analysis versus unnecessary remediation cannot be rationally balanced.

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Preliminary Site Investigation

A preliminary site investigation utilizes all available historical and physical information to develop the initial scope of remediation. In the absence of historical data, the areas around the suspected source and potentially contaminated adjacent areas must be sampled to determine if a contamination problem exists. Potentially contaminated areas can be inferred from the mode of contamination. Clearly, it is easier to demonstrate the presence of contamination rather than its absence. If little or no contamination can be found, it may be reasonable to conclude the investigation. The correctness of this conclusion is largely a function of the adequacy and density of preliminary sampling. There is a direct relationship between sampling density and the quality and precision of the resulting estimates. Figure 1 graphically presents the increasing risk of failure to detect contamination with diminished sample size. Although the determination of "no contamination" requires a fairly intensive sampling effort, compositing of samples for analysis can be used to achieve a greater sampling density without increasing analytical costs. On the other hand, a single high sample can be construed as evidence of contamination. When a preliminary site investigation indicates that contamination is present, its spatial distribution throughout the facility must be carefully resolved.

Determination of Spatial Distribution of Contamination

Many sampling strategies involve judgment sampling, a process by which experts select what they believe to be representative specimens. In a recent sampling campaign, sampling locations were chosen by both random selection and judgment sampling. Random sampling locations were selected by a two-part grid scheme, while judgment sampling locations were based on physical characteristics such as areas of obvious staining. These two methods produced virtually identical results, as presented in Figure 2. Judgment sampling was no more effective than simple random sampling in detecting areas of high PCB contamination and, in fact, was potentially biased. The absence of bias adds strength to the belief that the samples taken by a particular sampling method are representative. In addition, the risks of falsely declaring a contaminated area "clean" cannot be quantified if the sampling scheme does not result in representative selections.

Identification of Sources of Contamination

PCB contamination may be present in and around fluorescent lights, air handling system bearings, elevator hydraulic systems, paint, equipment lubrication oils, generators, transformers and capacitors. Representative sample material from each of these sources is required to ascertain the presence of other aroclor contamination. The sampling objective is only to ascertain presence or absence of sources of PCB materials--sometimes a particularly important consideration from a liability standpoint. A nonrandom sampling approach may be used to identify possible sources of contamination by sampling oil and grease found in and around equipment locations.

Risk Assessment

Selection of sampling locations for health risk assessment may be based upon a probability quota sampling design. A quota sampling design is a stratified sample with a nonrandom selection of sampling units within strata (King, 1985). The facility is divided into activity zones to develop strata based on logically

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related activities, resulting in decreased variability of PCB concentration within strata. Within each zone, a quota of sample types and locations is developed, based on the desired relative precision of estimated PCB concantration.

Additionally, since the quota variable (e.g., surface type) is related to PCB concantration, relatively homogeneous selection units are produced. To the degree that homogeniety can be achieved, selection bias is eliminated, and variance is greatly reduced. Selection of precise sampling locations within quota groups should be random, thereby achieving unbiased estimates within those groups. If groups are to be combined in the computation of exposure, the combined estimate must be weighted by the probability of contact with that source. Only if sampling locations are chosen with probability proportional to frequency of contact can an overall estimate of exposure be formed without application of external weights. The formation of meaningful contact probabilitias can require extensive observation of deily plant activity.

Statistical Basis for Sampling Strategies

Conventional transformation of the data often fail to achieve normality, precluding the use of parametric statistical methods. Because the data are so highly skewed, the mean and standard deviation are artificially high. Rather than eliminate certain data points from the analysis or make unsupported assumptions about the underlying distribution of the data, order statistics such as the median serve as appropriate measures of central tendency (Lehmann, 1975). Based on ranking of the data from low to high magnitudes, order statistics make no assumptions about the distribution of the data. The median characterizes the typical observation without being overly influenced by outliers. Use of rank statistics does not preclude the characterization of uncertainty in a risk assessment. A greater emphasis on the inherent uncertainty of these estimates is required. With clear, meaningful precision objectives, the necessary number of samples can be determined to achieve the desired level of precision.

Decontamination Support/Confirmatory Sampling

First, potential remediation units are identified based on known or suspected contamination areas. These areas are divided into remediation units. The same is done in locations adjacent to the area where cleanup is to be initiated. These adjacent locations are sampled to test for possible lateral spread of contamination.

Each remediation unit is then divided into approximetely equal blocks. Areas that are highly contaminated require less information to make the remediation decision. Conversely, areas that are not heavily contaminated or may contain only small isolated areas of residual contamination require more extensive sampling to increase the probability of finding these hot spots. Additionally, large portions of areas that are not heavily contaminated may require no remediation, and, because they were extensively sampled, the decision not to remediate can be made with confidence. These results may qualify for inclusion in the project closure data base if the clean aree cen be effectively isolated during cleaning of contaminated areas.

Sample locations may be selected at random within blocks, a procedure easily accomplished with a random number table.

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If the decision criteria is based upon a mean or other measure of central tendency, at least two composite samples must be formed from samples taken within a remediation unit to obtain an estimate of variability. When the decision rule is to remediate if even one sample is greater than the action level, all samples taken from within a remediation unit can be composited as long as the product of the detection limit and the number of samples forming the composite does not exceed the action level.

Closure Sampling

An alternative approach to closure is to use order statistics. A closure guideline based on order statistics does not require the assumption of asymptotic normality but still allows probablity-qualified decisions. For example, a random sample of size 22 provides 90 percent confidence that no more than 10 percent of subsaquent samples taken from the fecility will exceed the maximum value observed in the closure data base. If the maximum value observed is less than or equal to the negotiated cleanup level, closure has been demonstrated. There are nonparametric tests of central tendency equivalent to the Student's t-test, such as the Wilcoxon signed rank test, which can be used to demonstrate closure (Wilcoxon, 1945).

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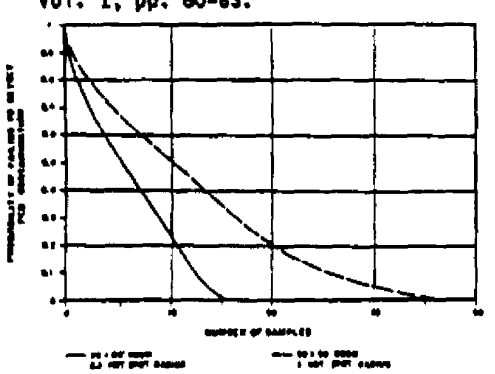

FIGURE 1
RELATIONSHIP OF THE PROBABILITY OF FAILING TO DETECT PCB CONTAMINATION AND SAMPLING EFFORT

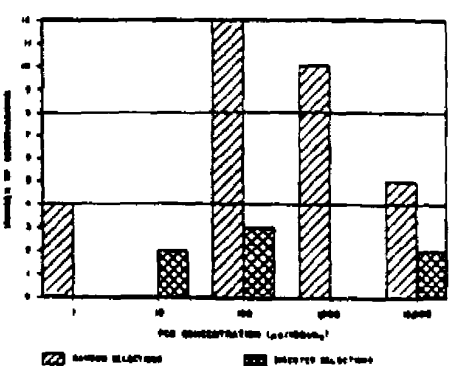

FIGURE 2
FREQUENCY DISTRIBUTION OF SAMPLE RESULTS

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PART 7:

PCDF AND RELATED COMPOUNDS

ANALYTICAL AND SAMPLING TECHNIQUES FOR THE DETERMINATION OF ULTRATRACE QUANTITIES OF POLYCHLORINATED DIBENZOFURANS AND DIBENZODIOXINS

by

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INTRODUCTION

Combustion of Askarel or polychlorinated biphenyl (PCB) containing materials can produce polychlorinated dibenzofuran (PCDF) or polychlorinated dibenzodioxin (PCDD). Retrofil transformer liquids (silicones) that contain 50 ppm (w/w) or less PCB have low conversion ratios for production of PCDF/PCDD in fires. The combustion of PCB in retrofill transformer liquids was studied in model fire experiments by Battelle and retrofilling was found to be an efficient way of removing PCB and potantial "furan" contamination from transformers in service (1). Various techniques to remove PCB or reduce PCB levels in transformers below the federally regulated 50 ppm (w/w) limit have been investigated. Retrofilling is a cost-effective alternative to replacement, especially in situations where transformer location makes removal difficult, or other situations where switch-out is economically unattractive.

The U.S. Environmental Protection Agency (U.S. EPA) promulgated the "Fire Rule" to address the risks posed by uncontrolled fires that involve high levels of PCB, situations where PCDF (or PCDD) might be formed in significant amounts (2). Exposure of PCB to a building fire can occur when Askarel-containing transformers are present at the fire site. Recognition of the risks posed by formation of PCDF/PCDD has prompted most utilities to remove PCB from active service by either replacing or retrofilling existing appliances.

To demonstrate the environmental safety of a recently designed facility for handling large amounts of PCB, a Toxic Substances Control Act (TSCA) demonstration test was conductad in September 1986. This paper describes the sampling and analytical procedures that were used to measure PCDF/PCDD levels in the liquid process streams handled at the plant and gaseous emissions generated in the decontamination process.

FIELD FACILITY AND SAMPLING

During September 1986, a demonstration test on a model PCB decontamination facility was conducted. This facility required TSCA compliance tests that were approved and monitored by both state regulators and the U.S. EPA. A formal Work Plan and Quality Assurance Plan were submitted for approval before actual testing was initiated in 1986.

The decontamination process involves separation procedures that isolate PCB from other liquid constituents. To meet the goals of the TSCA permit application, liquid and gaseous plant streams were sampled and analyzed for PCDF/PCDD concentrations at several locations in the feed, recovery, and discharge streams. A schematic diagram of the main process streams sampled is given in Figure 1.

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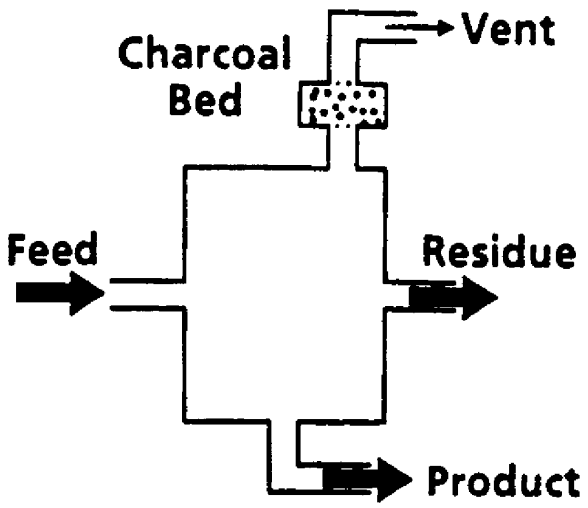

Figure 1. Schematic Diagram of Process Streams Sampled.

The plant is designed so no PCB emissions will be discharged outside the plant, total fugitive organic emissions from the plant will be less than 400 lb/yr, and no PCDF or PCDD emissions will occur from operations in the plant. To eliminate gaseous emissions of PCB/PCDF/PCDD, the final gas stream for the plant is vented through carbon adsorbers. The plant is designed to process large volumes of Askarel and Aroclor-containing liquids. The analysis strategy for this study required highly selective analytical methods because the organic compounds found in these samples can interfere with the determination of PCDD/PCDF.

SAMPLING

Both liquid process streams and vent gases were sampled during this program. A list of the measurement samples and quality control samples is given in Table 1

Liquid samples were collected through sampling valves built into the transfer piping. Sample valves were well flushed at the time of collection, and liquid samples were stored at 4°C in amber glass bottles fitted with Teflon-lined caps until analysis.

The gaseous emissions collected at the exit of the carbon vent system were found to be the most difficult matrix to sample. A schematic diagram of the gas vent sampling system is shown in Figure 2. The gas samples were collected in resin cartridges containing 20 g of XAD-2. Samples were collected under positive pressure and sampling system data such as gas flow, gas meter inlet and outlet temperature, system pressure, and XAD-2 column outlet temperature, were recorded for each run.

ANALYTICAL PROCEDURES

Liquid Samples (Feed, Product, Residue)

Liquid process samples were composited by mixing 0.15 g each of the triplicate samples taken at equal time intervals in the 6-hour tests. The composited liquid samples were stored in glass vials and were spiked with 5.0 ng of the internal standards listed in Table 2.

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TABLE 1

SAMPLES AND QUALITY CONTROL SAMPLES COLLECTED DURING THE TSCA DEMONSTRATION STUDY

Sample	Sample Type	Labelled Spike(s)	Native Spikes
Liquids			
Feed Stock 1	Grab	Yes	Yes
Feed Stock 2	Grab	Yes	Yes
Product 1	Grab	Yes	Yes
Product 2	Grab	Yes	Yes
Residue 1	Grab	Yes	Yes
Residue 2	Grab	Yes	Yes
Laboratory Blank	Solvent	No	No
Laboratory Spike Sample	Solvent	Yes	Yes
Gases			
Vent 1	XAD-2	Yes	No
Vent 2	XAD-2	Yes	No
Laboratory Blank	XAD-2	No	No
Field Blank Spike Sample	XAD-2	Yes	No

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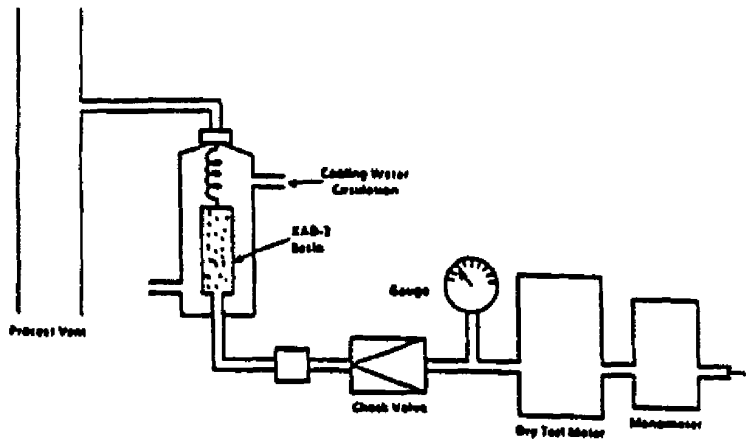

Figure 2. Gas Vent Sampling System.

TABLE 2

PCDF/PCDD LABELLED INTERNAL STANDARDS[a]

2,3,7,8-Tetra-CDD-$^{13}C_{12}$
1,2,3,7,8-Penta-CDD-$^{13}C_{12}$
1,2,3,6,7,8-Hexa-CDD-$^{13}C_{12}$
1,2,3,4,6,7,8-Hepta-CDD-$^{13}C_{12}$
1,2,3,4,6,7,8,9-Octa-CDD-$^{13}C_{12}$
2,3,7,8-Tetra-CDF-$^{13}C_{12}$
1,2,3,7,8-Penta-CDF-$^{13}C_{12}$
1,2,3,4,7,8-Hexa-CDF-$^{13}C_{12}$
1,2,3,4,6,7,8-Hepta-CDF-$^{13}C_{12}$

[a]CDD = chlorinated dibenzo-p-dioxin;
CDF = chlorinatad dibenzofuran.

The samples were diluted with hexane and analyzed using a high resolution gas chromatograph coupled to a high resolution mass spectrometer (HRGC/HRMS). A laboratory blank, 5 mL of hexane, was analyzed using the same internal standards listed in Table 2, and 10 native PCDF/PCDF compounds are listed in Table 3. The purpose of adding native standards to the laboratory blank was to demonstrate method recoveries in a clean matrix.

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TABLE 3

NATIVE PCDF/PCDD STANDARDS[a]

2,3,7,8-Tetra-CDD
1,2,3,7,8-Penta-CDD
1,2,3,4,7,8-Hexa-CDD
1,2,3,4,6,7,8-Hepta-CDD
1,2,3,4,6,7,8,9-Octa-CDD
2,3,7,8-Tetra-CDF
1,2,3,7,8-Penta-CDF
1,2,3,4,7,8-Hexa-CDF
1,2,3,4,6,7,8-Hepta-CDF
1,2,3,4,6,7,8,9-Octa-CDF

[a]CDD = chlorinated dibenzo-p-dioxin;
CDF = chlorinated dibenzofuran.

Vent Gas Samples

The two XAD-2 resin samples collected for PCDD/PCDF analysis and two XAD-2 field blanks were extracted with benzene using a Soxhlet apparatus. The samples were spiked in the Soxhlet extration thimble with 2.0 ng of each of the internal standards listed in Table 1.

Sample Preparation

The diluted process liquid samples and the extracted vent gas samples were prepared for analysis by fractionation through multilayer chromatography columns. Two sorbent media, silica gel and alumina, were used to isolate the PCDF/PCDD from interfering species. The silica gel columns contained alternating silica gel layers of differing pH: NaOH treated, neutral, and H_2SO_4 treated. Final eluates were spiked with the internal standard for recovery and quantification, 2,3,7,8-tetrachlorinated dibenzo-p-dioxin [$^{37}Cl_4$].

Samples Analyses

The sample extracts were analyzed for PCDD and PCDF by HRGC/HRMS. A Carlo Erba Model 4160 gas chromatograph interfaced to a VG Model 7070 high resolution mass spectrometer (HRMS) was used for these analyses. The mass spectrometer was operated in the Multiple Ion Mode (MID) to increase sensitivity, and a 50-M CP Sil-88 fused silica capillary chromatographic column was used to achieve compound separation.

The following criteria were used for positive identification:

(1) Correct retention time (± 2 seconds) relative to an authentic, synthetic standard (if available), or within the time window defined by the analysis of mixed CDD/CDF standards,

(2) Simultaneous response at two principal ion masses,

(3) Chlorine isotopic ratio within ±10 percent of the theoretical value.

PCDF/PCDD quantification was based on relative HRMS response ratios of the native analytes and three reference internal standards.

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RESULTS

Process Liquids

The process liquid samples were found to have traces (low ppb) of PCDF in the feed stocks and no measurable PCDD/PCDF in the product liquids. This result demonstrated the efficiency of the decontamination plant to separate any low level PCDD/PCDF contamination from the feed stocks without causing contamination of the product liquids. Table 4 shows results of the duplicate, composited field samples of feed, product and residues collected during plant operation. In Tables 3 and 4 the numbers in parenthesis are the estimated detection limits for each analyte or congener class. By including detection limits the method sensitivity for each measurement can be estimated. When the estimated detection limits are exceeded for any measurement, the actual values are given in the tables of data.

TABLE 4

RESULTS OF PCDD/PCDF ANALYSES
OF PROCESS LIQUID SAMPLES (ng/g)[a,b]

Sample	2,3,7,8-Tetra-CDF	Tetra-CDF	Penta-CDF	Hexa-CDF	Hepta-CDF	Octa-CDF
Feed 1	19	38	21	140	530	97
Feed 2	11	45	26	25	25	33
Product 1	(0.14)	(0.14)	(0.035)	(0.17)	(0.51)	(0.098)
Product 2	(0.10)	(0.10)	(0.08)	(0.20)	(0.24)	(0.22)
Residue 1	120	590	350	400	520	880
Residue 2	87	190	490	240	..[c]	..[c]
Laboratory Blank	(0.12)	(0.12)	(0.20)	(0.026)	(0.046)	(0.094)

[a]CDD = chlorinated dibenzo-p-dioxin; CDF = chlorinated dibenzofuran.

[b]Numbers in parentheses are estimated detection limits for the analyte or congener class.

[c]Not measured.

Vent Gas Samples

No measureable PCDF were found in any of the vent gas samples. Residual hepta- and octachlorinated dibenzo-p-dioxins were found at near ambient concentrations, however, field blanks showed equivalent levels indicating that these measured levels were due to contamination in the field operations, shipping, or laboratory operations. The results of vent gas analyses for PCDD are given in Table 5, which shows the analytical sensitivity obtainable by the methods employed in this study.

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QUALITY CONTROL

In the analyses conducted for this program, a large number of laboratory blanks, field blanks, and spiked reference samples were analyzed to demonstrate freedom from contamination and method performance. As mentioned in the previous section, traces of hepta- and octachlorinated dibenzo-p-dioxin were found in the vent gas field blanks. This quality control check averted reporting a false positive analysis for PCDD in the discharge vent gas. Table 6 gives recoveries of labelled 1,2,3,4-TCDD-$^{13}C_{12}$ spiked into the XAD-2 resin traps used for vent gas sampling. These spikes were administered to the XAD-2 traps before shipment to the test site. If a low recovery were found in these spiked samples, losses during sampling (e.g., sample breakthrough), handling, or sample workup would have been suspected. The true recoveries were all above 94 percent.

TABLE 5

RESULTS OF PCDD ANALYSES OF VENT GAS SAMPLES[a]

Sample	Units	2,3,7,8-Tetra-CDD	Tetra-CDD	Penta-CDD	Hexa-CDD	Hepta-CDD	Octa-CDD
Run 1	pg/ft^3	(0.14)	(0.14)	(0.22)	(0.15)	1.0	2.6
Run 2	pg/ft^3	(0.028)	(0.028)	(0.055)	(0.050)	0.38	0.73
Field Blank 1	ng	(0.007)	(0.007)	(0.009)	(0.006)	0.045	0.1
Field Blank 2	ng	(0.006)	(0.006)	(0.006)	(0.005)	(0.010)	0.067
Laboratory Blank	ng	(0.012)	(0.012)	(0.016)	(0.016)	0.092	(0.10)

[a]CDD = chlorinated dibenzo-p-dioxin; CDF = chlorinated dibenzofuran.

[b]Numbers in parantheses are estimated detection limits for analytes.

TABLE 6

RECOVERIES OF 1,2,3,4-TETRACHLORINATED DIBENZO-p-DIOXIN-$^{13}C_{12}$ SPIKED INTO THE XAD-2 RESIN TRAPS USED IN THIS STUDY.

Sample	Amount Spiked (ng)	Amount Found (ng)	Recovery (%)
Run 1	3.75	3.73	99.5
Run 2	3.75	3.53	94.1
Field Blank 1	1.25	1.26	101
Field Blank 2	1.25	1.25	100

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CONCLUSIONS

The results of this program demonstrated the sensitivity and reliablity of ultra-trace sampling and analysis methods to determine PCDF/PCDO concentrations in the liquid process streams and gaseous discharges from a demonstration PCB dacontamination facility.

In addition to proving the validity of the measurement techniques, the plant was found to operate efficiently to remove any residual PCDF/PCDO from process feed stocks, rendering a "clean" product. No PCDO or PCDF compounds could be found in any of the product samples, even at the ultra-trace concentrations measured by the HRMS technique. Also, no PCDF or PCDO vent discharges were measured above the blank levels found in XAD-2 cartridges used to collect vent gases.

The plant met the design criteria for PCDF and PCDO removal and was further found to meet the environmental goals established by local and national regulators.

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MONS 020266

PYROLYSIS AND COMBUSTION OF PCB AND TCB AS CONTAMINANTS IN TRANSFORMER FLUIDS

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ABSTRACT

Numerous laboratory simulations and real world events have demonstrated the thermal conversion of neat or high concentration PCBs into the much more toxic PCDFs; the conversion of concentrated tri and tetrachlorinated benzenes (TCBs) into PCDDs and PCDFs is similarly well established. However, as PCB and Askarel-filled transformers are removed from service, the thermal behavior of transformer fluids containing PCBs and/or TCBs at 50-5000 ppm becomes of increasing interest. This paper describes the pyrolysis (by heating in an open tube at 550°C) and combustion (by injection into a natural gas fueled flame) of dilute Aroclor 1254 and TCB solutions in mineral oil, silicone oil and tetrachloroethylene (TCE), and measurement of the resulting yields of PCDD and PCDF. In all experiments, dilution of PCB or TCB drastically decreased the mass of PCDF formed per gram of mixture. Alternatively, the yields expressed as mass of PCDF or PCDD per gram of chlorinated precursor exhibited modest but interesting variations with concentration providing some mechanistic insight into the formative and destructive processes underway.

INTRODUCTION

Abundant laboratory data and catastrophic real world events demonstrate that concentrated PCBs subjected to high temperatures can form elevated concentrations of PCDFs and that concentrated tri and tetrachlorobenzenes (TCBs) can, under similar conditions, form both PCDDs and PCDFs. However, the behavior of solutions containing low concentrations (less than 5000 ppm) of PCB or TCB is less studied and of some practical interest. For example, an estimated 2,000,000 mineral oil transformers have inadvertently become contaminated with PCBs at 50 ppm or higher and 200,000 such transformers have become contaminated above 500 ppm. Furthermore, the retrofilling process produces substantial volumes of waste dielectric fluid containing low PCB/TCB concentrations. Since the facility of PCDF/PCDD formation in such dilute solutions could be a significant factor in evaluating the hazards associated with operating contaminated or retrofilled transformers, in assessing the viability of the retrofilling process and in government regulation, EPRI sponsored a study in this laboratory to assess the ability of 5000-50 ppm Aroclor 1254, 124-trichlorobenzene and 1234-tetrachlorobenzene in mineral oil, silicone oil and tetrachloroethylene (TCE) to form such compounds.

METHODS

At the outset, it was recognized that no finite set of laboratory experiments could simulate the full range of real-world thermal events. Consequently, two thermal regimens were selected to be different from one another and distinct from

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the most widely used literature methodologies. Pyrolysis was conducted in vertically-mounted glass tubes (50 X 0.4 cm i.d.) sealed at their lower end and mounted in a thermostatically controlled furnace. Combustion was accomplished by syringe-drive injection of the subject mixture into a natural gas flame, passage of the combustion products through a three zone thermostatically controlled furnace and into a collection device.

Each sequence of experiments proceeded in two stages. First, preliminary experiments were performed on relatively concentrated solutions (usually 5000 ppm) to develop conditions capable of producing maximum PCDD/PCDF yields. Then, runs were performed with less concentrated solutions (usually 500 and 50 ppm) under identical conditions.

RESULTS AND DISCUSSION

The result of greatest practical interest is the observation that for both pyrolysis and combustion, for all three solvents, and for Aroclor 1254, trichlorobenzene and tetrachlorobenzene dilution sharply decreases the mass of PCDFs formed per gram of mixture. Yields from the pyrolysis of Aroclor 1254 in mineral oil and silicone oil, expressed now as mass PCDF formed/mass of chloroaromatic consumed, trended slightly upward in the 5000-50 ppm range. This result suggest that a process bimolecular in PCB and probably involving one or more solvent molecules may be consuming PCB and PCDF. Formation of PCDF could not be demonstrated in pyrolysed 5000-50 ppm solutions of Aroclor 1254 in TCE; scanning GC/MS experiments demonstrated facile reaction between biphenyls and TCE to add C_2Cl_3 groups. Pyrolysis of neat TCBs forms substantial yields of PCDFs and PCDDs; however, reaction of 5000 ppm solutions of various TCBs in mineral oil, silicone oil and TCE were not productive. This result suggests that the rate limiting and yield limiting step in PCDF/PCDD formation is kinetically second order in TCB.

Combustion of 5000 ppm solutions of Aroclor 1254 in mineral oil, silicone oil and TCE generated substantial yields of PCDF. Results obtained on 500 and 50 ppm solutions were consistent with the hypothesis that yields (mass PCDF formed/mass PCB combusted) were unaffected by dilution in 5000-50 ppm range.

Combustion of 5000 ppm solutions TCB in mineral oil, hexane or silicone oil either did not produce detectable PCDF or produced PCDF that was detectable only through heroic measures. This is probably a further manifestation of the fact that PCDF formation from TCBs is proceeding through a rate and yield limiting step that is second order in TCB. The yield of PCDF from 5000 ppm solutions of TCB in TCE, though still low, was at least an order of magnitude higher. This result may indicate the existence of a pathway of PCDF that is <u>not</u> kinetically second order in TCB. For example, TCB may instead react sequentially with three molecules of TCE and oxygen to form PCDF. TCE itself forms various PCDFs when combusted, although at much lower concentrations than when 5000 ppm TCB is present.

MONS 020269

Ultra Sensitive Bioassay for Dioxin

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ABSTRACT

2,3,7,8-tetrachlorodibenzo-p-dioxin (TCDD) has been shown to induce ethoxyresorufin-O-deethylase (EROD) activity or other biological responses in a number of cell cultures. Using a new cell line, WBAS 86-163B, an ultra sensitive bioassay for the detection of TCDD and related compounds has been devised. The target cells can detect TCDD concentrations as low as 2 X 10^{-13}M, which is at least 1.5 logs more sensitive than other bioassay systems. With the WBAS cell line, an induction response can be obtained after treatment of the target cells for only 18-24 hours, four to 10 days less than required by other bioassays. These cells have been used successfully to detect TCDD in soil and water samples.

Several dibenzo-p-dioxins, chlorinated phenols and polychlorinated biphenyls were tested for their abilities to induce EROD in the target cells. TCDD was at least several thousand fold more potent in eliciting a response than were the other chemicals. TCDD was 8 times more active as an EROD inducer than the next most effective chemical, 2,3,7,8-tetrachlorodibenzofuran.

INTRODUCTION

2,3,7,8-tetrachlorodibenzo-p-dioxin (TCDD) contamination in the environment has become an major public health issue. TCDD and and related compounds such as polychlorinated dibenzofurans are produced as by-products of the manufacture of industrial organic compounds (eg. 2,4,5-T) and also as the result of several combustion procedures (1-3). TCDD is an extremely stable compound which has been shown to cause liver and kidney damage, chloracne, mutagenesis of bacterial and mammalian cells as well as immunosuppression in mice (1,4-7). Thus, rapid and reproducible assays are needed to meet regulatory requirements for the detection of TCDD.

Several bioassays for the detection of TCDD have been described, including <u>in vitro</u> systems based upon inhibition of cell division, altered cellular morphology and enhanced differentiation of specific cell types, as well as induction of enzymes <u>in vivo</u> and <u>in vitro</u> (2, 8-10). In this paper we describe a detection method for TCDD which is quicker and more sensitive than other bioassays. The assay has a dynamic range of 0.060 pg/ml to 1.0 pg/ml and has been used to detect TCDD in water samples, sediment from water samples, and benzene extracts of soil samples.

METHODS

<u>Target Cells:</u> WBAS cell culture 86-163B (patent pending) was used in all experiments. The parent cell line was derived from mouse liver cells. After mutagenesis and cloning, cells were isolated based upon the inducibility of the enzyme ethoxyresorufin-O-deethylase (EROD) by TCDD. The cells were grown in Williams Medium E (WME) with 10% fetal bovine serum (FBS), 2 mM L-glutamine and 10 mM Hepes buffer (pH 7.4).

Chemicals and Sample Preparation: TCDD was obtained from the National Bureau of Standards (Gaithersburg, MD) or Cambridge Isotope Laboratories (CIL; Woburn, MA). Other dibenzo-p-dioxins, chlorinated phenols and polychlorinated biphenyls were purchased from Chem Services (West Chester, PA) or were synthesized at WBAS. 2,3,7,8-tetrachlorodibenzofuran (TCDBF) was obtained from CIL.

Assay: WBAS 86-163B cells were planted into 35 mm tissue culture dishes 20-24 hours before treatment. The medium was then removed from the cells and they were refed with 3 ml WME as described above, except that the FBS concentration is reduced to 1% (1% WME). For comparison of induction of EROD, the cells were treated with stock solutions of dibenzo-p-dioxin and 12 selected chlorinated compounds prepared in DMSO. Duplicate wells were treated with 10 ul of sample and an additional 10 ul of DMSO, resulting in a final DMSO concentration of 0.67%. A standard curve was generated by treating duplicate wells with appropriate concentrations of TCDD to yield final doses of 1, 0.5, 0.25, 0.125, 0.0625 and 0.0 pg/ml, all in a final concentration of 0.67% DMSO.

RESULTS

Performance of the Assay: In a typical bioassay a standard curve, generated from the induction of EROD by known amounts of TCDD, is used to quantitate dioxin in test samples. Each standard point is the average of two to three replicates. The r^2 values are generally above 0.950. To demonstrate the reproducibility of the system, results from seven assays were normalized with respect to the luminescence response of the 1.0 pg/ml TCDD control sample, setting this value to 100% (Table 1). All other values were determined by dividing the luminescence at each dose by the luminescence at 1.0. (eg. if the luminescence at 1.0 pg/ml was 400 and at 0.25 pg/ml was 130, the relative response at 0.25 pg/ml was 0.325). The r^2 of the data is 0.999; the midpoint of the curve is approximately 0.5 pg/ml (approximately 1.6×10^{-12} M). Inclusion of values for 2.0 pg/ml (which is outside the dynamic range of the system) lowers the r^2 to approximately 0.900. However, if the 2.0 pg/ml point is included, 4-parameter logistic analysis can be used and an r^2 of 0.990 can be obtained (not shown). Generally, the lowest concentration of TCDD used as a standard is 0.0625 pg/ml. However, in preliminary studies the inclusion of 0.03125 pg/ml does not adversely effect the r^2 of the standard curve. This lack of effect on the r^2 indicates that the 0.03125 pg/ml standard is within the dynamic range of the curve. At present, 0.0625 pg/ml (approximately 5×10^{-13}M) is used as the limit of detection. Samples falling below that level are retested at higher concentrations.

Comparison of Inducers: Thirteen compounds have been tested for their abilities to induce EROD in target cells (Table 2). The concentration of each chemical needed to induce an EROD response is compared to the amount of TCDD needed to induce a similar response (Table 2). For example, 20,000 pg/ml of 2,4,5-trichlorophanol (2,4,5-TCP) induces a response equivalent to that induced by 0.063 pg/ml of TCDD. By this method, TCDD is estimated to be 3.2×10^5 more potent (20,000/0.63) than 2,4,5-TCP as an inducer of EROD in the WBAS target cells. Table 1 shows that none of the dibenzo-p-dioxins, polychlorinated biphenyls or chlorinated phenols are nearly as effective as TCDD in provoking a response in the target cells. Only TCDBF is within a log of TCDD in its ability to induce the enzyme in the target cells.

DISCUSSION

In this paper we describe a rapid, sensitive and reproducible bioassay for the quantification of TCDD and other EROD inducers in environmental samples. The target cells, WBAS 86-163B was selected for its hyper inducibility by TCDD. Like other cell culture-based assays for TCDD (2,10,11), related compounds also induce a response in the target cells. Except for TCDD, TCDBF was the most potent inducer of EROD in the WBAS cells. Approximately an 8-fold increase in TCDBF, compared to TCDD, was required to obtain the same response. This is similar to the data reported by Gierthy and Crane (2) using the "flat cell assay" (FCA) and by Safe (10) using H4IIE cells. However, the sensitivity of the WBAS target cells is greater than reported for other systems. The midpoint of the dynamic range for our cells is approximately 1.6 X 10^{-12}M, as compared to 1.9 X 10^{-10}M reported by Safe (10). The limit of detection in the FCA is 10^{-11}M; with the WBAS cells it is below 2.0 X 10^{-13}M. Another advantage of the WBAS bioassay is the rapid turnover rate. The bioassay data presented here are for 20-24 hour exposures, or 3 to 11 days less than for other *in vitro* bioassays.

The bioassay, while not able to distinguish between similar isomers, provides many advantages not available in traditional methods of analysis. In general, the assay does not require highly skilled personnel or complex equipment. The assay is inexpensive and rapid, allowing replicate testing of samples at multiple dilutions. Although isomer specificity is not provided by the bioassay in a single test, the presence or absence of toxic chemicals which induce EROD, can be determined and expressed as "TCDD equivalents". Since most of these compounds are toxic chemicals, the data obtained from bioassays can be used to establish priorities in toxic chemical decontamination programs within industry and government.

Table 1

Cumulative Bioassay Response to 2,3,7,8 TCDD

TCDD (pg/ml)	Relative % Luminescence*
1.0	100.0
0.5	49.6
0.25	22.2
0.125	9.5
0.0625	4.3
0.0	0.0

*all values normalized with respect to 1.0 pg/ml result. Values represent the average of seven assays. Average luminescence at 1.0 pg/ml was 418.7.

r^2=0.999
y-intercept=2.14
slope=102.0

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Table 2

Induction of EROD in WBAS Target Cells by Chlorinated Compounds

Test Chemical	Inducing Dose (pg/ml)	Equivalent TCDD Dose (pg/ml)[a]	Relative TCDD Effect[b]
Dibenzo-p-dioxin (DD)	40,000	0.20	2.0×10^5
7-Cl-DD	40,000	0.32	1.3×10^5
2,7-dichloro-DD	40,000	0.20	2.0×10^5
1,2,3,4-tetrachloro-DD	20,000	2.00	1.0×10^4
1,2,3,4,7,8-hexachloro-DD	4,000	1.00	4.0×10^3
Pentachlorophenol	40,000	0.25	1.6×10^5
2,4,5 trichlorophenol	20,000	0.063	3.2×10^5
2,4 dichlorophenol	20,000	0.063	3.2×10^5
2,2',3,3,'6,6' Poly-chlorinated biphenyl(PCB)	40,000	0.125	3.2×10^5
2,3,3',4,4'5 PCB	10,000	2.00	5.0×10^3
2,2',4,5,6' PCB	40,000	0.125	3.2×10^5
2,2',4,5,5' PCB	40,000	0.125	3.2×10^5
2,3,7,8-tetrachloro-dibenzofuran	8	1.00	8

[a] Dose of TCDD required to give EROD response equivalent to that of the test chemical

[b] Dose of the test chemical divided by the dose of TCDD required to give an equivalent EROD response.

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BACKGROUND CONCENTRATIONS OF POLYCHLORINATED BIPHENYLS, POLYCHLORINATED DIBENZO-P-DIOXINS, AND POLYCHLORINATED DIBENZOFURANS IN OFFICE BUILDINGS IN SANTA FE, NEW MEXICO

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ABSTRACT

On June 17, 1985, a 500 KVA transformer located in the New Mexico State Highway Department General Office Building overheated and vented a vaporous-liquid aerosol containing polychlorinated biphenyls (PCBs), polychlorinated dibenzo-p-dioxins (PCDDs), and polychlorinated dibenzofurans (PCDFs). A study was conducted to determine how the reoccupancy criteria established for the building compared to normal concentrations of background contamination that exist in other similar buildings in Santa Fe. Three office buildings were tested for air and surface concentrations of PCBs, and PCDDs and PCDFs (tetra- through octa-chlorinated homologs and the respective 2,3,7,8-tetra isomers). Measurable air and surface concentrations of these contaminants were present in the buildings with all samples showing concentrations below the reoccupancy criteria. A statistically significant correlation between the concentrations of PCBs and PCDDs, PCDFs, or 2,3,7,8-tetrachlorodibenzo-p-dioxin equivalents in air and on surfaces was not demonstrated ($p > 0.10$). The similarities of the PCDD and PCDF homolog profiles of the building air and ambient air suggest a common source: atmospheric transport of combustion particulates. The data represent a baseline for these contaminants present in office buildings in Santa Fe, but may be indicative of those concentrations present in similar buildings in other geographical areas.

INTRODUCTION

On June 17, 1985, an electrical malfunction occurred in a 500 KVA power transformer located in the basement of the New Mexico State Highway Department (NMSHD) General Office Building in Santa Fe, New Mexico (1). The transformer overheated and vented a vaporous-liquid aerosol containing polychlorinated biphenyls (PCBs) and pyrolysis products including polychlorinated dibenzofurans (PCDFs) and, to a lesser extent polychlorinated dibenzo-p-dioxins (PCDDs) throughout the building. The building was decontaminated according to reoccupancy criteria developed by the Governor's Expert Advisory Panel.

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The reoccupancy criteria established by the Advisory Panel on the NMSHD Building were based on the maximum concentrations of PCDDs, PCDFs, and PCBs that would not result in significant human health risk if a person were exposed to these levels for a working lifetime of 30 years. The potential toxicity of the PCDD and PCDF mixtures were assessed based upon the calculated concentration of 2,3,7,8-tetrachlorodibenzo-p-dioxin equivalents (TCDD equivalents). The procedure, first proposed by the New York State Department of Health (2), permits calculation of the amount of 2,3,7,8-TCDD that would have to be present to exhibit the same toxicity as the measured quantities of each of the various PCDDs and PCDFs that are present, and the summation of these calculated amounts of 2,3,7,8-TCDD equivalents is an estimate of the total TCDD equivalent toxicity of the mixture. The air and surface criteria for total concentrations of TCDD equivalents are 2 pg/m^3 and 1 ng/m^2, respectively (3). These criteria assume a 10^{-6} increased cancer risk for a person spending a working lifetime of 30 years in the building. The air and surface criteria for PCBs are 0.5 ug/m^3 and 50 ug/m^2, respectively (4). The criteria take into account the usual presence of background levels of PCBs in air (5) and on surfaces (6), and were intended to guide the cleanup of the building within a safe margin of this background level.

This paper presents the results of a study conducted to determine how these reoccupancy criteria compare to normal concentrations of background contamination that exist in similar buildings in Santa Fe, NM.

STUDY DESIGN AND METHODS

The buildings selected for testing were similar to the NMSHD Building in both architectural design and age (constructed prior to 1970); and had no history of experiencing an electrical transformer fire or failure. Three commercial office buildings were tested for air and surface concentrations of PCBs, as well as PCDDs and PCDFs (tetra- through octa-chlorinated homologs and the 2,3,7,8-tetra isomers). The buildings were tested with the HVAC system operating under normal occupancy conditions. The fresh-air intake plenums were sampled to determine the concentration of these contaminants in the ambient air entering the buildings.

The surfaces tested for both PCBs, PCDDs, and PCDFs were limited to high skin contact surfaces. High skin contact surfaces are those which are repeatedly contacted, often for relatively long periods of time, by the building occupants. In this study, these surfaces were limited to include desks, tables and counters. The interior surfaces of the fresh-air intake plenums were tested for PCBs; these surfaces were limited to the floor and face of the air intake louvers.

Air Sampling Methods for PCBs, PCDDs, and PCDFs

The air samples for PCBs were collected using a two-stage sampling device (47-mm, 0.3 um binderless glass fiber filter proceeded by a glass tube containing two 400-mg sections of Florisil adsorbent). The samples were

collected for approximately a 50-hour period at a flow rate of 1.0 L/min to achieve an air volume of approximately 3 m^3. The samples were extracted and analyzed using capillary column gas chromatography/electron capture detection (7).

The air samples for PCDDs and PCDFs were collected using a two-stage sampling device developed by the New York State Health Department (8). The first stage is a 47-mm diameter, 0.3 um pore size binderless glass fiber filter. The second stage is a cartridge of 8 gms of silica gel adsorbent which is spiked with 2.5 ng each of labelled 2,3,7,8-TCDD and 2,3,7,8-TCDF before sampling. The samples were collected for approximately a 50-hour period at a flow rate of 20 L/min to achieve an air volume of approximately 60 m^3. The samples were extracted and analyzed using coupled high resolution gas chromatography-high resolution mass spectrometry (9).

Surface Sampling Methods for PCBs, PCDDs, and PCDFs

The surface wipe samples were collected using 3" x 3" soxhlet extracted cotton gauze pads wetted with 8-ml of pesticide grade hexane. The sampling procedure consisted of marking off a surface into a 0.25 m^2 area using a galvanized steel template or a metal tape measure. Each 0.25 m^2 area was wiped, with a gauze pad held with a gloved-hand, in two directions; the second direction was performed at a 90° angle to the first direction. The gauze pad sample was then placed in a glass sample container equipped with a Teflon-lined lid. Each PCDD and PCDF sample was a composite of four 0.25 m^2 samples for a total area of 1.0 m^2, whereas the PCB samples consisted of a single sample from an area of 0.25 m^2. The samples were extracted and analyzed according to the procedures previously referenced.

RESULTS AND DISCUSSION

Airborne Concentrations of PCBs, PCDDs and PCDFs

A total of 24 air samples were collected for the analysis of PCBs. Twenty-one of these samples were collected in the occupied space of the buildings; and three samples were collected in the fresh-air intake plenums. Four of the 21 samples showed concentrations above the detection limit (0.03 ug/m^3); these four samples ranged from 0.10 to 0.18 ug/m^3 (arithmetic mean = 0.14 ug/m^3). The PCB was identified as Aroclors 1254 and 1260. The ambient air samples did not show detectable concentrations at the same detection limit.

A total of 13 air samples were collected for tetra- through octa-chlorinated PCDD and PCDF homologs and the respective 2,3,7,8-tetra isomers. Nine samples were collected in the occupied workspace of the buildings (Table 1); and four samples were collected in the fresh-air intake plenums (Table 2). The concentrations of PCDDs in the building air ranged from 2.4 to 221 pg/m^3 (arithmetic mean = 96 pg/m^3). The 2,3,7,8-TCDD isomer was not present above the detection limit (0.09 to 0.09 pg/m^3) in any of the samples. The corresponding concentrations of PCDFs ranged from non-detected (0.49 pg/m^3)

to 162 pg/m^3 (arithmetic mean = 22 pg/m^3). The 2,3,7,8-TCDF isomer was present above the detection limit in two of the nine samples at a concentration of 1.0 pg/m^3 in each sample. The calculated concentrations of total TCDD equivalents for the building air samples ranged from 0.15 to 1.5 pg/m^3 (arithmetic mean = 0.50 pg/m^3), and that for the ambient air samples ranged from 0.01 to 0.21 pg/m^3 (arithmetic mean = 0.14 pg/m^3).

The PCDD and PCDF homolog profiles (tetra- through octa-CDDs and CDFs) for the building air and ambient air samples are presented in Figures 1 and 2, respectively. The hepta- and octa-chlorinated homologs predominated in both of the PCDD and PCDF profiles. The similarities of the homolog profiles of the building air and ambient air suggest a common source: atmospheric transport of combustion particulates (10-11).

The nine PCDD and PCDF samples were collected at locations paired to the collection of PCB samples to determine the existance of a statistical relationship. The concentrations of PCBs, PCDDs, PCDFs, and TCDD equivalents were log transformed and used in linear regression analysis. The correlation coefficients (-0.34,-0.09, and 0.31, respectively) were not statistically significantly different from zero ($p > 0.10$) in any case. Thus, a significant relationship between airborne concentrations of PCBs and concentrations of PCBs, PCDDs, PCDFs, or TCDD equivalents was not demonstrated.

Surface Concentrations of PCBs, PCDDs, and PCDFs

A total of 43 surface wipe samples were collected for the analysis of PCBs. Thirty-seven of these samples were collected on high skin contact surfaces; and six samples were collected from surfaces within the fresh-air intake plenums of the buildings. Fifteen of the 37 samples showed concentrations above the detection limit (1.0 ug/m^2); these 15 samples ranged from 1.2 to 5.9 ug/m^2 (arithmetic mean = 2.5 ug/m^2. The six samples collected within the fresh-air intake plenums ranged from non-detected (1.0 ug/m^2) to 34 ug/m^2 (arithmetic mean = 9.9 ug/m^2); five of the six samples showed concentrations above the detection limit. The PCB was identified as Aroclors 1242, 1254, and 1260.

A total of 12 samples were collected on high skin contact surfaces and analyzed for tetra- through octa-chlorinated PCDD and PCDF homologs and the respective 2,3,7,8-tetra isomers (Table 3). The surface concentrations of PCDDs ranged from 1.9 to 24 ng/m^2 (arithmetic mean = 8.5 ng/m^2). The 2,3,7,8-TCDD isomer was not present above the detection limit (0.02 ng/m^2) in any of the samples. The concentrations of PCDFs ranged from 0.35 to 4.3 ng/m^2 (arithmetic mean = 1.4 ng/m^2). The 2,3,7,8-isomer was present above the detection limit in all of the samples (range 0.02 to 0.76 ng/m^2, arithmetic mean = 0.29 ng/m^2); the field blanks did not show detectable concentrations at a detection limit of 0.01 ng/m^2. The calculated concentrations of total TCDD equivalents ranged from 0.04 to 0.42 ng/m^2 (arithmetic mean = 0.15 ng/m^2).

The PCDD and PCDF homolog profiles (tetra- through octa-chlorinated CDDs and CDFs) are presented in Figure 3. The PCDD profile shows a predominance of the

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higher chlorinated homologs with the maximum concentrations represented by the octa-CDDs. The PCDD profile is similar to that shown for both the building and ambient air samples. It is also similar to that found in commercial grade pentachlorophenol (12-13); PCDDs are manufacturing contaminants in chlorinated phenols (13). The PCDF homolog profile shows that the tetra- through octa-CDFs are more evenly distributed with the maximum concentrations represented by the tetra-CDFs.

The 12 PCDD and PCDF samples were collected at locations paired to the collection of PCB samples to determine the existence of a statistical relationship. The surface concentrations of PCBs, PCDDs, PCDFs, and TCDD equivalents were log transformed and used in linear regression analysis. The correlation coefficients (0.43, 0.31, and 0.42, respectively) were not statistically significantly different from zero ($p > 0.10$) in any case.

CONCLUSIONS

A study was conducted to determine the background concentrations of PCBs, PCDDs, and PCDFs in air and on surfaces in three commercial office buildings in Santa Fe, New Mexico. Measurable air and surface concentrations of these contaminants were present in the buildings. Comparison of the data to the reoccupancy criteria developed by the Governor's Advisory Panel for the HMSHD Building shows that the concentrations of PCBs, PCDDs, and PCDFs (converted to 2,3,7,8-TCDD equivalents) present as normal contamination in these office buildings are below the values used as the reoccupancy criteria.

The concentrations of PCBs, PCDFs, and PCDDs measured in these buildings may be indicative of the concentrations present in similar buildings in other geographical areas.

The similarities of the PCDD and PCDF homolog profiles of the building air and ambient air suggest a common source: atmospheric transport of combustion particulates.

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Table 1

AIRBORNE CONCENTRATIONS OF PCDDs AND PCDFs
IN THREE OFFICE BUILDINGS IN SANTA FE, NM

	n/N*	Concentration - pg/m^3	
		Mean**	Range
2,3,7,8-TCDD	0/9	0.13	(0.09 - 0.49)[a]
TCDDs	0/9	0.14	(0.09 - 0.49)
PeCDDs	0/9	0.24	(0.18 - 1.2)
HxCDDs	2/9	0.46	(0.23) - 1.5
HpCDDs	8/9	15	(0.13) - 26
OCDDs	9/9	80	2.0 - 170
Total PCDDs		96	2.4 - 221
2,3,7,8-TCDF	2/9	0.37	(0.11) - 1.0
TCDF	2/9	1.1	(0.11) - 5.0
PeCDF	4/9	0.62	(0.08) - 4.1
HxCDF	4/9	1.7	(0.05) - 14
HpCDF	6/9	6.8	(0.15) - 53
OCDF	5/9	11	(0.38) - 92
Total PCDFs		22	(0.49) - 162

* n/N denotes the number of samples above the detection limit/the total number of samples.

** Arithmetic mean calculated with non-detected values treated as one-half the detection limit.

[a] Value in parentheses is the limit of detection.

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Table 2

CONCENTRATIONS OF PCDDs AND PCDFs
IN AMBIENT AIR IN SANTA FE, NM

	n/N*	Concentration - pg/m^3 Mean**	Ranks
2,3,7,8-TCDD	0/4	0.06	(0.06 - 0.19)[a]
TCDDs	0/4	0.06	(0.06 - 0.19)
PeCDDs	0/4	0.11	(0.12 - 0.39)
HxCDDs	1/4	0.30	(0.16 - 0.72)
HpCDDs	4/4	15	4.8 - 22
OCDDs	4/4	116	14 - 170
Total PCDDs	4/4	132	19 - 193
2,3,7,8-TCDFs	0/4	0.08	(0.11 - 0.18)
TCDFs	0/4	0.08	(0.11 - 0.18)
PeCDFs	0/4	0.11	(0.07 - 0.28)
HxCDFs	1/4	0.10	(0.03 - 0.27)
HpCDFs	3/4	1.1	(0.22) - 1.6
OCDFs	3/4	1.8	(0.58) - 2.7
Total PCDFs		3.2	(0.65) - 4.5

* n/N denotes the number of samples above the detection limit/the total number of samples.

** Arithmetic mean calculated with non-detected values treated as one-half the detection limit.

[a] Value in parentheses is the limit of detection.

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Table 3

CONCENTRATIONS OF PCDDs AND PCDFs ON HIGH SKIN CONTACT SURFACES IN THREE OFFICE BUILDINGS IN SANTA FE, NM

	n/N*	Concentration - ng/m^2 Mean**	Range
2,3,7,8-TCDD	0/12	0.02	(0.01 - 0.10)[a]
TCDDs	0/12	0.02	(0.01 - 0.10)
PeCDDs	0/12	0.02	(0.01 - 0.11)
HxCDDs	10/12	0.70	(0.04) - 4.3
HpCDDs	12/12	1.7	0.23 - 5.3
OCDDs	12/12	0.6	1.6 - 14
Total PCDDs		8.5	1.9 - 24
2,3,7,8-TCDF	12/12	0.29	0.02 - 0.76
TCDFs	12/12	0.53	0.13 - 1.6
PeCDFs	9/12	0.08	(0.01) - 0.34
HxCDFs	11/12	0.17	(0.01) - 0.56
HpCDFs	12/12	0.27	(0.02) - 0.84
OCDFs	11/12	0.36	0.10 - 1.6
Total PCDFs		1.4	0.35 - 4.3

* n/N denotes the number of samples above the detection limit/the total number of samples.

** Arithmetic mean calculated with non-detected values treated as one-half the detection limit.

[a] Value in parentheses is the limit of detection.

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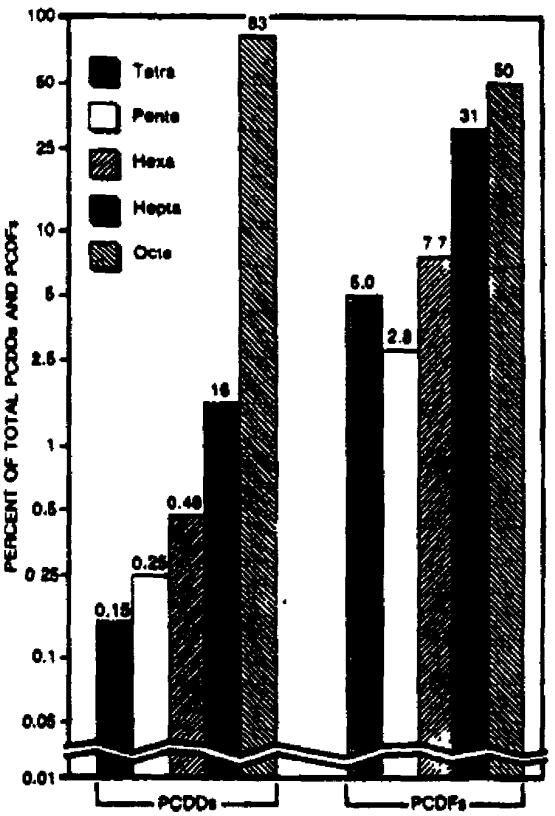

Figure 1: Tetra- through octa-chlorinated dibenzo-p-dioxin (PCDD) and dibenzofuran (PCDF) homolog profiles for air samples in three office buildings in Santa Fe, NM.

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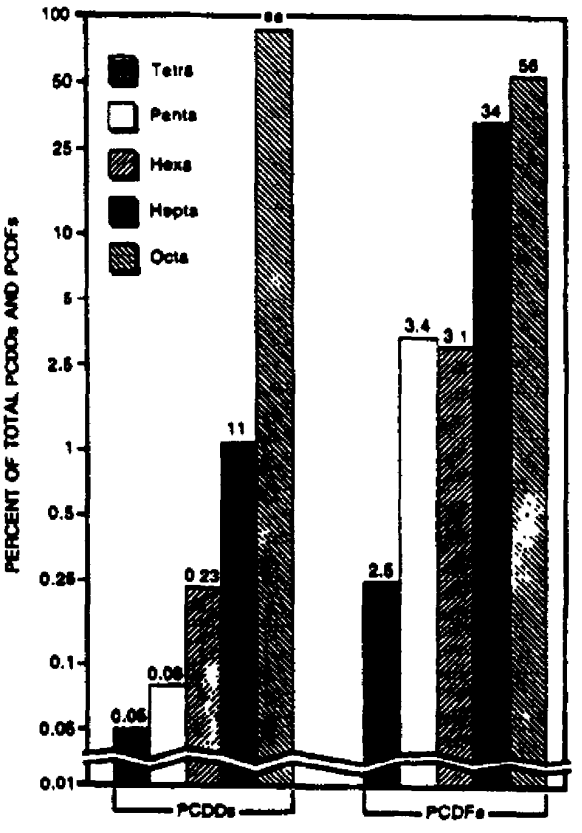

Figure 2: Tetra- through octa-chlorinated dibenzo-p-dioxin (PCDD) and dibenzofuran (PCDF) homolog profiles in ambient air samples obtained in Santa Fe, NM.

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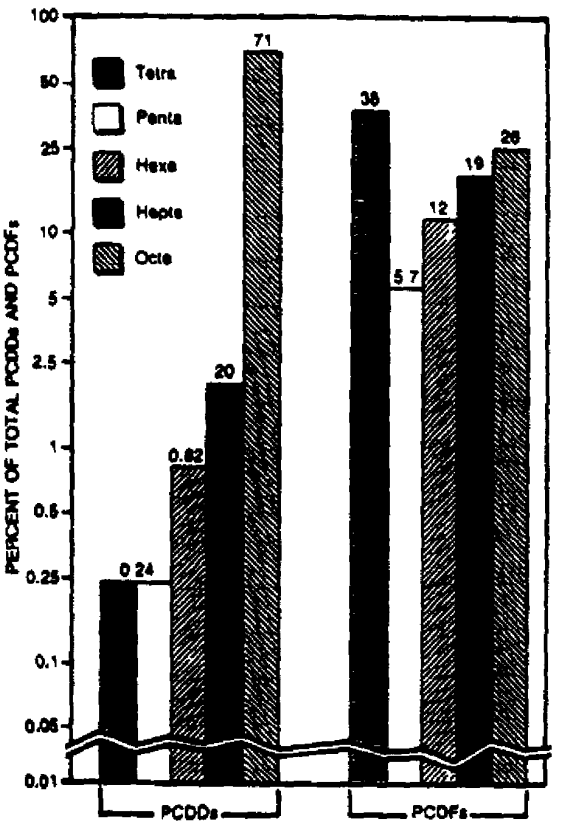

Figure 3: Tetra- through octa-chlorinated dibenzo-p-dioxin (PCDD) and dibenzofuran (PCDF) homolog profiles for wipe samples obtained on high skin contact surfaces in three office buildings in Santa Fe, NM.

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EVALUATION OF POTENTIAL HEALTH HAZARDS FROM FIRES INVOLVING LIQUID AND SOLID UTILITY MATERIALS

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ABSTRACT

Concern over the toxicity of PCB fluids has focused on both the major and trace pyrolysis and combustion products and has led to increased attention to the fire toxicity of all materials used in utility applications. To build a database on the same level as previous PCB degradation studies, it is necessary to perform thermal analyses on all potential substitutes. In this initial study, a laboratory screening approach has been applied to the evaluation of toxic pyrolysis and combustion products for a large number of replacement utility fluids. Following the evaluation of the screening data, creosote-treated wood was chosen for in-depth thermal analysis due to the high number and yields of toxic thermal reaction products produced. This paper discusses the results of these studies and the implications with regard to in-depth thermal analyses of all potential substitutes.

BACKGROUND

Because of potential health risks associated with exposure to polychlorinated biphenyl (PCB) combustion products, fluids containing these materials have provided utilities with previously unforeseen and very expensive problems [1]. This scenario has prompted concern regarding other materials used in constructing and insulating utility equipment. While extensive field and laboratory documentation exists on combustion byproducts from PCB fluids (including tetrachlorodibenzodioxins), systematic studies of the byproducts from fires involving other utility materials had not previously been investigated [2]. In light of this need, we have investigated the nature and yields of pyrolysis and combustion products from a number of utility materials. These materials include Kraft paper, Kraft-phenolic laminates, PCB replacement fluids, Nomex, various epoxies, creosote, and several cable insulations. Maple wood was used as a benchmark material to assess whether these utility materials pose any unique hazards when involved in fire (combustion) or arcing (pyrolysis) situations.

EXPERIMENTAL APPROACH

Thermal degradation experiments for replacement utility materials were conducted using a highly controlled laboratory test system referred to as the System for Thermal Diagnostic Studies (STDS). The STDS is a thermal degradation unit housed in a high temperature furnace interfaced downstream to a high resolution gas chromatographic/mass spectroscopic analytical system. This unique system has been used to resolve, detect, and quantify organic thermal reaction products [3].

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For the screening studies, two experiments were performed for each utility material. Each material was initially flashed in an air or nitrogen atmosphere using a CDS pyroprobe. The gaseous degradation products were then swept through the tubular quartz reactor at a gas-phase residence time of 1.5 sec with either air (combustion) or nitrogen (pyrolysis) as the reactor gas, depending on the reaction conditions simulated. Products from the oxidative atmosphere are those likely to form from open burning while those from the pyrolytic atmosphere are those most likely to form during arcing events.

For the in-depth thermal analysis, creosote-treated wood was initially degraded in an air or dry nitrogen atmosphere using a CDS pyroprobe. The gaseous degradation products were then swept through the tubular quartz reactor at a gas-phase residence time of 1.5 sec with either air (combustion) or nitrogen (pyrolysis) as the carrier gas, depending on the reaction conditions simulated. Data was obtained over a temperature range of 300-930°C.

RESULTS AND DISCUSSION

The screening studies indicated that a large majority of pyrolysis and combustion products from replacement utility materials are also produced from the similar degradation of wood-based materials. These toxic products included benzene, phenol, toluene, styrene, naphthalene, and acenaphthylene. However, these particular products are not unique to this material. They are also produced in the pyrolysis and/or combustion of a variety of man-made and natural materials. These include organic industrial wastes, sewage sludge, coal and coal-derived products, plastics, wool, fabrics, etc. Therefore, their presence should not constitute the basis for a decision on the safety and replacement of utility materials.

Table 1 presents a summary of unique pyrolysis and combustion products of replacement utility materials which appear to exhibit "significant" toxicity. In this context, "significant" toxicity is defined as a material which is a known or suspect carcinogen, mutagen, or a material with a toxicity threshold level of less than 20 mg/kg. Major products are defined as having yields of greater than 0.1%. Several observations are deduced upon critical inspection of Table 1:

1. For all utility materials, yields of toxic products are considerably higher under pyrolytic conditions.
2. With the exception of trace levels of aniline, quinoline, and benzotriazole produced from Herkolite pyrolysis, cellulose-based utility materials produce relatively low toxicity products.
3. Biphenyl and methylanthracene represent toxic products from replacement capacitor fluids with relative yields dependent on fluid composition and oxygen concentration. Comparison of toxicity data for potential thermal decomposition products from PCBs, i.e., TCDF, and chlorobenzenes, i.e., TCDD, with replacement alkylaromatic capacitor fluids indicates a substantial improvement in terms of human health hazard. However, it should be noted that the toxicities of many of the products observed in this study have not yet been quantified.
4. For the remaining materials studied, ethylene propylene rubber and RTEmp fluid represented low toxicity utility materials; Nomex, various epoxies, and polyvinyl chloride produced moderate levels of toxic products; and pyrolysis and combustion of creosote produced the largest number of toxic products, all with very high yields.
5. The results of the screening studies indicated that creosote was a prime candidate for an in-depth thermal analysis due to the high level of toxic thermal reaction products produced.

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A creosote-treated utility pole was chosen as the utility material for in-depth thermal analysis. This complex sample was chosen in an attempt to simulate the overall fuel composition which would be present during creosote pyrolysis or combustion in an actual utility fire or arcing situation. The wood substrate was also chosen to provide the possibility of synergism with regard to the number of possible toxic pyrolysis and combustion byproducts.

Table 2 presents a summary of the thermal stability of unique toxic combustion and pyrolysis byproducts from creosote-treated wood. The results indicated that the combustion of this material at temperatures in excess of 600°C will likely result in minimal (detection limit = 40 ppm) yields of toxic thermal reaction products. Under oxygen-deficient conditions, minimum byproduct yields were not observed over the temperature range (300-930°C) of experiments, indicating that higher temperatures will likely be required to thermally degrade this utility material into minimal yields of toxic thermal reaction products. The in-depth thermal analysis of creosote-treated wood has also resulted in the following specific conclusions:

1. Numerous toxic combustion and pyrolysis byproducts are produced with a majority of these compounds unique to this material; the combustion products included 2-propenal, 3-buten-2-one, 2-butenol, 2-furancarboxaldehyde, benzene and biphenyl; in addition to these compounds, other pyrolysis products included 5-hydroxy-2-furancarboxaldehyde, 2-methyl-1,3-butadiene, phenol, and isoquinoline;
2. Analysis of combustion byproducts indicates significant yields at low temperatures; however, all of these compounds are thermally labile with detection limit yields at temperatures equal to or greater than 600°C;
3. Analysis of pyrolysis byproducts indicates significant yields at low temperatures; in addition, several of these compounds exhibit an increase in concentration at high temperatures (800-900°C).
4. In-depth thermal analysis is the best methodology to completely elucidate the nature, yield, and stability of pyrolysis and combustion byproducts of utility materials.

The significant yields of toxic pyrolysis and combustion byproducts observed from screening studies of Herkolite, all five replacement capacitor fluids (Pemcol, Edison II, Edisol III, Selectrol, and Dielektrol), all three epoxies (the anhydride cure, the amine cure, and the dry-transformer epoxy), Nomex, PVC, and creosote suggest that these utility materials represent prime candidates for in-depth thermal degradation analyses. As evidenced in the creosote-treated wood in-depth thermal analysis, this methodology represents an excellent scientific approach to the determination of the nature, yield, and stability of potential toxic combustion and pyrolysis byproducts produced from utility materials.

ACKNOWLEDGEMENTS

We wish to offer special gratitude to Dr. Gil Addis for his invaluable guidance for the duration of this research project. Thanks also to EPRI for sponsorship of this work and the EPRI advisors too numerous to name for providing guidance, samples, and support in this program. Last, but not least, thanks also to Mrs. Diane Leach for typing the extended abstract.

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TABLE 2

SUMMARY OF THE THERMAL STABILITIES OF UNIQUE TOXIC COMBUSTION AND PYROLYSIS PRODUCTS FROM CREOSOTE-TREATED WOOD

COMPOUND	$TEMPERATURE_{bdl}$ (°C)(1) (air)	(nitrogen)	
2-Propanal	500	700	
3-Buten-2-one	550	600	
2-Butenol	500	700	
2-Furancarboxaldehyde	550	800	
Benzene	700	--	(2)
Phenol	700	900	
Biphenyl	550	930	
Isoquinoline	ND (3)	900	
2-Methyl-1,3-butadiene	ND	800	
5-Hydroxy-2-furancarboxaldehyde	ND	500	

(1) $Temperature_{bdl}$ refers to the temperature at which the compound is $\leq$ the detection limit of 0.004% yield.
(2) Although benzene is seen in the nitrogen runs of the creosote-treated wood study, its presence in this atmosphere had been seen before in the maple wood screening studies.
(3) ND indicates that this compound was never detected in air at any temperature.

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TABLE 1

TOXIC PRODUCTS UNIQUE TO REPLACEMENT UTILITY MATERIALS

UTILITY MATERIAL	TOXIC PRODUCT	TOXICITY	YIELD(AIR)	YIELD(N2)
Creosote	Biphenyl	Mutagen	Major	Major
Creosote	Chrysene	Carcinogen	Major	Major
Creosote	Methylenthrecene	Mutagen	Major	Major
Creosote	Acenephthene	Mutagen	Major	Major
Creosote	Quinoline	Mutagen	Major	Major
Dielektrol	Methylenthrecene	Mutagen	ND*	Major
Edieol III	Biphenyl	Mutagen	Major	Major
Edieon II	Methylenthracene	Mutagen	ND	Major
Edieon II	Biphenyl	Mutagen	Major	Major
Epoxy(amine cure)	Acrylonitrile	Carcinogen	ND	Major
Epoxy(amine cure)	Pyridinamine	LD_{50}:590 ug/kg	Major	Major
Epoxy(anhydride cure)	Acenephthene	Mutagen	ND	Major
Epoxy(anhydride cure)	Methylphthelate	LC_{50}:5.5mg/kg	Major	Minor
Epoxy(dry treneformer)	Acenephthene	Mutagen	ND	Minor
Epoxy(dry treneformer)	Biphenyl	Mutagen	ND	Minor
Epoxy(dry treneformer)	Propenel	Carcinogen	Major	ND
Herkolite	Benzotriazole	Mutagen	Minor	ND
Herkolite	Aniline	Carcinogen	ND	Minor
Herkolite	Quinoline	Mutagen	ND	Minor
Nomex	Acrylonitrile	Carcinogen	ND	Major
Nomex	Aniline	Mutagen	ND	Major
Nomex	Benzenediamine	Mutagen	ND	Minor
PVC-MRI	Biphenyl	Mutagen	Minor	Minor
PVC-MRI	Benzylchloride	Carcinogen	Minor	ND
PVC-VP	Acenephthene	Mutagen	ND	Major
PVC-VP	Benzylchloride	Carcinogen	Minor	ND
PVC-VP	Biphenyl	Mutagen	Minor	Major
Selectrol	Biphenyl	Mutagen	ND	Major
Selectrol	Methylenthrecene	Mutagen	Major	Major
Wemcol	Biphenyl	Mutagen	Major	Major

*not detected

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FIRES INVOLVING LIQUID AND SOLID UTILITY MATERIALS: A POTENTIAL UTILITY CONCERN. II. PVC.

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ABSTRACT

The pyrolysis and combustion products of poly(vinyl chloride) (PVC) were studied by the use of on-line pyrolysis-GC/MS systems in order to investigate the potential role of PVC in the formation of toxic or otherwise hemful species during utility fires. PVC was thermally degraded alone and in the presence of ethylene-propylene rubber, rust, and steel and found to produce benzene, styrene, and other aromatic species in abundance under both air and inert atmospheres. The presence of oxygen is shown to facilitate the formation of chlorinated benzenes along with trace amounts of polychloronaphthalenes as a result of PVC degradation. Factors influencing the relative amounts of products included sample residence time in the heated zone, presence or absence of air, and presence or absence of metal species.

INTRODUCTION

Since the restriction of manufacture and commercial use of polychlorinated biphenyls (PCBs) in 1976, there has been considerable interest within the electrical utility industry regarding the toxicity of the alternative dielectric fluids and their thermal degradation products. In addition, the PCB issue has also sparked interest regarding the other types of materials (such as plastics, binders, paints, support materials, and electrical insulators) presently used in utility devices and the potential of these materials to produce toxic or otherwise undesirable species in the event of a fire. Of particular interest are chlorine-containing materials, which have been implicated as one of the sources of polychlorinated dibenzo-p-dioxins and furans (PCDDs and PCDFs) in fires and incineration. Materials in this category include PCBs, some of the alternate dielectric fluids such as tetrachloroethene or trichlorobenzene mixtures, and poly(vinyl chloride) (PVC), which is used widely as a wire insulation.

In this study, the pyrolysis and combustion of PVC was investigated in detail using an on-line pyrolysis-gas chromatography/mass spectrometer system. Furthermore, as PVC is rarely used alone as a utility material, the effects of added organic material or catalytic surfaces that might be found with PVC in a utility device, on the

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thermal degradation of PVC, were investigated. For these preliminary experiments, the materials ethylene-propylene rubber (EPR), iron(III) oxide (rust), and steel were chosen to be copyrolyzed with PVC since they may be expected to be found near or with PVC in utility devices. The objective of this work is to obtain additional information regarding the potential of PVC to form harmful or undesirable pyrolysates in a utility fire.

EXPERIMENTAL SECTION

The PVC and EPR were obtained as standard grade insulation from electrical cables and were not treated in any way prior to analysis. Samples were taken by cutting small pieces of approximately 100 µg from the cable insulation by the use of a razor blade. The rust (Fe_2O_3) and steel wool were obtained as reagent chemicals and were used as received.

Thermal degradation experiments were performed with two different pyrolyzers. A furnace-type pyrolyzer in which the sample is forced to pass through the hot zone was used to characterize secondary reactions of the material and its primary pyrolysis products. A filament-type pyroprobe which can heat a sample rapidly and reproducibly to a specified set temperature was used to characterize the primary pyrolytic processes of the utility materials. Experiments were carried out either under an atmosphere of helium (pyrolysis) or air (combustion) over the temperature range of 500 to 900°C.

Pyrolysis products were characterized by gas chromatography/mass spectrometry (GC/MS) using electron impact (EI) ionization and negative chemical ionization (NCI). Full scan, low resolution mass spectra were acquired to enable tentative identification of the pyrolysis/combustion products by library searching and manual interpretation. Confirmation of trace components was accomplished by the use of selected ion current profiles (SICPs) of major ions in the mass spectra of the components of interest.

RESULTS

The major products resulting from the pyrolysis and combustion of neat PVC along with the PVC/EPR and PVC/rust mixtures using the filament pyrolyzer are shown as an example in Table 1. PVC characteristically produces abundant nonhalogenated aromatic degradation products, such as benzene, styrene, and naphthalene, under both helium and air at 900°C, which is in good agreement with the literature (1-4) Thermal degradation in air results in a surprising increase in the levels of polychlorobenzenes (CBs) and the expected increase in the levels of oxygenated species but no evidence (0.001% yield) of chlorinated aromatics which contain oxygen (i e

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PCDDs or PCDFs). The addition of rust or EPR does not seem to effect the production of halogenated species to any major extent.

When the furnace pyrolyzer is used, similar results are obtained, except that the nonhalogenated aromatic species account for a larger proportion of the total product yield and the CBs are very minor products. This reduction in CB production and increase in aromatic production is a result of the secondary reactions of PVC and its thermal degradation products in the furnace pyrolyzer to form highly stable aromatic species such as benzene and naphthalene. Examination by NCI-MS of the products generated by the furnace pyrolyzer, however, revealed that higher CBs (penta- and hexachlorobenzene) along with polychloronaphthalenes are produced by PVC under combustion conditions. Although these polychlorinated aromatics represent one of the smaller fractions of the overall yield of degradation products, they remain as evidence that some of the chlorine liberated by PVC as HCl (1) can become incorporated into the aromatic nuclei.

CONCLUSION

It may be concluded from this work that PVC alone and admixed with other common utility materials thermally degrades in the absence of oxygen to predominantly form nonhalogenated aromatic species such as benzene and naphthalene. In the presence of oxygen, however, PVC displays a tendency to form low, but real levels of chlorinated aromatics which could be of concern in a fire since they are frequently implicated as precursors of PCDDs and PCDFs. No evidence of PCDD or PCDF formation above the 0.001% yield level was observed in this work; however, modifications to the on-line pyrolytic instrumentation used here may allow future studies to examine yields well below 0.001%.

ACKNOWLEDGMENTS

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Table 1

BREAKDOWN OF MAJOR PYROLYSIS AND COMBUSTION PRODUCTS OF PVC
(Filament Pyrolyzer)

Material	Experiment Type*	Percent Relative Abundance by Product Type**				
		A	B	C	D	E
PVC	PY900	73.5	N/D	N/D	10	16.5
PVC	CO900	28.1	11.4	9.4	26.2	24.9
PVC + EPR	PY900	46.1	22.1	1.3	0.6	29.9
PVC + EPR	CO900	27.7	19.7	5.3	27.6	19.7
PVC + Rust	PY900	44.4	8.8	1.5	19.1	26.2
PVC + Rust	CO900	28.1	13.2	5.5	52.5	0.7

*PY900 = 900°C pyrolysis; CO900 = 900°C combustion.

**A = nonhalogenated aromatic products (benzene, styrene, naphthalene, etc.);
B = aliphatic products (alkanes, alkenes, cycloalkanes);
C = chlorobenzenes;
D = oxygenated products (phthalates, benzaldehyde, etc.);
E = unidentified, low abundance products.
N/D = not observed.

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INTERLABORATORY COMPARISON OF PCDD AND PCDF ANALYSIS

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ABSTRACT

A statistical analysis of interlaboratory data from five laboratories was performed to assess the accuracy and precision of analysis for selected PCDD and PCDFs in three baseline and seven in-service utility samples. Comparisons are made by compound, sample type and laboratory.

INTRODUCTION

The Electric Power Research Institute (EPRI) initiated a research program with the following major elements:

- Round-robin method evaluation using the best available in-house extraction and analytical techniques, and
- Analysis of in-service dielectric fluids for the presence of polychlorinated dibenzofurans (PCDFs) and polychlorinated dibenzodioxin (PCDDs) of interest.

The round-robin method evaluation was designed to validate analytical methodology and to determine the accuracy with which specific congeners could be quantified. It included the synthesis of native and isotopically-labeled standards, and the analysis of spiked sample matrices (baseline samples). Standards and the spiked matrix samples were prepared by a contract laboratory and distributed to the participating round-robin laboratories. The samples were analyzed "blind" by five independent contract laboratories (2-5).

The in-service dielectric study consisted of (1) developing appropriate sample selection criteria, (2) obtaining representative samples, and (3) analyzing the selected samples. General Electric Company prepared the selection criteria document (1), the samples were collected by one of the laboratories (2), and the analyses were performed by the five laboratories.

The study was conducted in two phases. Phase I involved the analysis for PCDFs and PCDDs in five spiked "baseline" samples (Aroclor 1016, Aroclor 1242, Aroclor 1260, a tri- and tetrachlorobenzene mixture and aged mineral oil) and four in-service dielectric fluids, with each participating laboratory using their best available in-house extraction and analysis techniques. Since validated analytical methods did not exist before this program was initiated this phase

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was necessary In this phase response factors for the comoponents of interest were generated at a single concentration using a standard calibration solution After evaluating these results, and implementing several improvements to the methodology, Phase II was initiated.

Phase II was designed to provide quantitative data on PCDFs and PCDDs which allowed a statistical interlaboratory comparison to be made. The specific aims were to:

- determine the accuracy of analysis by each perticipating laboratory for a selected list of PCDFs/PCDDs in spiked samples.
- determine the precision of analysis by each laboratory for PCDFs/PCDDs in spiked and in-service samples.
- perform an interlaboratory comparison, and thus a comparison of methods employed, for precision and accuracy, and
- measure the levels of PCDFs and PCDDs in in-service samples.

Phase II samples included three spiked baseline samples (Aroclor 1016, Aroclor 1260, and aged mineral oil) and seven in-service dielectric fluids. The in-service samples were selected to attempt to be representative of a wide range of conditions to challenge the analytical methodology

The results of the interlaboratory study for samples analyzed in Phase II are presented.

SAMPLE SELECTION, SAMPLE PREPARATION, AND CHEMICAL ANALYSIS

Three spiked baseline samples (Aroclor 1016, Aroclor 1260, and aged mineral oil) were prepared as previously reported (5). Selected PCDD and PCDF congeners were added to each sample at known levels to assess accuracy of the methods employed for sample extraction, purification and analysis.

Seven in-service dielectric fluids were chosen according to specified criteria (1) and used to determine the precision of the methods for PCDD and PCDF determination.

Five independent laboratories participated in the interlaboratory study. Each laboratory was provided with aliquots of the three baseline and seven in-service samples. Also native and isotopically labeled PCDD and PCDF standards were synthesized (5) and provided to each participating laboratory. Subsequently, each laboratory employed their best available in-house extraction and analysis techniques (2-5). Calibration curves were generated over the linear response range of the gas chromatograph/mass spectrometer system (GC/MS) at five concentrations using the same initial stock calibration solutions containing eight native and five isotopically labeled PCDD and PCDFs (5). All analyses employed capillary GC/MS analysis, one of the five systems included high resolution MS, and one negative ion chemical ionization, while the remaining four systems used electron impact

RESULTS AND DISCUSSION

Table 1 gives a listing of the PCDD and PCDFs that were chemically and statistically analyzed. From each laboratory the concentrations were reported

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TABLE 1

PCDD AND PCDF COMPOUNDS MEASURED IN BASELINE AND IN SERVICE SAMPLES

Compound	Compound
Total TCDF	2,3,7,8- + 2 3,4,8-TCDF
Total PNCDF	1,2,3,7,8- + 1,2,3,4,8-PNCDF
Total HXCDF	2,3,4,7,8-PNCDF
Total HPCDF	1,2,3,4,7,8- + 1,2,3 4,7,9-HXCDF
OCDF	1,2,3,7,8,9-HXCDF
Total TCDD	1,2,3,6,7,8-HXCDF
OCDD	2,3,4,6,7,8-HXCDF
2,3,7,8-TCDD	1,2,3,4,6,7,9-HPCDF

as ng/g (ppb), and the means and standard deviations of the determinations were computed by laboratory, sample and PCDD/PCDF compound. One laboratory (LAB 5) reported only one determination for each sample and PCDD/PCDF compound, while LAB 4 reported three extracts with three determinations for each extract. The remaining laboratories reported one extract per sample and three determinations per extract. The database contained large variations in concentrations among the PCDD/PCDF compounds within a sample, and among samples for a given compound Thus, there was a substantial dynamic range in levels which allowed for assessing accuracy and precision at low and high levels and in different sample types (matrices).

The standard deviations varied and equal variances could not be assumed. Since the standard deviations tended to increase as the concentration levels increased, basic data transformations of taking logarithms and square roots were applied to this database. Unfortunately, this did not minimize the problem and therefore, limited the types of statistical tests that could be performed since many depend on the equality of variances

The coefficiencies of variation (CVs) for the LABS (excluding LAB 5) were calculated using only those concentrations reported as measured amounts These CVs are summarized in Table 2 showing the minimum, median, and maximum CVs over compounds by lab and sample (Note: there appeared to be little relation between level and the CV, i.e., the CV did not tend to increase or decrease as the individual PCDD/PCDF compound level increased)

Overall, LAB 3 depicted the largest median and maximum values for CVs, but variation among the other LABs also occurred

A comperison of spiked versus found amounts in samples 1 and 2 are compared in Table 3. The percent accuracy was calculated as:

$$\% \text{ Accuracy Mean} = \frac{\text{MEAN}}{\text{SPIKE}} \times 100$$

The minimum, median and maximum percent found by laboratory are listed LAB 3 again had by far the largest ranges Among the other LABS, LAB 2 had the highest maximum values, while LAB 5 had the lowest minimums

Analysis of variance were calculated to determine the sources of variation Generally, the TCDF, PNCDF, and OCDD compounds showed smaller variances, while

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OCDF showed the largest variances. There was some variability among LABS in amount of total variance with LAB 5 having a tendency to be higher. The greatest percent of variation was due to sample differences as reflected in comparing percent sample variance with percent error variance.

For LAB 4, the variance component for extract could be included because three extractions were performed per sample (Table 4). Here as before, at least 95% of the variance was in sample extraction and determination (error) contributing little.

Table 5 provides approximate 95 percent prediction intervals for future analysis of the samples. The LABS were treated as a random sample from a large population of LABS, and a variance component, denoted as VL, of the log concentrations due to LABS was estimated. A measurement error variance component, VM, was also estimated. This component is the within-extraction variance of the log concentrations. The two components were used to calculate the intervals via the following formula:

$$\exp (X \pm 2 V)$$

where X = mean of the log concentration (across LABS and extracts)

$$V = \sqrt{VL + VM}$$

With about 95 percent confidence of being correct, each of the reported intervals is expected to cover a future laboratory's analytical results for the given compound and sample. This assumes that the concentrations are lognormally distributed.

ACKNOWLEDGMENTS

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TABLE 2

MINIMUM, MEDIAN, AND MAXIMUM CVS BY LAB AND SAMPLE OVER COMPOUND

	Sample							
	1	2	3	11	12	13	14	17
MINIMUM								
LAB 1	4.01	3.45	0.83	0.99	1.90	3.25	0.57	1.71
LAB 2	0.00	0.67	1.02	5.83	3.71	4.41	1.24	1.96
LAB 3	11.36	2.55	8.33	9.36	5.13	10.20	6.68	2.16
LAB 4-1	1.25	0.40	0.74	6.97	6.84	6.25	4.48	4.84
LAB 4-2	0.98	0.41	2.04	0.00	0.58	1.75	1.28	0.83
LAB 4-3	0.71	0.96	1.31	0.17	1.55	0.00	0.98	0.34
MEDIAN								
LAB 1	11.78	11.15	3.26	8.63	19.21	15.54	2.86	4.34
LAB 2	4.25	2.25	4.56	14.10	6.62	17.63	12.62	19.22
LAB 3	41.53	13.87	29.82	58.92	23.94	31.23	30.49	22.18
LAB 4-1	4.85	2.44	2.68	13.60	12.22	11.08	12.39	11.94
LAB 4-2	4.00	2.70	5.39	2.08	4.54	7.53	14.65	4.90
LAB 4-3	2.40	3.08	2.21	2.53	4.92	8.24	1.93	2.07
MAXIMUM								
LAB 1	16.06	40.29	5.42	46.96	58.69	33.18	56.57	20.47
LAB 2	16.99	45.81	30.59	45.73	20.24	39.41	46.63	28.49
LAB 3	159.06	86.90	144.88	121.26	56.85	62.39	125.10	53.35
LAB 4-1	26.52	5.30	11.82	32.44	22.05	39.04	28.47	22.04
LAB 4-2	6.53	4.29	30.30	20.80	7.86	24.12	26.89	22.51
LAB 4-3	20.86	7.64	7.11	6.21	27.56	26.91	16.84	8.84

TABLE 3

MINIMUM, MEDIAN, AND MAXIMUM PERCENT FOUND FOR SPIKED SAMPLES 1 AND 2

	Sample 1			Sample 2		
Lab	Minimum	Median	Maximum	Minimum	Median	Maximum
LAB 1	68.0	87.8	105.3	74.7	106.8	135.4
LAB 2	68.0	96.0	124.0	55.3	112.0	149.2
LAB 5	56.0	103.2	116.0	42.4	113.9	120.2
LAB 3	8.2	94.2	3371.4	12.4	120.3	3939.4
LAB 4-1	80.7	98.7	114.5	90.5	100.8	120.8
LAB 4-2	88.0	100.8	113.8	89.4	104.9	119.4
LAB 4-3	66.7	94.5	107.2	90.6	98.7	120.8

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TABLE 4

VARIANCE COMPONENTS BY COMPOUND FOR RADIAN

Compounds	Variance Components				As Percent of Total		
	Total	Sample	Extract	Error	Sample	Extract	Error
Total TCDF	1.40	1.38	0.01	0.01	98.9	0.6	0.5
Total PNCDF	1.62	1.61	0.00	0.01	99.2	0.0	0.8
Total HXCDF	3.66	3.59	0.05	0.01	98.3	1.3	0.4
Total HPCDF	5.13	5.00	0.09	0.03	97.6	1.8	0.6
OCDF	7.73	7.41	0.30	0.02	95.9	3.9	0.2
Total TCDD	2.25	2.25	0.00	0.00	99.9+	0.0	0.0
OCDD	0.00	0.00	0.00	0.00	99.9+	0.0	0.0
2,3,4,8-TCDD	2.25	2.25	0.00	0.00	99.9+	0.0	0.0
2,3,7,8 + 2,3,4,8-TCDF	1.48	1.47	0.00	0.00	99.5	0.2	0.3
1,2,3,7,8 + 1,2,3,4,8-PNCDF	1.84	1.82	0.01	0.01	99.2	0.4	0.4
2,3,4,7,8-PNCDF	1.31	1.30	-0.00	0.01	98.9	0.0	1.1
1,2,3,4,7,8 + 1,2,3,4,7,9-HXCDF	5.80	5.80	0.00	0.00	99.9	0.0	0.1
1,2,3,7,8,9-HXCDF	4.73	4.71	0.01	0.01	99.5	0.2	0.3
1,2,3,6,7,8-HXCDF	2.73	2.69	0.02	0.01	98.6	0.8	0.5
2,3,4,6,7,8-HXCDF	2.25	2.16	0.08	0.01	96.1	3.4	0.5
1,2,3,4,6,7,8-HPCDF	3.92	3.87	0.02	0.03	98.7	0.5	0.7

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TABLE 5

APPROXIMATE 95 PERCENT PREDICTION INTERVALS BY COMPOUND AND SAMPLE

Compound	Sample 1	2	3	11	12	13	14	17
Total TCDF	144 222	57 67	1046 2321	57 362	37 321	389 1247	532 1389	249 729
Total PnCDF	206 431	220 517	1004 1682	1125 3079	143 590	179 442	5846 13471	562 1999
Total HxCDF	155 1862	40 2118	798 10654	3041 21535	726 1320	11 198	5897 21657	610 13977
Total HpCDF	103 1105	137 1532	819 4341	1581 41116	1467 3090	2 52	1391 3483	242 11063
OCDF	2 89	67 208	2280 6436	9641 64743	3822 10453	7 75	96 457	442 24465
Total TCDD	6 60	40 60	63 132	<1 21	<1 14	<1 15	<1 20	<1 10
OCDD	1 59	1 40	2 32	7 19	5 30	<1 70	1 43	5 32
2,3,7,8-TCDD	15 30	40 60	63 132	<1 25	<1 20	<1 13	<1 32	<1 11
2,3,7,8 + 2,3,4,8-TCDF	144 221	57 67	977 1355	24 61	15 81	90 459	251 459	119 241
1,2,3,7,8 + 1,2,3,4,8-PnCDF	123 168	138 193	226 425	41 7913	27 264	8 30	742 1577	74 746
2,3,4,7,8-PnCDF	99 248	90 321	164 618	84 145	28 59	46 80	1324 2389	106 403
1,2,3,4,7,8 + 1,2,3,4,7,9-HxCDF	182 347	1 15	825 1838	2138 12579	447 809	13 36	3144 5860	486 5023
1,2,3,7,8,9-HxCDF	<1 34	<1 25	2 965	1 8869	4 167	<1 61	64 7800	<1 6136
1,2,3,6,7,8-HxCDF	31 72	45 72	254 412	218 603	20 97	2 10	561 1385	43 134
2,3,4,6,7,8-HxCDF	40 1041	24 1912	55 3975	26 879	9 68	1 51	196 2882	10 2237
1,2,3,4,6,7,8-HpCDF	103 1104	137 1530	96 3258	580 16405	418 976	1 17	482 1130	79 5652

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PRODUCTION OF PCDFs AND RELATED PRODUCTS FROM PCBs - AN OVERVIEW*

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PRODUCTION OF PCDFs AND RELATED PRODUCTS FROM PCBs - AN OVERVIEW

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ABSTRACT

In the mid- to late-1970s, several workers demonstrated the formation of PCDFs from PCBs at elevated temperatures in the laboratory. This work went largely unnoticed by those concerned with the scientific, toxicity, regulatory, and product liability issues surrounding PCBs until February 1981 when a fire in a PCB-containing transformer in the Binghamton, New York State Office Building brought "PCB fires" to international attention. Since that event, numerous other PCB fires and related events have underscored the need to "do something." That something has included new regulations from the U.S. EPA and other legal restrictions in various countries, increased removal of PCB-containing electrical equipment from service, financial pressure from insurance companies, much better and faster cleanup and verification of the cleanup, and more research.

The research into the thermal production of PCDFs and related compounds from PCBs has addressed the following areas:

- identification of products formed (e.g., PCDFs, PCDDs, PCBPs, etc.) and their amounts
- research on reaction mechanisms
- determination of matrix effects (e.g., mineral oil, silicone oil)
- studies of effects of conditions (e.g., temperature, residence time, oxygen) and type of event (e.g., fire, arcing)
- study of formation of PCDFs and related compounds from other dielectric fluids (e.g., trichlorobenzenes)

Research has been conducted under both laboratory and real-world conditions. Laboratory experiments have ranged from small, uncontrolled heating in ampules to arcing experiments with actual electric equipment. To examine products formed under real-world conditions, samples from events such as Binghamton have been exhaustively analysed.

In the PCB fire of 1981 in Binghamton, New York, leakage from a PCB transformer was dispersed throughout the 18-story State Office Building (1,2). The average PCB concentration on exposed horizontal surfaces was 162 µg Aroclor 1254/ft^2. Samples of the soot were found to contain PCDDs (20 µg/g), PCDFs (700-2,200 µg/g), and polychlorinated biphenylenes (54 µg/g). Hilker et al. (3) analysed samples from the same fire and found 200 ppm of 2,3,7,8-TCDD. Similar results at several other PCB fire sites were reported by Williams et al. (4-6). The contaminants from Binghamton and other "accident" sites were recently reviewed (1). Table 1 presents a summary of the compounds found at these sites.

Some research has been conducted to identify the products, especially the PCDFs, of incomplete PCB combustion. PCDFs were observed to form on heating in sealed ampules containing PCBs (Aroclors or individual congeners) and oxygen (7). Even at temperatures as low as 300°C, increased levels of PCDFs were observed after 1 week (8). PCDFs are formed from PCBs by at least four reaction mechanisms (7). In addition, secondary reactions can degrade the initial products by dechlorination and other reactions (9). The net result is that the PCDF mixtures formed in PCB fires (and experiments) are complex. The optimum experimental conditions for the PCB-to-PCDF reaction appear to be in the 500-700°C range, with reaction times in the low-second range and excess oxygen present in the atmosphere (10-14).

While most of the attention has focused on the formation of PCDFs and PCDDs from PCBs, other precursors present in utility equipment can also form these products. Chlorobenzenes are 30-40% components of transformer askarels along with the PCBs. Tetrachloroethylene is used as a retrofill fluid and also as a PCB substitute dielectric. The reaction path for these two precursors is more complex than with PCBs (15). The chlorobenzenes must not only acquire an oxygen atom like PCBs, but two "chlorobenzenes" (molecules, free radicals, or derivatives) must collide with sufficient energy to react. In the case of tetrachloroethylene, the reaction pathway is even more tortuous. A series of free-radical condensations must form the benzenoid nuclei, in addition to the three ring PCDF or PCDD structure. In general, the longer the pathway, the lower the anticipated yield would be. Both PCDFs and PCDDs have been observed in laboratory experimental combustion of chlorobenzenes and tetrachloroethylene (16,17). These levels are much lower than the PCDFs observed from PCB combustion under similar conditions.

This presentation will review the work done in the above areas.

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Table 1. Summary of Chlorinated Aromatic Hydrocarbons Detected During Different PCB Accidents[a]

Accident	PCDDs	PCDFs	PCQs	PCQEs	PCTs	PCBPs	PCPYs	PCCYs
Toronto		3 ppm						
Norrtalje, Sweden		45-167 ng/g						
Binghamton, NY	3.6-10.6 ppm	134-3,143 ppm			b	b	b	b
Stockholm (1981)		43-164 ng/g			b	b	b	b
Skovde, Sweden		0.81-8.771 ng/m^3				b	b	b
Yusho, Japan		1-18 ppm	b	b	b			
Yusho, Taiwan		0.16-1 60 ppm	b		b			

[a] PCQs = polychlorinated quarterphenyls
PCQEs = polychlorinated quarterphenyl ethers
PCTs = polychlorinated terphenyls
PCBPs = polychlorinated biphenylenes
PCPYs = polychlorinated pyrenes
PCCYs = polychlorinated chrysenes
[b] Quantification and confirmation not possible due to lack of standards.

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DETECTION OF 2,3,7,8-TETRACHLORODIBENZOFURAN ACTIVITY IN DIELECTRIC FLUIDS BY AN EPITHELIAL CELL CULTURE BIOASSAY

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ABSTRACT

Studies were performed to determine the feasibility of using an in vitro bioassay to detect the conversion of polychlorinated biphenyls (PCBs) to polychlorinated dibenzofurans (PCDFs) in Aroclor based dielectric fluids which were subjected to pyrolysis or combustion. The bioassay is based on polyhalogenated hydrocarbon induced alterations in growth and cellular morphology of epithelial cell cultures which is considered related to PCDF induced chloracne in humans. This endpoint is very sensitive and relatively specific for the more potent PCDFs. Pyrolysis of Aroclor 1254 at 550°C for 15 min results in a 100-fold increase in the bioassay activity, shown by gas chromatographic/mass spectrometric analysis to be correlated to enhanced PCDF levels. Results of testing of six chemically characterized, in-service transformer fluids demonstrated that the bioassay is potentially useful for economical screening of used dielectric fluids and other substances for PCDF-like aggregate activity.

INTRODUCTION

Polychlorinated biphenyl mixtures (PCBs), such as Aroclor 1254, used as dielectric fluid in electrical transformers and capacitors, can be converted to polychlorinated dibenzofurans (PCDFs) as a result of pyrolysis, combustion or electrical breakdown.

PCBs and PCDFs exist in different forms, which vary based on the number and position of chlorination and which determine their biological potency. For example, the congener 2,4,5,2',4',5'-hexachlorobiphenyl constitutes 6.3% of Aroclor 1254 and is converted to 2,3,7,8-tetrachlorodibenzofuran (TCDF), one of the most active PCDF congeners, by elimination of Cl_2. Most other PCBs in Aroclor 1254 are converted to PCDFs of lower biological activity.

A common clinical manifestation of PCDF exposure in humans is the development of chloracne, considered to be the consequence of induced epithelial cell differentiation resulting in hyperkeratinization and subsequent blockage of hair follicles. We have developed an assay based on the PCDF induced alterations of cultured mouse epithelial cell growth and morphology. These changes are considered to be the basis of the development of PCDF induced chloracne in vivo. The studies described in this report demonstrate the feasibility of using this bioassay, referred to as the flat-cell assay, to detect the conversion of PCBs to PCDFs in dielectric fluids and to determine the aggregate biological activity of the accumulated PCDFs in a sample to supplement analytical data.

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METHODS

Chemicals. Sources of compounds tested are described elsewhere (1). Purity was determined to be 99% or greater.

Cell Culture. The XBF cells were derived from the cloned XB mouse epithelial cell line which was a gift from Dr. H. Green, Harvard University (2,3). The derivation and propagation of the XBF line has been described elsewhere (4).

Assay Procedure. XBF cells were suspended by trypsinization and seeded into 24-well plates (16-mm diameter wells, 5 x 10^4 cells/ml) with irradiated 3T3 cells (5 x 10^3/ml per well) in Dulbecco's modified Eagle medium supplemented with 20% fetal bovine serum (FBS). After overnight incubation, the cultures were refed with a series of 10-fold dilutions of a stock solution of test chemical in dimethyl sulfoxide (DMSO) or DMSO alone in DMEM, 20% FBS. The maximum DMSO concentration was 0.1%. The culture medium containing test chemicals was replaced every 3-4 days. After 14 days of incubation, the cultures were evaluated for the alteration of growth and morphology indicative of PCDF exposures as described elsewhere (1) and characterized by a confluent monolayer of flat cells as compared to the higher density, control cultures comprised of multilayered spindle shaped cells.

RESULTS AND DISCUSSION

Baseline determination of TCDF equivalent flat-cell inducing activity was determined for 2,3,7,8-tetrachlorodibenzo-p-dioxin (TCDD), various polychlorinated dibenzofurans (PCDFs), polychlorinated biphenyls (PCBs), and dielectric fluids. The results are summarized in Table 1 and refer to the lowest concentration of a 10-fold dilution series which is capable of inducing the described changes in cell morphology and growth (the flat-cell effect).

TCDD was the most active compound tested and was about 10 times more potent than 2,3,7,8-TCDF; 1,2,3,7,8-PentaCDF; and 1,2,3,4,7,8-HexaCDF. The activity associated with 2,3,4,6,7,8-HexaCDF; 1,2,3,4,6,7,8-HeptaCDF was about 100-fold less than TCDD or one-tenth that of TCDF, while OctaCDF and 2,6-DiCDF were at least 1000-fold less active than TCDD.

The most active PCB tested was 3,4,3',4'-TetraCBP with activity about 10^{-5} that of TCDD, followed by 2,4,5,2',4',5'-HexaCBP with a potency of about 10^{-6} that of TCDD. The other PCBs tested (2,5,2',5'-Tetra, 2,3,4,2',4',5'-Hexa-, and 2,3,4,2',3',4'-Hexa-) had no detectable activity at a concentration of 10^{-7} that of TCDD.

Various Aroclors were tested and the results indicated that 1254 had an activity of about 3 x 10^{-7} that of TCDD. Aroclors 1016, 1242, and 1260 had possibly one-tenth the activity than 1254 although this estimate may be high since at the concentrations tested, cytotoxicity was becoming a factor.

The detection limit for TCDD in various dielectric fluids was then examined. The results indicated that it was possible to detect TCDD at approximately 0.3 ppm in

Aroclors 1254, 1260, and 1242. The maximum concentration of Aroclor tolerated by the assay was approximately 10 ug/ml. Mineral oil and silicon oil were also studied and were shown to be incompatible with the assay due to their insolubility in either the aqueous culture medium or the routinely used dimethyl sulfoxide solvent. Attempts were made to use an emulsifier to generate a stable emulsion of these oils. In the course of these experiments it was discovered that ultrasonication of the silicon oil without the presence of the emulsifier resulted in an emulsion which was stable and compatible with the bioassay. Subsequent preliminary experiments using silicon oil spiked with TCDD indicated that this procedure was comparable to using TCDD in DMSO (the positive control). These studies will continue for silicon oil and mineral oil to verify this result and to optimize the sonication procedure. This procedure could result in a very simple method for preparation of unfractionated oils and other insoluble, lipophilic samples for bioassay testing.

The next study involved testing dielectric fluids which had been subjected to pyrolysis and combustion to determine if the flat-cell assay could detect the conversion of PCBs to PCDFs as shown by chemical analysis. It was found that pyrolysis of Aroclor 1254 at 550°C for 15 min resulted in a 100-fold increase in activity in the flat-cell assay. This reflects the aggregate activity in the sample and is the first demonstration of the PCB to PCDF conversion as shown by a biological system under laboratory conditions. These results indicate that the flat-cell assay can be used to detect the presence of PCDFs in the laboratory simulated "used" Aroclor.

Testing of chemically characterized in-service transformer fluids (Aroclors) by the assay to demonstrate its use as a screen for the accumulation of polychlorinated dibenzofurans was undertaken. The six tested oil samples in hexane were obtained from Battelle (41159-11-12 through 41159-11-17), where they were spiked with various ^{13}C-labelled internal standards at 100 ppb for each component. TCDD equivalents were calculated for each sample based on the analytical results for PCDFs and PCDDs and the biological potency of each isomer and congener in terms of their potency using various biological endpoints (5). This activity, including the contribution of the internal standard, ranges from 130 ppb to 1030 ppb. The results of the bioassay, in a blind test, gave consistent low values of 100-300 ppb for all samples with the exception of sample 41159-11-14 which exhibited relatively high activity of approximately 1000-3000 ppb TCDD equivalents. These results indicate that the flat-cell assay was capable of detecting dioxinlike activity in each of the samples, and was able to distinguish the single sample with relatively high activity. The current results suggest that this biological assay can be used as a screen to test used PCB based dielectric oil for PCDF contamination.

ACKNOWLEDGEMENTS

This work was funded by the EPRI contract RP2028-4.

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MONS 020314

Table 1

INDUCTION OF THE FLAT-CELL EFFECT BY POLYCHLORINATED DIOXINS, DIBENZOFURANS, BIPHENYLS, AND AROCHLORS

	Minimum Detectable Concentration (ppb)
2,3,7,8-Tetrachlorodibenzo-p-dioxin	0.0032
2,3,7,8-Tetrachlorodibenzofuran	0.032
1,2,3,7,8-Pentachlorodibenzofuran	0.025
1,2,3,4,7,8-Hexachlorodibenzofuran	0.025
2,3,4,6,7,8-Hexachlorodibenzofuran	0.378
1,2,3,4,6,7,8-Heptachlorodibenzofuran	0.250
Octachlorodibenzofuran	4.48
2,6-Dichlorodibenzofuran	>2.38
3,4,3',4'-Tetrachlorobiphenyl	100
2,4,5,2',4',5'-Hexachlorobiphenyl	1,000
2,5,2',5'-Tetrachlorobiphenyl	>10,000
2,3,4,2',4',5'-Hexachlorobiphenyl	>10,000
2,3,4,2',3',4'-Hexachlorobiphenyl	10,000
Aroclor 1254	10,000
Aroclor 1016	>10,000[a]
Aroclor 1242	>10,000[a]
Aroclor 1260	>10,000[a]

[a] Possible slight flat-cell induction at this concentration. A concentration of 100,000 ppb was toxic.

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PART B:

RISK MANAGEMENT

PRELIMINARY ANALYSIS OF RISKS ASSOCIATED WITH USE OF PERCHLOROETHYLENE IN TRANSFORMERS

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ABSTRACT

There are acute and chronic toxicity concerns with the use of perchloroethylene (PCE) in transformers. Should a major leak or catastrophic failure of a PCE-filled unit occur, the relatively high vapor pressure of PCE could result in air concentrations high enough to present an acute risk of central nervous system (CNS), pulmonary, heart, liver and kidney effects. CNS effects could become severe enough to make safe escape difficult or impossible and is thus the chief acute hazard/risk concern.

Low-level leaks and exposure to workers conducting routine maintenance operations could present chronic risks of cancer as well as liver and kidney effects.

Risks associated with combustion/pyrolysis products are hard to estimate, since only very limited information has been available on rates and levels of production of these materials. Hexachlorobenzene is of particular concern in this regard.

INTRODUCTION

Polychlorinated biphenyls have been used as dielectric fluids in electrical power transformers and capacitors for decades because of their combination of good electrical properties and fire resistance. The toxicity of these chemicals and of their combustion/pyrolysis products, however, has required that substitutes be found for this application.

Among the liquids that have been found to possess electrical and physical properties useful for dielectrical fluid applications is PCE. However, PCE, like polychlorobiphenyls, possesses properties which result in some level of risk when the compound is used in electrical equipment. This paper is a preliminary look at those risks.

FINDINGS

Risk estimates presented here are based on health effects information contained in Health Assessment Document for Tetrachloroethylene (Perchloroethylene) (1) and its Addendum: Updated Carcinogenicity Assessment for Tetrachloroethylene (2). Levels of PCE in air anticipated to cause toxic effects, particularly CNS effects, were taken from the Health Assessment (1) and combined with assumptions about office building design (3) to estimate leak rates from PCE-filled transformers that could result in

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toxic effects. In addition to CNS effects, plausible, upper-bound cancer risk levels of 10^{-5}, 10^{-3}, 10^{-4} and 10^{-3} were assessed using this method. Risks associated with PCE combustion/pyrolysis products could not be estimated.

In an office building of 28,400 m^3 volume which has one air exchange per hour, a leak rate of 25 grams/hr would pose an extra cancer risk (plausible upper-bound) of 10^{-4} to an office worker, using the usual assumptions about the worker. A transformer containing 900 kg. of PCE and leaking at 25 grams/hr would lose 10% of its fluid in about 4 months.

For the same building parameters, a leak of 38,600 grams/hr would be required to produce PCE levels of 200 ppm (assuming instantaneous vaporization and equilibration), levels high enough to make safe escape from the building difficult for some people. Depending on containment failure conditions, instantaneous vaporization and equilibration may or may not occur. Thus, some parts of such a building would contain much higher levels of PCE while other parts would contain lower levels. In other words, under conditions of such a major leak, some parts of a building could become loaded with dangerous levels of PCE rather quickly.

Since hexacholorobenzene (HCB) is a broad spectrum, potent toxicant, its possible by-production in PCE-filled transformers could have serious implications for disposal of used fluid and used transformers. There are experimental data supporting this concern (4).

CONCLUSIONS

In summary, use of PCE-filled transformers in buildings that have no provision for securely isolating them from general air circulation could pose a danger to people trying to escape if a major breach of fluid containment occurs. Further, HCB may be by-produced in PCE-filled transformers, and units that have been in use for some years could be tested for its presence.

DISCLAIMER

This report has not been peer or administratively reviewed and should not be construed as final policy of the U. S. Environmental Protection Agency.

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MANAGING PCB RISKS IN POWER PLANTS

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ABSTRACT

Should PCB transformers in power plants be replaced? Even if health risks are minimal, PCBs can present economic risks to utilities. An incident involving PCBs can result in millions of dollars in cleanup, replacement power, and legal costs. On the other hand, incidents are very unlikely, and replacing or retrofilling transformers can be very expensive. PCB equipment management decisions are difficult because incident occurrence, severity, and cost are all highly uncertain. The PCB Economic Risk Management Model (ASK), developed for EPRI by Decision Focus, helps utility personnel balance the certain costs of replacement or retrofilling against the uncertain costs of keeping PCB equipment in service. ASK has been used successfully by more than a dozen utilities throughout the United States to help manage PCB transformers.

THE ASK MODEL

The PCB Economic Risk Management Model (ASK) is a decision support tool designed to help utility personnel manage equipment containing PCBs. Based on the methodology of decision analysis, the model allows utility personnel to compare alternative strategies on the basis of overall costs, including both equipment costs and the costs of potential incidents such as fires or spills. ASK allows model users to explicitly represent uncertainty regarding the occurrence, severity, and cost of incidents, and to explore the sensitivity of management options to changes in assumptions. These features are combined in an interactive software package that is designed for easy use on microcomputers.

Management decision and uncertain events are modeled in ASK using decision tree to organize a set of scenarios. For each management strategy the probability of a given scenario is simply the product of the individual outcome probabilities for each uncertain event in the tree.

For each scenario defined by the tree, incident and equipment costs are calculated using the models shown in Figure 1. The equipment model calculates all costs associated with existing and replacement equipment. The incident occurrence model calculates the likelihood, expected timing, and probable severity of an incident. The incident cost model calculates all costs of an incident, given that one occurs. Cost elements can include equipment replacement costs, cleanup and repair costs, costs of legal liabilities, and replacement power costs if the plant is shut down during the cleanup and repair effort. The financial model adds up all the cost elements and computes the discounted expected value of the scenario.

Weighting the cost of each scenario by its likelihood, ASK can determine the expected cost of each equipment management alternative. In addition to the most likely cost, ASK provides information regarding the cost ranges possible under each management strategy.

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DATA REQUIREMENTS

The data requirements of ASK are modest. In order to evaluate an equipment management alternative, the user needs to supply basic information about the existing equipment including its remaining useful life and disposal cost. The cost of new equipment is also required, along with cost of any risk reduction measures that are part of the strategy.

Estimates of incident frequency can be obtained from industry average data. Incident severity is usually characterized sufficiently by three possibilities: spills, arcing and smoke with no fire, and major fires or explosions. Associating appropriate cost estimates with each severity level must be done on a case-by-case basis, although the breakdown of costs into cleanup, replacement power, and legal liability components helps make the estimates manageable. Again, industry experience can provide valuable benchmarks, especially when estimating the cost of severe incidents.

ASK will provide formatted data tables, allowing convenient review and providing clear documentation of the assumptions underlying the analysis.

RESULTS

ASK produces four basic results tables, including summary results, detailed results that break down overall expected cost into the equipment and incident components, expected incident costs for each severity level, and cost ranges. Table 1 illustrates the detailed results table from a typical analysis. The results can be used to identify and communicate the least-cost option, as shown in Figure 2.

Using ASK interactively, extensive sensitivity analyses can be easily performed. Often, this information is most effectively communicated on a graph. Figure 3 illustrates a typical format. The horizontal axis represents the overall incident frequency; the vertical axis the duration of the shutdown resulting from a major fire in a PCB transformer. In the lower region of the graph, retention of the existing equipment has the lowest expected cost. In the upper region, retrofilling is the preferred option. The solid line through the center of the plot represents the combinations of incident frequency and fire shutdown duration that result in equal expected costs for the retention and retrofilling alternatives. The base case data combination is identified in he lower left-hand corner of tha graph. The distance of the base case data point from the equal cost line is a measure of the robustness of the analysis results.

SUMMARY

ASK provides a powerful tool for utility personnel charged with evaluating PCB equipment management alternatives. Development of a base case data set can often be completed within days. Experience by utilities using the model indicates that, in most cases, well-documented and defensible analyses can be planned, performed, and provided to management within several weeks of the initiation of the analysis.

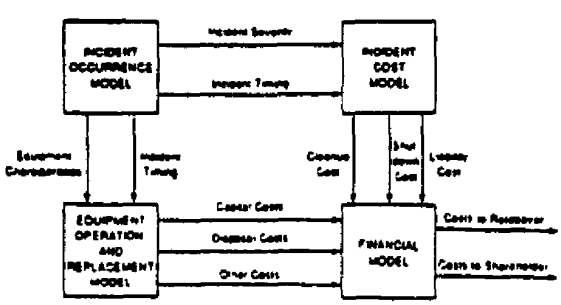

Figure 1. The Set of Models Used to Calculate Costs in ASK and Coil

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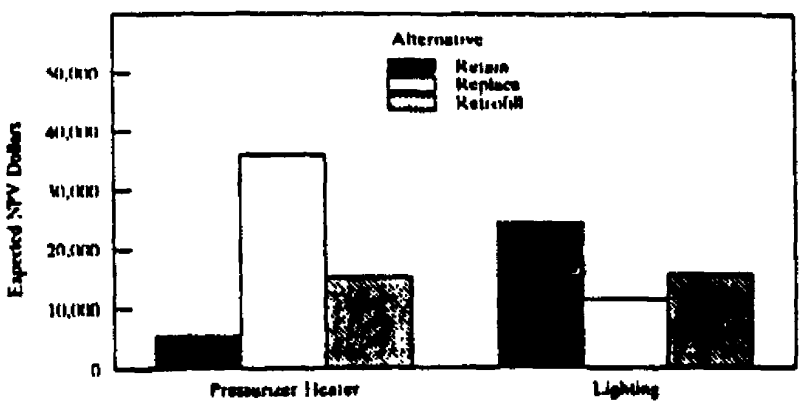

Figure 2. Summary of Transformer Management Options

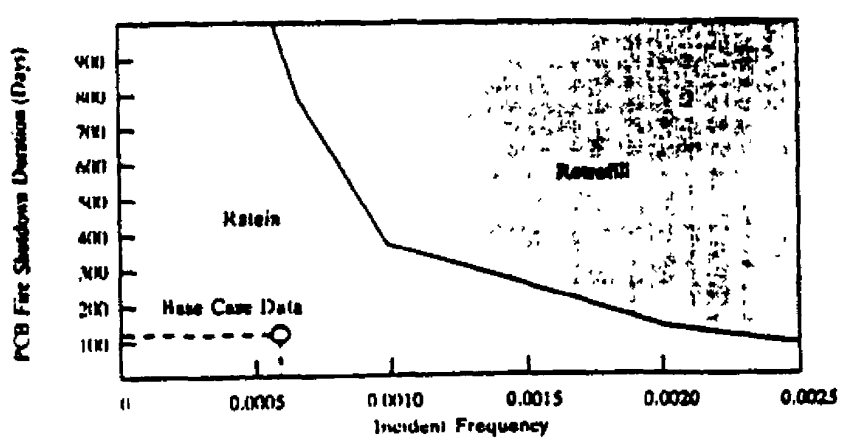

Figure 3. Sensitivity Analyses Can Be Presented Graphically

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Table 1

ASK DETAILED RESULTS TABLE

DETAILS OF EXPECTED COSTS
(* INDICATES BEST ALTERNATIVE)

ALTERNATIVE	EQUIPMENT COSTS	EXPECTED INCIDENT COSTS CLEANUP	SHUTDOWN	LIABILITY	TOTAL COST
Retain		PROBABILITY OF INCIDENT =.019612			
RATEPAYERS	285.	16089.	7417.	541.	
SHAREHOLDERS	0.	0.	0.	0.	
TOTAL	285.	16089.	7417.	541.	24333.
*Replace Immed.		PROBABILITY OF INCIDENT =.019612			
RATEPAYERS	11345.	108.	0.	0.	
SHAREHOLDERS	0.	0.	0.	0.	
TOTAL	11345.	108.	0.	0.	11453.
Retrofit		PROBABILITY OF INCIDENT =.019612			
RATEPAYERS	12115.	2608.	751.	55.	
SHAREHOLDERS	0.	0.	0.	0.	
TOTAL	12115.	2608.	751.	55.	15528.

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PCB RISK ASSESSMENT USING THE ASK MODEL

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PCB incidents (spills, fires, etc.) are rare, but the potential costs from an incident can be quite large. The likelihood, severity, and cost of an incident are highly uncertain. Consequently, the equipment management decisions facing electric utilities are challenging.

A study of the risks posed by PCB equipment at Virginia Power's generating plants was performed using the PCB Economic Risk Management Model (ASK). Probability inputs to the model were obtained from the use of a risk ranking worksheet. The results presented management with a life-cycle cost for each alternative course of action and allowed management to compare its own risk standards to a "break-even" probability.

INTRODUCTION

Virginia Power, in January 1984, began a PCB removal program designed to eliminate all PCB and PCB contaminated equipment from the transmission and distribution system. However, the Power Operations Department had no program in place at the power stations that mandated equipment removal or other risk reduction measures. In late 1985 a Power Operations staff group initiated a study to assess the economic risks posed by PCB equipment at the generating facilities. Performance Services, a technical service group within Power Operations, was chartered to use the PCB Economic Risk Management Model (ASK) to evaluate the risks and to present the findings to management in a concise format.

ASK is an integrated decision support tool incorporating equipment cost and performance calculations, a financial model, and explicit representation of uncertainties in the occurrence, severity, and cost of incidents. The model runs on a personal computer and the software has been designed to facilitate input and analysis. The output of the model presents the expected value of the life cycle cost for each alternative.

The model requires the user to specify the management alternatives under consideration and the branches for each of five areas of uncertainty. These five areas are: (1) incident frequency, (2) incident severity, (3) cleanup costs, (4) plant shutdown costs, and (5) liability costs.

Equipment data to be input to the model for existing equipment include the original capital cost, KVA rating, power factor, capacity factor, O&M cost, disposal cost, useful life, tax life, book life, and remaining life. The information for the replacement equipment includes the capital cost, O&M cost, power factor, and risk reduction cost (retrofill, curbing, etc.) in expensed or capitalized form.

ESTIMATING PROBABILITIES

Perhaps the most difficult input to develop is the probability of an uncertainty branch. Sensitivity analysis indicated that the annual incident occurrence rate had a significant effect on the model output. During initial model runs, the incident frequency uncertainty was represented by three branches with different values for the incident rate and varying probabilities based on the condition of the equipment. Industry data indicate that the current physical condition of the equipment (age, casing condition, etc.) may not correlate with the potential for failure. Therefore, the incident occurrence rate section was changed to one branch. The value of this branch is .001 incidents per year. This figure represents the industry average based on a number of studies by different groups and implies that out of one thousand transformers, there will be one incident. This does not speak to the magnitude of the incident; it could be a spill, large or small, an arcing incident producing soot, or a catastrophic incident such as fire or explosion.

Three branches were chosen to represent the uncertainty involved with the severity of an incident. These were: (1) PCB spill, (2) PCB arcing/smoke, and (3) PCB fire/explosion. The probabilities assigned to each branch were .95, .04, and .01 respectively. The probabilities were chosen as a result of an analysis of industry data. These branches were applied to the "retain alternative". The study did not assign severity branches to the retrofill and replace options because it was assumed that no PCB incidents will occur under those alternatives.

Representing the uncertainties of cleanup costs, shutdown costs, and liability costs is more of a challenge. Three branches (minimum, moderate, and maximum) were chosen for each uncertainty. The value assigned to each branch is discussed later.

The probabilities were chosen in a manner based on a relative ranking of each piece of equipment. This ranking, the Incident Severity Ranking, was a result of field inspection of each item using an Incident Risk Ranking Worksheet (adapted from EPRI Document CS-4094: Design Manual for Reducing Airborne PCB Contamination). The study assumes that the magnitude of the costs of an incident, providing an incident occurs, is related to the Incident Severity Ranking of the piece of equipment in question.

The probabilities for the branches representing cleanup costs were chosen by using the graph in Figure 1. This graph was constructed by recognizing that if the Incident Severity Ranking was low, and an incident occurred, there is a greater probability that the associated cost will be on the low side. For example, if the equipment had a ranking of 15 the probability of a minimum cost would be 56%, the probability of moderate cost would be 32%, and the probability of maximum cost would be 12%. By using this graph with the Incident Severity Ranking, the process achieves a relative risk comparison between PCB items.

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The probabilities for the branches representing the shutdown costs and liability costs were selected in the same manner with one modification. A Shutdown Factor and an Exposure Factor were added, separately, to the Incident Severity Ranking, and those sums were used to select probabilities from the graph. These factors were used to account for individual characteristics of the different PCB items that indicated one may be more prone than another to cause a shutdown or to expose personnel to contamination.

VALUES FOR BRANCHES

The study assigned values for the minimum, moderate, and maximum branches of the cleanup cost, shutdown cost, and liability cost uncertainties. The variance from item to item will occur in the associated probability, not in the value of the branch. The cleanup cost values were based on industry experience for various incidents. The liability cost values were based on lawsuit figures from actual incidents, but were modified to show only the awarded portion of the suits. The shutdown costs are computed by the model based on the expected value of the shutdown duration, the MW rating of the unit, and the capacity factor of the unit.

RESULTS

The results were presented in tabular form giving the life-cycle cost for each alternative and a "break-even" probability for each situation. This break-even probability is the point at which the life-cycle cost of the retain option is equal to the life-cycle cost of the next best option: retrofill or replacement. This is a numerical representation of the intersection of the lines on the graph in Figure 2. The graph is shown to illustrate how management may compare the results to their own risk standards. One should note that the probability that an incident will occur is given by

$$PROB = 1 - (1-p)^t$$

where: t = Remaining life of equipment
p = Annual incident occurrence rate

The study was effective in focusing management's attention to PCB risks. Several power stations are now in the process of replacing or reclassifying their PCB equipment. We have since used the ASK program on several occasions for "what-if" analysis and have also re-run certain scenarios using updated information on cost and life expectancy.

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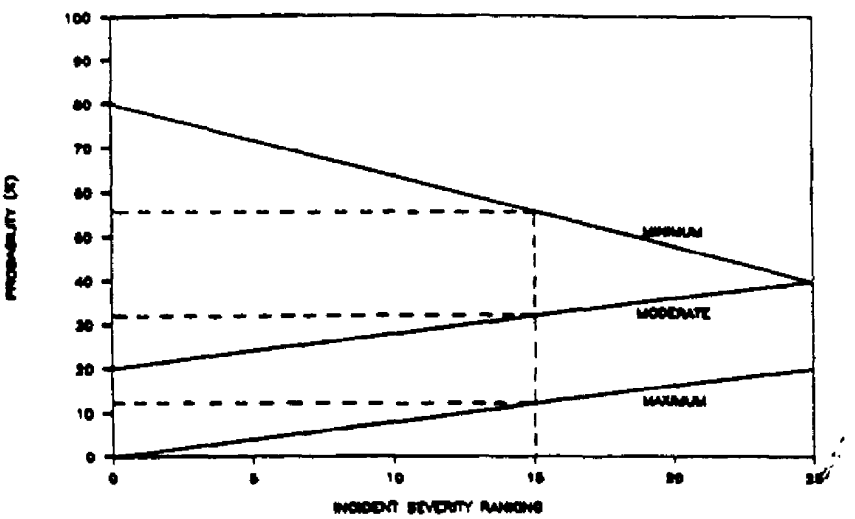

Figure 1. Probability Selection Graph

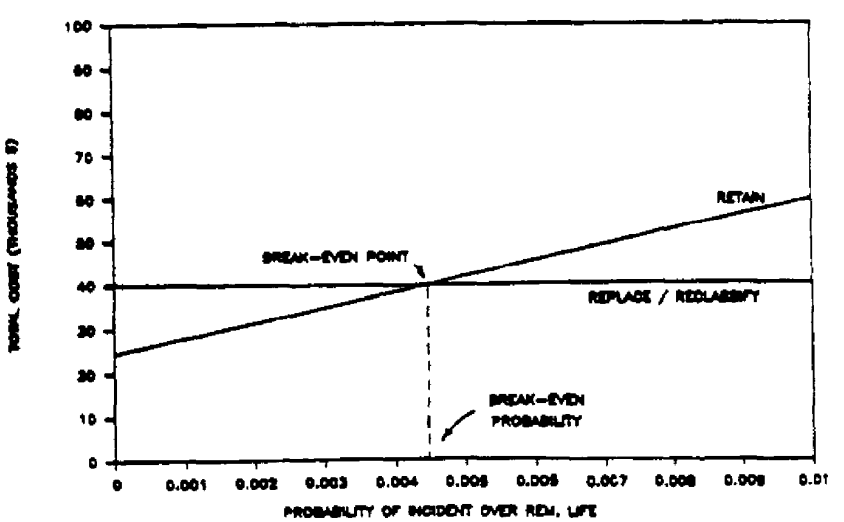

Figure 2. Incident Probability vs Total Cost

MONS 020327

RISK ANALYSIS OF USING PCB FILLED TRANSFORMERS IN INDUSTRIAL AND RESIDENTIAL APPLICATIONS.

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ABSTRACT

This paper describes the application of the Management Oversight and Risk Tree (MORT) method for the analysis of the hazards which may occur due to the use of PCB filled transformers exposed to fire, as this may yield in specific circumstances the production of dioxins (PCDD) and furanes (PCDF). The method reveals a number of measures which have to be taken in order to avoid the production of these extremely toxic products.

INTRODUCTION

Although the Belgian law (Royal Decision dated July 9, 1986) forbids the selling of new systems containing PCB's, it allows the use of existing equipment as long as its normal lifetime. As static electric equipment as e.g. transformers and capacitors has a typical lifetime of 30 years and more, the problems and hazards coming from the use of these products will still be present for a long time, especially when the equipment is exposed to fire, yielding the creation of extremely toxic products like dioxins (PCDD) and furans (PCDF).

As shown by various examples in the past [1,2,3], this might lead to dangerous situations and to major economic damages. Therefore the authors used a systematic approach, called MORT (Management Oversight and Risk Tree) method to analyse the risks due to the use of PCB filled transformers.

The major features of the MORT analysis are discussed briefly.

MANAGEMENT OVERSIGHT AND RISK TREE (MORT)

MORT is a systamic approach to the management of risks in an organisation. It was developped by the U.S. Department of Energy (DOE) and incorporates ways to increase reliability, to assess risks, to control losses, and to allocate resources effictively.

It is conceived for decreasing the safety-related losses of an organization with an already excellent safety record. Its philosophy can be summarized by the following points [4]:

1. Management takes risks of many kinds.

2. Risks in one area affect operations in other areas.

3. Risks should be made explicit where practicable.

4. Risks management tools should be flexible enough to suit a variety of diverse situations.

The various interections in the system which may yield the undisered dangerous phenomenon, are represented using logical analytical diagrams (trees).

The full MORT approach is a "universal tree" developped for an antire safety system discipline. As the system discussed here, is relatively simple when e.g. compared to a chemical plant, a lot of the faatures are not used here.

Figure 1 represents all the grephic systems needed for the analysis.

We will now discuss the application of the MORT analysis for the creation of PCDD and PCDF from PCB used as an insulator in a transformer.

USE OF THE MORT ANALYSIS FOR A PCB FILLED TRANSFORMER

With respect to the problems which may occur when using PCB filled transformers in industrial and residential areas, two major risks have two be analysed:

- the leaking of PCB out of the transformer and the direct pollution caused by it;
- the generation of dioxines and furanes when PCB is burned during a fire.

Both situations were analysed and will be discussed during the seminar. However only the second one, which is by far the most dangeroue, will be explained here briefly.

Figure 2 shows the risk tree. As we can see, a number of conditions have to be satisfied in order to generate to toxic products. A lot of electrical as well fire protection measures have to be less than adequate (LTA).

The detailled discussion of the various elements of the tree will be presented at the seminar.

Therefore we may conclude that a lot of protective measures must be taken care of and must be checked regurlarly in order to avoid major disasters when using PCB filled transformers.

ACKNOWLEDGMENT

The authors are mostly grateful to the Council of the Joint Basic Research Fund (F.K.F.O.) of the Belgian Science Foundation (N.F.W.O.) for granting a project that financed this research.

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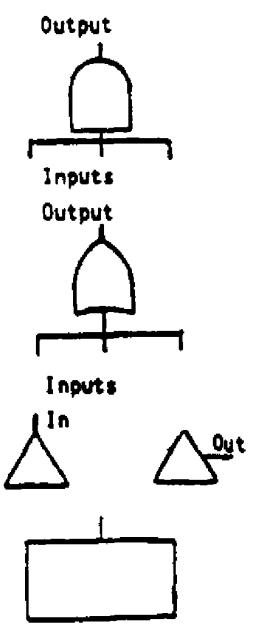

AND-gate: Coexistence of all inputs required to produce output

OR-gate: Output will exist if at least one input is present

TRIANGLE: A connecting or transfer symbol. All tree construction below the "out" triangle is transferred in at "in" triangle location(s).

RECTANGLE: An event resulting from the combination of more basic events acting through logic gates

Figure 1. Basic symbols of the MORT analysis

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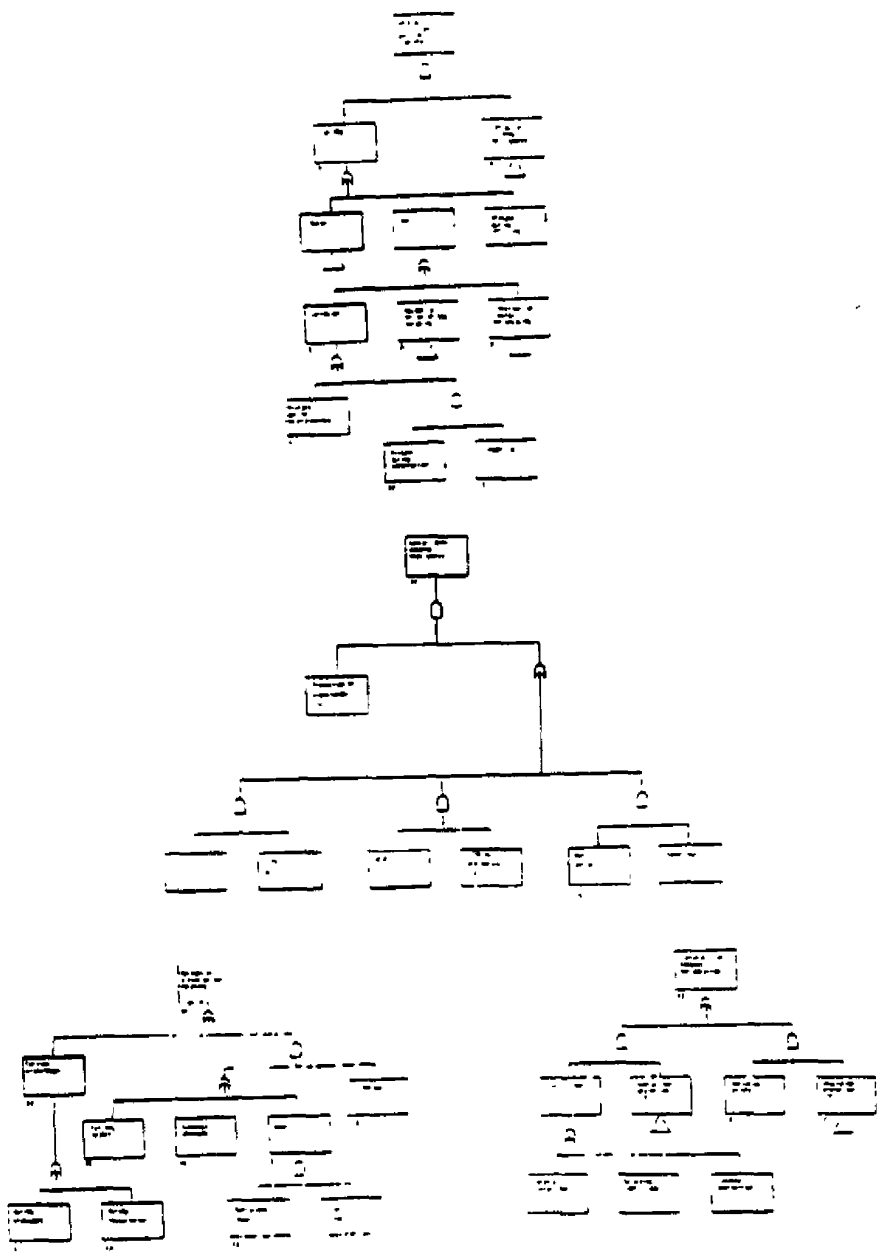

Figure 2. MORT analysis of generation of PCDD and PCDF from PCB's

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PART 9:

DECONTAMINATION OF OIL

"GE's Non-Sodium Process For Chemically Decontaminating Mineral Oil Dielectric Fluid"

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ABSTRACT

As early as 1979, scientists at GE's Corporate Research and Development Center, Schenectady, NY, recognized that PCB contaminated transformer dielectric fluid could be chemically treated to reduce PCB content to non-detectable levels. This led GE to develop and patent a chemical decontamination process in 1981 using metallic sodium. Because of the potential difficulties in handling sodium (sodium and water produce hydrogen which can be explosive), GE embarked on a research program aimed at identifying a safe, economic means of reacting PCBs in transformer oil that would reduce PCB levels to below the U.S. Environmental Protection Agency (EPA) 2 part per million (ppm) upper limit for chemical destruction. To be successful, this process would be safe, efficient and economical and would not require the use of sodium.

This paper describes the successful development by GE of a patented non-sodium process meeting these objectives and the design of a trailer mounted PCB decontamination unit for commercial use utilizing the new advanced GE chemistry.

THE PCB REGULATORY ENVIRONMENT

Polychlorinated biphenyls (PCBs) are a class of hydrocarbon compounds that have long been used as dielectric (insulating) fluid in electrical equipment. The majority of liquid filled transformers in service today use either mineral oil or PCBs as their insulating fluid. While both fluids were used separately in their intended application, many mineral oil-filled transformers were contaminated with PCBs during manufacturing or servicing. While the estimated number of contaminated units in service varies, there are at least 2.5 million transformers contaminated with more than 50 ppm PCBs.

In 1976, Congress enacted the Toxic Substances Control Act (TSCA) as a response to public concern over hazardous waste. On May 31, 1979, EPA published the PCB "Ban Rule" regulating fluids containing over 50 ppm PCBs. Since promulgation of the "Ban Rule", EPA has issued many additional regulations, addressing PCB related issues and defining various uses and restrictions of PCB fluid and equipment containing PCBs.

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CHEMICAL DESTRUCTION OF PCBs IN MINERAL OIL

EPA disposal regulations require destruction of PCB dielectric fluids, by thermal or "alternative" destruction methods. Thermal methods include high temperature incineration and destruction in "high efficiency boilers".

Although a number of "alternative destruction methods" have been tested, this category principally includes PCB chemical destruction systems.

Most of the currently permitted sodium chemical destruction systems can decontaminate fluid containing PCBs up to 5,000-10,000 ppm. However, because chemical costs increase with the PCB level treated, sodium based chemical systems are usually economic up to about 3,000-5,000 ppm. Beyond that level, incineration becomes more economical when transportation costs are equal. Besides being economical for the low contamination levels, chemical decontamination systems can be trailer mounted. Additionally, these systems produce a non-PCB fuel oil with a high BTU content.

Because of the above advantages, many owners are now selecting chemical destruction as the preferred method of destroying PCBs in mineral oil.

THE GE PROCESS

GE found that PCBs in transformer oil react with polyethylene glycol (PEG) and potassium hydroxide (KOH) under relatively mild operating conditions. Destruction of PCBs occurs by mixing heated transformer oil with small amounts of PEG and KOH in a stirred vessel. Both PEG and KOH are non-proprietary chemical substances commonly used in industry and do not pose any unusual risks. Complete reaction of PCBs occurs quickly producing decontaminated transformer oil and a non-PCB byproduct. The small quantity of byproducts produced are insoluble in transformer oil and are easily removed from the decontaminated oil by decanting. Figure 1 schematically depicts the GE process in simplified form.

While sodium based processes destroy PCBs by sequentially stripping away chlorine atoms from the biphenyl molecule over time, GE's process destroys PCBs as a result of a simple chemical substitution of a glycol atom (from the PEG) for a chlorine atom in the biphenyl system [Figure 2]. While multiple substitutions of chlorine atoms may occur, with the GE process only a <u>single</u> substitution is needed to render the PCB product molecule insoluable in the transformer oil. This non PCB product can be easily removed from the oil by simple decanting. The non PCB byproducts can be incinerated and the decontaminated oil used as fuel.

Because of the simplicity of the process, PCB destruction is fast, efficient and highly predictable. Because the oil and chemicals are easy to handle, a trailer-mounted PCB decontamination unit can be built using conventional and proven process equipment. Because metallic sodium is not used, there is no possibility of explosive hydrogen production.

Unlike the sodium based processes, the GE process is not particularly sensitive to water. Additionally, it is not necessary to precisely measure the amount of PEG and KOH added to the oil.

Therefore the GE process offers significant advantages over sodium based process (Table 1).

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LAB EXPERIMENTS AND EVALUATION

Commercial development of the GE process began in 1981 with a series of laboratory bench scale experiments using a variety of process conditions and reagents.

Following the extensive experiments which proved the effectiveness of the decontamination chemistry (PCBs were destroyed to less than 2 ppm per EPA's requirements), an engineering analysis was conducted to establish the feasibility of commercializing the process; to evaluate the effects of using different process parameters; and to obtain scaleup and design data.

As a result of this laboratory evaluation, GE concluded that a safe, economic, simple and efficient method of destroying PCBs in mineral oil had been achieved without using sodium. PCBs were destroyed in a simple reaction under mild operating conditions. The non-PCB reaction products were easily separated from oil.

DEVELOPMENT OF A TRAILER-MOUNTED PCB DECONTAMINATION UNIT

Based on the success of the laboratory experiments, GE commenced process design and engineering of a PCB decontamination system using the advanced GE chemistry.

Prior to beginning design of a full scale commercial unit, GE established strict design criteria that would ensure proper operation and safety of the unit consisting of:

- Maximized Safety Features
- Failsafe Design
- Computer Controls
- Maximized Daily Throughput
- Meeting EPA Performance Objectives
- Onboard PCB Laboratory
- Online Data Acquisition System
- Proper Materials of Construction
- Use of Conventional Equipment
- Ease of Operation

Using the basic process configuration proven in the lab, GE has designed a trailer-mounted PCB decontamination unit [Figure 3] with three (3) vessels operating in a batch type process. Batch processing was selected over continuous because of its operating simplicity, higher reliability and failsafe features. The simplicity of the chemistry enabled GE to select commercially available components and to provide for automated operation using GE's "Series Six™" programmable controller. The "Series Six" automatically controls the chemical process from the control room and provides the operator with continuous on-line process information and periodic status reports for critical process parameters.

Figure 4 provides a simplified process flow schematic of the system. PCB contaminated oil from storage is pumped through a filter to the effluent heat exchanger where the incoming oil is partially heated by exchanging heat from the previous batch of decontaminated oil. In the electric heater the oil reaches the reaction temperature before entering one of the three (3) reactors.

KOH added to the feed hopper is fed in the required amount to the reactor via the KOH feeder. PEG from the feed tank is metered and pumped to the reactor where the oil, PEG and KOH are mixed for the required reaction period. The reactor is blanketed with nitrogen during the reaction. Any excess nitrogen and vapors are passed through an activated carbon bed. In this way, any hydrocarbons are absorbed in the carbon bed.

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The reactor contents are pumped through the heat exchanger, thereby heating the next incoming batch of oil. From the heat exchanger, the decontaminated oil and reaction products are pumped to a decanting tanker where the oil and reaction products are easily separated.

The entire sequence of filling, reacting and dreining of the three (3) reactor loops is accomplished automatically with the onboard microprocessor which also provides the operator with status reports and alarm.

All equipment and components are mounted in a specially designed 48 foot long air ride trailer which includes separate process and operational lab areas.

Design of the equipment and trailer includes many safety featuras such as an integral spill pan, air ride suspension, halon fire protection system, trouble alarm system, automatic system shutdowns, emergency exits, safety shower, eye wash and air emissions control system.

SITING AND OPERATING CONSIDERATIONS

The GE decontamination unit is designed to be operated in multiple shifts either at a fixed location or in e mobile environment.

Operation of the decontamination unit requires two operators, one of whom is skilled in PCB product analysis. Prior to and following decontamination, the oil and products are tested for PCB content. These tests are performed using the onboard electron capture gas chromatograph.

GE will operate the decontamination unit with the highest degree of safety and in accordance with a strict risk control program.

SUMMARY

GE has developed and proven, through extensive laboratory experiments that the GE decontamination process is a safe, economic and reliable method of chemically decontaminating mineral oil containing PCBs. PCB levels are reduced to less than 2 ppm thereby meeting EPA's destruction requirements. The GE process offers a number of significant advantages over sodium-based processes including:

- No Chance of Hydrogen Production
- Insensitivity to Water in the Oil
- Precise Amount of Reagents Not Critical
- Fast Turnaround

Laboratory and scaleup tests have shown that the process can effectively decontaminate oil containing up to 10,000 ppm PCBs.

The simplicity of the GE process supports the use of conventional chemical processing equipment in a trailer-mounted unit with large volume of PCB destruction capacity.

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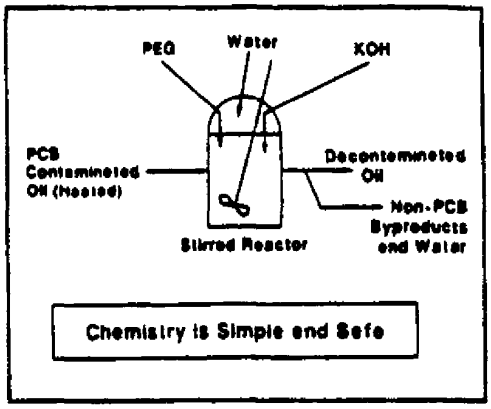

Figure 1. GE Process (Simplified Diagram)

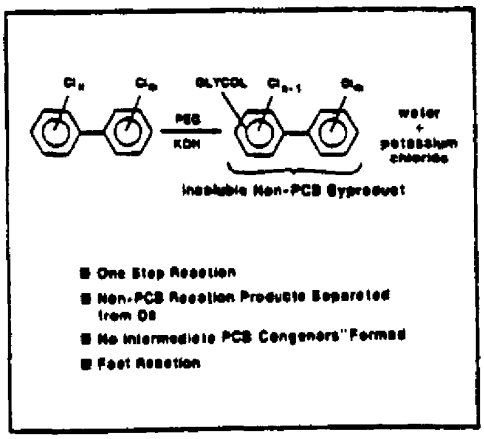

Figure 2. Destruction by GE Process

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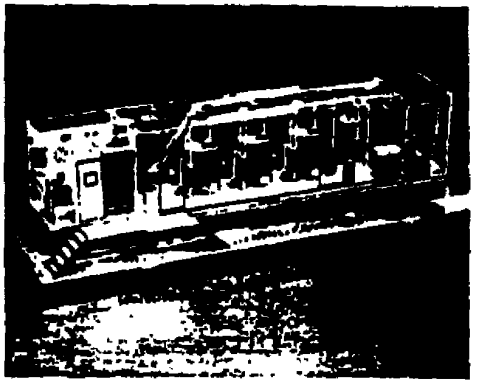

Figure 3. PCB Decontamination Unit Model

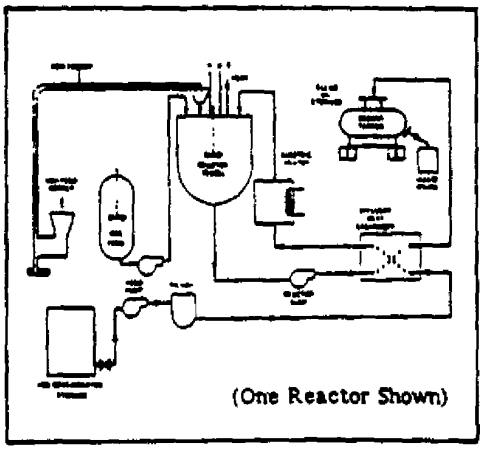

Figure 4. Simplified Process Schematic
GE PCB Decontamination System

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Table 1

Advantages of the GE Process

- Simple Chemistry
- Fast ⟶ Efficient
- Highly Predictable Outcome
  (No Intermediate PCBs Formed)
- Non Proprietary Reagents
- Cost Effective
- No Sodium Used (No Hydrogen)
- Mild Operating Conditions
- Not Sensitive to Water
- Conventional Process Equipment Used

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THE VALUE OF DEHYDRATION PRIOR TO RETROFILL OF LARGE PCB-CONTAINING TRANSFORMERS

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ABSTRACT

Field retrofill activities are highly desirable to reduce PCB contamination and to ensure prolonged transformer life. Particularly in larger transformers, an operating constraint (water build-up) which can limit or even curtail the success of retrofill must be eliminated prior to retrofill in order to ensure prolonged safety of transformer performance and life. A particularly well-established procedure involving the use of kerosene in conjunction with a well-designed vacuum dehydration apparatus allows virtually complete and totally safe removal of water build up. This paper will detail this method.

INTRODUCTION

As might be expected, the PCB situation in Puerto Rico is much the same as in the US. In this respect, Puerto Rico may be considered a microcosm of the larger PCB world in the US (as a Commonwealth, Puerto Rico is covered by US laws and regulations). Although the numbers of transformers containing PCBs is smaller- roughly 1500 PCB containing transformers- the threat posed by the presence of PCBs is no less visible, real or apparent to the populace. Consider the situation which would arise if a PCB spill or incident (similar to and even smaller than that of Waukegan Harbor or the Hudson River) should occur on an island which relies solely on ground or surface water for water. The results would be catastrophic, whether politically, economically or environmentally. The situation would be compounded by the fact that the water table on an island is, ecologically, so fragile.

Recently, the island narrowly missed a PCB transformer fire incident which would have rivaled the incident at the State Office Building in upstate New York. On New Years Day, 1986, a fire in a luxury hotel in San Juan gutted most of the ground and the first floor of the structure, leaving the remainder structurally sound. Fortunately, here the parallel with the New York situation ceased. The fire very narrowly missed large PCB transformers located on the lower floor, whose involvement would have compromised the entire hotel.

APPROACHES TO THE PROBLEM

As in the US, there are two approaches for removal of the PCBs. One involves removal of the PCB and the transformer. In this approach commonly called retrofitting, the PCBs are removed from the transformer by a simple drain and shipped to an EPA permitted facility for destruction. As Puerto Rico has no EPA authorized PCB

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destruction facilities, the PCBs must be shipped stateside for destruction. The transformer carcass, which still contains typically between 10 and 100 pounds of PCBs must be eliminated in some manner, generally by landfill. Fortunately for the Island, there are no PCB permitted landfills, and the unit likewise must be shipped stateside for burial. Fortunately because of the previously mentioned dependance on a fragile water table. Like our US neighbors, we are subjected to the restrictions of the CERCLA Regulations, and the PCBs once buried cannot be forgotten. Any problems at the landfill become our problems even though only one of our transformers may have made their way to the landfill.

The alternative to retrofit involves technology known as retrofill, whereby the PCBs are removed by a series of fluid exchanges, culminating in the reclassification of a PCB transformer to non-PCB status per US-EPA rules. The benefit of this approach is that only the PCBs are destroyed, leaving the valuable and still functional transformer in place. That in itself is a tremendous financial advantage. Retrofill of mineral oil transformers contaminated with PCBs has been successfully conducted on the Island for the past 4-6 years. However, retrofill of PCB transformers has only recently been introduced to the Island and utilizes a temporary dielectric fluid to remove PCBs. It is the author's understanding that the service has been available to the States for over two years. In our opinion, this retrofill service, which does not require the operation of unattended peripheral equipment, is superior to the replacement option in a number of areas and offers the guarantee of attaining non-PCB status.

TRANSFORMERS IN PUERTO RICO

Transformer design in Puerto Rico is similar to the designs found in the States and, in a few exceptions, Europe, including conservator transformers which generally are limited to mineral oil units. The occurrence of conservator units in Puerto Rico is rare because these from breathing units have a tendency to pick up water more than common sealed or gas blanketed units. The relative humidity in Puerto Rico is approximately 70% year round (a subtropical island setting). The safe use of free breathing transformers would be difficult at best. Exceedingly rare in Puerto Rico are units which are hermetically sealed with no air space. In the author's experience, most of the transformers in Puerto Rico are sealed or gas blanketed units.

Transformer KVAs in Puerto Rico span a wide range, from 25-7500 KVA for PCB units and up to 500 MVA for mineral oil units. These huge oil units are really step-up transformers as the secondary voltage is, usually, 230 KVA. Gallon to KVA ratios are no different from their US counterparts. This is contrary to first expectations, as one would expect that in the hotter environment the transformers would be physically larger in order to enhance the cooling efficiency. This is not the case in general, a 1000 KVA transformer contains on average 200 gallons up to a rare 300 gallons. The larger PCB transformers on the Island can contain up to almost 2,000 gallons of pure PCB prior to retrofill. On this small island, the presence of such large PCB quantities on one spot clearly pose a hazard that must be considered.

The location of transformers in the subtropical environment poses special concerns as well. Such concerns center around the always present high humidity of 70%. If transformers are improperly gasketed and are allowed to breath, the transformer dielectric coolant can rapidly be contaminated with water to a level which would compromise the transformer integrity. If the effect is severe enough, this could result in breakdown of the insulation, a short developing, severe arcing and in the extreme an explosion of the transformer. Care should be taken to ensure that the transformers are properly gasketed and that gaskets are routinely checked for leaks.

Preventive maintenance programs are rare in Puerto Rico except for the power industry and the more knowledgeable industrial owners. This habit is not wise and unfor

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tunately, is not changing rapidly. PCB units are at risk in this environment One concludes that if a transformer is exposed to water (unavoidable here), then undesirable incidents are likely to occur. If this happens, it would be far better if the transformer is non-PCB. If the transformer still functions well, there is no real reason to replace the entire unit Rather, the use of retrofill techniques and ultimate reclassification to non-PCB status would be recommended.

DEHYDRATION TECHNIQUE

Prior to, or as part of standard servicing techniques including retrofill, it is recommended that transformers in Puerto Rico be dehydrated to remove as much water as possible. This is particularly recommended for the larger transformers (higher KVA) found on the island. A number of techniques have been utilized to dry transformers. For example, simple vacuum dehydration is effective for the smaller units However, in the larger units, insufficient energy is available to remove the water present in anything approaching a reasonable timeframe. During the procedure, the core of the transformer remains cool and the water simply does not vaporize rapidly

Hot fluid exchanges may be acceptable for mineral oil contaminated units. However, for PCB units, the use of this technique would create a large hazardous waste problem. The several volumes of fluid required to remove the water would require disposal as if it were pure PCB. Consideration of this would lead to the conclusion that an alternative method for dehydration would have to be utilized short of taking over the national treasury.

Fortunately, a method can be utilized which allows for the safe, rapid and economical dehydration of these units. This process, utilizing inexpensive kerosene coupled with a vacuum dehydration is described in this paper. The first steps prepare the transformer for the dehydration itself. Initially, the PCB is drained and disposed of per EPA regulations. The unit is rinsed to remove as much PCB as possible and to limit fumes for the regasketing step by circulating about 20% by volume of kerosene for a period of time depending on the transformer size and geometry.

The transformer is then regasketed, a step required because: 1. some of the gaskets may have been damaged or worn during the years and may not support the vacuum dehydration sequences; 2. the purpose of the entire procedure is to remove water and a leaking gasket if not repaired would only allow the problem of water contamination to occur again; 3. depending on the final fluid (particularly silicone) regasketing may be required in order to prevent leaks from developing. In many instances, a large number of gaskets are involved (we have changed over 200 gaskets in one transformer). Such a large number is unusual, however.

Once the regasketing has been completed, the unit is checked for leaks. A vacuum pump and a manifold with a Freon detector are attached to the unit. Vacuum is drawn on the unit, and Freon is sprayed sequentially around each gasket. If a leak is present, the Freon is immediately drawn into the transformer and to the Freon detector. By sequentially testing every gasket, the operator can identify any leaking gaskets. If leaks are detected, the vacuum is removed, and the gasket is repaired or replaced. A recheck (same method) is carried out to assure no leaks.

Once the transformer is leak free, approximately 10% by volume of kerosene is added to the transformer. A heated, kerosene recirculating loop is attached to the transformer such that the heated kerosene enters the top of the transformer, is sprayed over the core, then exits from the bottom of the transformer. At top another opening is attached a vacuum pump with two dry ice/ acetone cold traps between pump and transformer. A temperature gauge is installed in the core if not already present

The kerosene is heated and circulated within the transformer until the temperature

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of the core reaches approximately 90°C. The time required is dependent on the transformer size and geometry, but can be expected to take at least 12-24 hours. During this time, the valves on the cooling fins must be closed (not the fins cool the kerosene, requiring a longer heat-up and down time. After the core has attained the desired 90°C the kerosene is drained from the transformer and disposed of properly The drained, sealed transformer is now ready for dehydration.

A vacuum of at least 100 torr is drawn on the transformer. Any water present will be vaporized and condensed at the dry ice/ acetone traps. Because there is no efficient means for cooling (the cooling fin valves have been closed and the transformer is under vacuum), the core of the transformer remains hot for prolonged periods of time. If the temperature falls below 75-80°C, the vacuum should be broken with dry nitrogen, and the heating procedure with the kerosene repeated. Periodically during the dehydrating, the water from the traps should be removed and quantified. The timing of the dehydrating process is fully dependent on the transformer size, geometry and total water content. However, a minimum of three (3) days can be expected to complete the process. The process can be considered complete when no additional water is collected in the cold traps. Throughout the dehydration sequence, temperature and pressure readings should be taken every hour. A check of the completeness of water removal from the transformer can be obtained by the use of Piper Curves, a discussion of which is beyond the scope of this paper.

At the conclusion of the drying, the vacuum is broken with dry nitrogen and the transformer is rinsed one time with the operating dielectric fluid (silicone oil, mineral oil, or other). After draining the rinse, the transformer is again placed under vacuum, removing most of the remaining kerosene (volatile under these vacuum conditions). The vacuum is held for at least one hour up to 24 hours, and the unit is filled with the operating dielectric fluid while under vacuum in order to eliminate air voids.

Other fluids may be utilized to heat the transformer prior to the dehydration step However, kerosene provides some benefits not realized with alternatives. For example, kerosene and water have an azeotrope which effectively removes more water than is possible with other fluids. In addition, at the finish, residual kerosene is removed by the vacuum dehydration step, flashing over to the cold trap. Finally, the kerosene is relatively inexpensive, particularly important since the material is to be considered PCB material and must be appropriately destroyed.

One may question whether the draining of the kerosene prior to the vacuum dehydration is required. This probably is not essential, however, the pumping/ recirculation operation under full vacuum of less than 100 torr is not easy to accomplish. In order to successfully recirculate the liquid, the pump (necessarily a positive displacement pump) must be below the transformer tank where it is fed by gravity. It would then feed the liquid up to the top of the transformer. We have had little success with this approach, because too little kerosene is recirculated and the transformer cannot be properly heated to 90°C. Accordingly, we have abandoned this practice in favor of draining the kerosene prior to the dehydration step.

CONCLUSION

Not every transformer requires or can be dehydrated with this technique. Only the larger transformers (generally greater than 700 gallons) need be dehydrated this extensively. The smaller KVA transformers do not require this procedure. In addition, the transformer must be capable of withstanding and holding the vacuum required to complete the dehydration step (the transformer tank must be designed to hold at least 50 torr or less). In total, this is an excellent, safe and economical method for dehydrating large PCB containing transformers prior to retrofilling and reclassification of these units

COMMERCIAL NON-SODIUM TREATMENT OF PCB CONTAMINATED MINERAL OIL

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ABSTRACT

This paper describes the current state of development of the first commercial KPEG technology for treating PCB's and other halogenated organic compounds. The process has already been permitted for full-scale use in USEPA Region 2 and New York State, and is currently scheduled for a national TSCA mobile treatment permit demonstration in October. The process has also been used commercially to treat PCB and dioxin contamination in waste oils as part of several major Superfund projects, illustrating its versatility in handling waste contaminated with water, solids and other contaminants not conducive to treatment by other available processes. The paper includes several case studies in which different waste materials have been treated successfully, and identifies plans for second and third generation technology designs for commercial deployment. The concept of joint IT/utility ownership of semipermanent facilities is described, including the status of several such projects in the U.S.

BACKGROUND

In 1982, researchers under contract to the Niagara Mohawk Power Corporation (NMPW) developed a series of non-thermal processes for removal of PCB from transformer oil. These processes use low toxicity, low hazard reagents to react the PCB to a dechlorinated material which is not soluble in the original oil. The process does not use metallic sodium, thereby providing a safe and economical method for removal of PCBs, either in a mobile or stationary fashion.

In 1985, NMPC was granted a mobile PCB treatment permit by USEPA Region 2 and the NYDEC, using a full-scale demonstration treatment rig. To date, the full-scale unit has treated over 6,000 gallons of transformer oil and 20,000 gallons of dioxin-contaminated waste oil under a variety of contracts.

CHEMISTRY

The basic chemistry of the NMPC process was described in an earlier paper presented at the 1985 EPRI PCB Seminar (Ref. 1). To summarize, tests conducted for NMPC confirmed that PCBs mixed directly with a hydroxide/glycol reagent were rapidly dechlorinated to less than 2 ppm. However, hydroxide/glycol reagents used to dechlorinate 500 ppm PCB in transformer oil reacted very slowly (6-7 hours).

In an effort to improve the rate of extraction of contaminant from the oil phase into the reagent phase, a variety of co-solvents were tested, including sulfoxides, amines, alcohols and, ketones. The most promising results were obtained with sulfoxides such as dimethyl sulfoxide or sulfolane and amines such as ethylene diamine. The addition of sulfoxide to the hydroxide/alcoh

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mixture in laboratory testing caused a substantial increase in the overall rate of reaction.

The reagent for the oil treatment process consists of two components: a solid component (KOH) and a mixture of liquid reagent materials. The proportions of these reagent components are varied to meet the needs of a particular decontamination problem, primarily due to considerations of viscosity and reactivity.

The KOH is first loaded into the reactor in a flake or prill form. The PEG, DMSO, and TMH are then added, followed by the contaminated oil. The reagent is then drawn into the recirculation line and crushed by the mixer into a powdered form which flows easily. Nitrogen is drawn from the vapor space at the top of the reactor for the conveying process.

The mixture is slowly heated using a on-board boiler/heat transfer system. The oil/reagent mixture continues to heat until a pre-determined temperature is reached. The mixture is then allowed to recirculate through the heat exchanger until analysis of the oil indicates that the PCB content is less than 2 ppm PCB. The oil is then cooled by circulating the oil through coolers until the oil/reagent mixture reaches the desired temperature.

The reagent then settles over a period of several hours, providing a well defined phase separation from the oil. The oil phase is then withdrawn and stored for any additional filtration or pre-treatment for subsequent use. Several tests of the treated mineral oil product have shown the oil to be suitable for reuse as transformer oil, although certain other tests have shown that oil quality prior to treatment can impact its reusability.

The solvent phase can be recycled for up to nine batches of oil, depending on the degree of contamination and general condition of the incoming oil. The reaction time for each batch tends to lengthen as the number of recycles increases, until the cycle time for the last batch becomes inconveniently long.

The waste reagent is a mixture of potassium chloride, unreacted reagent, and small quantities of dechlorinated byphenol. The mixture is currently being incinerated, as it represents only around 1 to 5% of the treated product. Current testing is being conducted to determine the feasibility of solidification for landfill disposal or delisting as a hazardous waste.

TEST RESULTS

The KPEG technology has been used on several commercial projects involving PCB or dioxin decontamination in hydrocarbon fluids such as mineral oil and waste oil, all with success.

The most noteworthy early project using the technology was conducted for USEPA in Butte, Montana at the Montana Pole Superfund site. Approximately 9,000 gallons of dioxin-contaminated waste oil were processed over a period of approximately two weeks. Table 1 illustrates the results of the project and shows that the dioxin was removed to below normal detection limits.

Subsequent treatability studies have been conducted for several other materials to determine the potential for the technology. Some examples of treatability results are shown in Table 2. Attempts to decontaminate oil at one Superfund site were moderately successful, reducing the level of PCB to below 10 ppm in waste oil. Because the phase separation was not as clean as in mineral oil and much of the reagent was lost to the oil phase, the speed and associated cost of treatment were relatively high compared to mineral oil. Similar tests conducted on gas line condensate have shown the process to be effective at reducing PCB concentration from 150 ppm to below 2 ppm with the first reagent pass. Again, reagent reutilization was not always feasible based on these test results.

Field demonstrations using the gas line condensate and selected waste oil products are planned for later in 1987 and early 1988. The laboratory test results have shown promise for treatment of virtually any hydrocarbon fluid contaminated with PCB of any aerochlor type, although the cost of processing will vary significantly depending on the effectiveness of reagent recovery.

PERMITS

NMPC successfully demonstrated the KPEG technology on PCB for USEPA Region 2 and NYDEC in 1985, resulting in the granting of a permit to operate the system at several NMPC owned sites in New York State. Upon licensing the technology, IT was subsequently granted operator status in New York State and is currently treating PCB-contaminated mineral oil under that permit.

IT has applied for permission to demonstrate the technology to USEPA and hopes to obtain a permit to operate on a mobile basis throughout all fifty states. The USEPA demonstration is scheduled to take place in October, attempting PCB decontamination for levels up to 5,000 ppm in mineral oil.

PLANS FOR DEVELOPMENT

Upon receipt of the initial USEPA mobile treatment permit for PCB, IT plans to construct several additional units specifically designed for different fluid treatment problems, including mineral oil and waste oil. Pending the results of the demonstration, IT hopes to offer this service commercially later this year.

In addition, several utilities and other private organizations have expressed interest in developing fixed facilities using the treatment technology, primarily for mineral oil. These projects are currently under study, particularly in locations that are remote from alternative means of disposal. The results of one or two studies should be announced shortly.

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The unit as currently designed is capable of processing between 500,000 and 1,000,000 gallons per year of oil, depending on operating schedule. Modifications to the next generation of design will allow for faster processing, perhaps increasing the unit capacity by as much as 100%.

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TABLE 1

KPEG DIOXIN TREATMENT IN WASTE OIL

SAMPLE	INST.	TETRA CDD	PENTA CDD	HEXA CDD	HEPTA CDD	OCTA CDD	TETRA CDF	PENTA CDF	HEXA CDF	HEPTA CDF	OCTA CDF
First Batch	C					ND (10)					
Second Batch	A	0.52	ND (1.1)	ND (2.1)	ND (1.0)	ND (2.9)	ND (0.60)	ND (2.3)	ND (0.52)	ND (0.67)	ND (0.35)
	B	ND (2)	ND (2)	ND (2)	ND (4)	ND (4)					
Third Batch	A	2.1	1.4	ND (1.1)	ND (1.2)	ND (3.2)	ND (2.6)	ND (1.5)	ND (0.60)	ND (1.4)	ND (1.1)
	B	4	ND (4)	ND (4)	ND (5)	ND (6)					
Fifth Batch	C					70					

A = GC/MS
B = GC/MS/MS
C = GC/EC

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TABLE 2

KPEG PCB TREATABILITY RESULTS FOR
SELECTED CONTAMINATED HYDROCARBONS
(Concentrations in ppm)

	ASPHALTIC WASTE OIL				GAS LINE CONDENSATE			
PARAMETER	INITIAL CONC.	BATCH 1	BATCH 2	REAGENT	INITIAL	BATCH 1	BATCH 2	REAGENT
AROCLOR								
1242					150	<4	110	46
1248	138	9	40	16				
1254	48	<4	<5	<4	<30	<4	<10	<4
1260					<30	<4	<10	<4
TOTAL	178	9	40	16	150	<4	110	46

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GEORGIA POWER'S SOLVENT EXTRACTION PLANT TO REMOVE PCBs FROM MINERAL OIL

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ABSTRACT

An EPRI project to evaluate PCB removal processes and oil reusability led to concentrated effort being directed toward developing a solvent extraction process. This was seen as a low-cost separation method for treating contaminated mineral oil as an alternative to the currently available destruction methods. Selecting diethylene glycol monomethyl ether as an ideal solvent, the theoretical predictions of high extraction efficiency were verified by pilot plant testing. A full scale commercial plant was then designed and constructed as a joint venture between EPRI and Georgia Power Company. Located at Georgia Power's Forest Park facility, this plant has demonstrated the ability to reduce the PCB concentration from as high as 330 ppm to less than 1 ppm. Operating costs are given for several applications of the solvent extraction process.

DEVELOPMENT

In 1981, EPRI initiated a development program with General Electric to evaluate PCB removal processes for contaminated mineral oil. The processes considered were 1) sodium, 2) electron beam irradiation, 3) critical fluid extraction, and 4) solvent extraction. The treated oils from these processes, as well as other treated oils, were evaluated by GE in a number of ways to determine their suitability for reuse in transformer service. This included analysis of changes in the hydrocarbon composition of the oils, measurement of the standard physical, electrical, and chemical properties by ASTM methods, comparative oxidation resistance, and accelerated aging studies to evaluate the effect of these treated oils on paper degradation. The results of this work have been the subject of various EPRI reports. It was concluded that the various treatment processes for removal of PCBs all produced oil suitable for reuse in electrical equipment, and in several aspects, the oils were improved.

At the same time, Veridyne, as a subcontractor to GE, prepared engineering designs for the various processes, with comparative plant and operating cost estimates.

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Based on the results of this combined effort, a decision was made by EPRI to concentrate further work on the solvent extraction process. The goal was a process for PCB removal based on separation as an alternative to the in situ destruction processes currently available.

The critical factor of a solvent extraction process is of course the solvent. The solvent selected ultimately determines the extraction efficiency, and the processing cost. The ideal solvent would extract only PCBs and no other components of the oil, and would not itself be soluble in the oil. But real solvents have a mutual solubility nature, so that the selection process is a compromise and trade off of the good and bad characteristics. Solvent selection was based on an evaluation of the following factors:

- Good selectivity for PCBs relative to oil components.
- Low solubility of oil in the solvent, and of the solvent in the oil.
- Ease of removing and recovering the solvent from the product oil.
- Ease of separating the solvent from the PCB residue.
- Low toxicity
- Moderate cost.

On this basis, GE screened a number of different solvents, and concluded that the best all around solvent was a glycol ether, diethylene glycol monomethyl ether.

FROM PILOT PLANT TO COMMERCIAL FACILITY

The next step was the construction of a pilot plant to prove out the theoretical predictions of the solvent extraction process. A 10 GPH unit was designed by Veridyne, for installation at GE's Pittsfield plant. Both new oil doped with PCBs, and aged contaminated oils were processed under a variety of conditions. During this work, it became evident that the more highly chlorinated PCB species were more resistant to removal. Yet even Aroclor 1260, the most chlorinated one in common use, could be extracted with efficiencies in the high 90's by proper adjustment of the operating variables. And a special design feature, that of recycling the PCB residue back to the extractor, proved effective in limiting the residue rate to a few percent of the feed.

The success of the pilot plant led to negotiations between EPRI and Georgia Power for the construction of a full scale commercial facility at Forest Park, Georgia. A plant capacity of 120 GPH was selected as the design basis, so as to give an annual production capacity of approximately 500,000 gallons, with two shift operation. At that time the EPA had not established required levels of PCB removal for separation processes, thus the State of Georgia's cleanup requirements were used to fix the product purity at 10 ppm. With a feed concentration of 500 ppm, this meant an extraction efficiency of 98 percent, which was within the range of performance achieved by the pilot plant. However, late in the project, after equipment orders had been placed, the EPA rendered an opinion that removal of PCBs by solvent extraction would require the same product level as had been established for the destruction processes, which was less than 2 ppm. To meet this requirement, an extraction efficiency of 99.4 percent would be needed to process 500 ppm oil.

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Construction of this facility at Forest Park was completed in the fall of 1986. It consisted of two skid-mounted units which were shop fabricated and assembled as a packaged plant. These were enclosed, together with the five 30,000 gallon feed and product tanks, and tanker unloading area, in a common building.

PROCESS DESCRIPTION

The flow diagram illustrates the main components of the solvent extraction process. It consists of two pairs of similar extraction systems, each with an extraction column and a distillation column. The first is the solvent extraction system, which contacts the oil with the solvent, and then separates the solvent from the extracted oil fraction and PCBs by distillation. The pure solvent is collected in a surge tank, and is then returned to the extractor to complete the solvent loop. A portion of the PCB residue from the distillation is recycled back to the extractor, and the remaining portion is drummed for disposal.

The second is the water wash system, which contacts the oil from the solvent extractor with wash water to remove the small amount of solvent dissolved in the oil, and which then distills the water from the extracted solvent to prooduce solvent-free water, and water-free solvent to return to main solvent loop. The distilled water is pumped back to the water wash column.

The two extraction columns, which are identical in construction, were supplied by the Otto H. York Company. They contain multiple stages for the countercurrent contacting of the oil and solvent, and the oil and water.

The agitation for each mixing stage is provided by rotating turbine blades attached to a central shaft. Surrounding each mixing stage is a coalescing zone and baffles, consisting of a ring of wire mesh which separates the two phases, with the light phase (oil) flowing up to the next higher stage, and the heavy phase (solvent or water) flowing down to the next lower stage. The upflowing oil, in the case of the solvent system, progressively contacts solvent that contains less and less of the PCBs. Thus at each mixing stage PCBs pass from the oil phase into the solvent phase, to establish a new equilibrium condition. The upflowing oil becomes gradually depleted of PCBs, while the downflowing solvent becomes progressively richer in the contaminant. At the top stage, from which the oil exits, the oil contacts the fresh incoming solvent, which is completely free of PCBs. At the bottom stage, as the solvent exits the extraction section, the solvent contacts the oil with the highest PCB concentration, which is the incoming feed. An extra section has been added to the solvent extractor below the feed point to enable processing the residue recycle as a secondary oil feed. The solvent phase from the extraction section continues down through this section to remove the PCBs from the recycle stream.

The distillation columns that are coupled to each of the extractors operate under a moderate vacuum, so as to limit the bottoms temperatures to a maximum of 155 C. The heat supply to the distillation columns if 175 psig steam. Cooling for the overhead condensers is provided by a closed-loop cooling tower.

PLANT OPERATIONS AND MODIFICATIONS

Shakedown and calibration runs were carried out for the first two months of operations using non-PCB oil as feed. Then during November, 1986, PCB-contaminated oil was processed under a 30-day EPA R&D permit. At the end of this period, it was evident that minor process modifications were needed to correct a problem in the residue handling system. The solubility of the oil in the extract leaving the extractor was appreciably greater than what had been experienced with the pilot plant. This resulted in an increased oil residue rate, an increase that the residue recycle could not handle. Since this problem did not surface during the pilot plant runs, it appeared that the oil for those runs was not as severely aged as the oil that was typical of Georgia Power's operations.

In any event, this difficulty brought to light an added feature of solvent extraction not at first recognized. The solvent used not only did a good job of removing PCBs, but also of removing the products of thermal decomposition and oxidation. Removal of these highly polar compounds further upgrades the quality of the product oil, and will undoubtedly extend the service life in reuse applications. The only penalty is that a larger percentage of the oil ends up as PCB residue for disposal. In the original design, the residue rate was controlled at about 2 percent of the feed by recycling the excess residue back to the extractor. However, with the higher content of aging products in the oil, the larger soluble oil fraction could not be recycled to the same extent. Equipment changes were made to permit a more flexible residue handling system.

Another important change was made in the process. During the laboratory work that was undertaken at this time, it was discovered that a slight change in the composition of the solvent lessened this increase in oil solubility in the solvent, and thus the quantity of net residue produced. But at extreme limits, this reduction in solubility by changing the composition was accompanied by a reduction in the extraction efficiency. In practice, then, this finding suggests that a compromise could be sought between residue rate and efficiency, in processing oils of different levels of PCBs.

Plant modifications in these two areas were completed early this year. Finally, after extended delays in obtaining a renewal of the EPA R&D permit, a second test run was conducted during July.

Various types of contaminated oils were processed during these runs, varying in PCB content from 73 to 330 ppm. At the higher concentration, product oil of less than 1 ppm was obtained, with a residue rate of about 12 percent. This is an extraction efficiency far better than the design value of 98 percent. For this condition, the solvent composition could have been adjusted to lower the residue rate to around 10 percent, allowing the PCB content in the product to increase to the 2 ppm level. This was done for oil feeds with lower PCB concentrations, and residue rates in the range of 6 to 9 percent were obtained. It should be noted that the solvent composition can be adjusted either way while the plant is in operation.

The operating permit expired before full process optimization was completed. This work had been hampered by uncertain and conflicting results from the different laboratories doing the PCB determinations. But the test run did demonstrate that

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high extraction efficiency is achievable, with reasonable residue rates, to process oils with high levels of PCBs.

Economics.

A cost analysis of the solvent extraction process is shown in Table 1. Two cases are presented, one in which the oil is produced for reuse in electrical equipment, and the other in which the oil is simply decontaminated for sale as a fuel. For each of these cases, there are two options for residue disposal. The first is disposal by outside means, as is the case at Georgia Power. The scond is in-plant processing of the residue by one of the non-sodium methods, then disposing of the refined residue as a fuel product. In both cases, the cost of replacement oil is included in the total cost - the 10 percent residue loss in the reuse case, and the 100 percent replacement in the fuel case.

The lowest cost option is processing oil for reuse, employing the in-plant residue option, at $1.32/gal. But the corresponding fuel case is also attractive, depending on the cost of new oil. At a projected cost of $1.50/gal., the processing cost after deducting the fuel credit adds only $0.41 to give a total cost of $1.91/gal.

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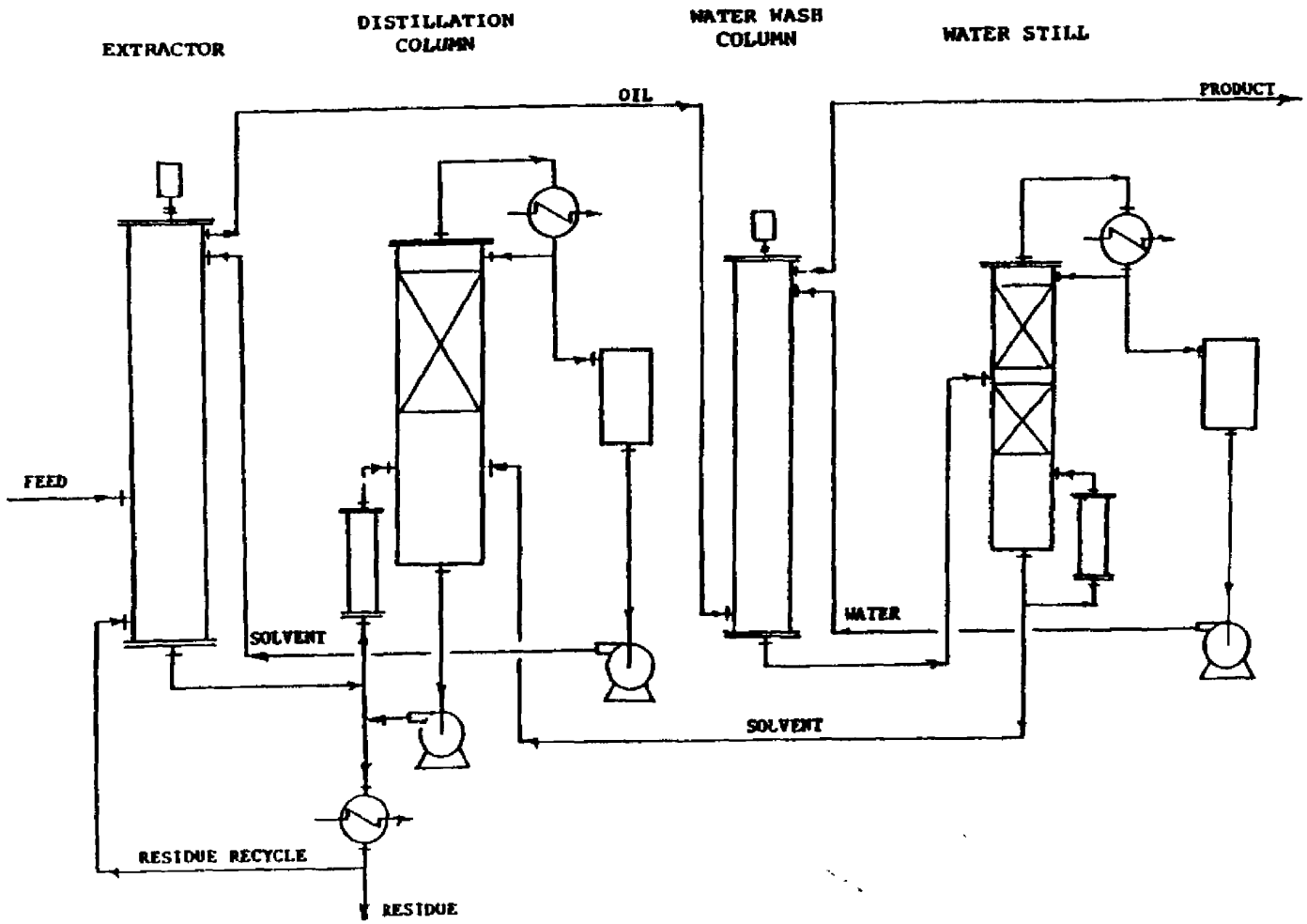
EXTRACTOR
DISTILLATION COLUMN
WATER WASH COLUMN
WATER STILL
OIL
PRODUCT
FEED
SOLVENT
WATER
SOLVENT
RESIDUE RECYCLE
RESIDUE

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Table 1

SOLVENT EXTRACTION COST ANALYSIS

Annual Production - 500,000 Gal

	$/Gal	
Solvent Extraction	Oil for Reuse	Oil for Fuel
Utilities & Solvent	0.12	0.12
Operating & Maintenance Costs	0.27	0.27
Total Operating Cost	0.39	0.39
Fixed Charges (capital replacement, int.)	0.32	0.32
Processing Cost	0.71	0.71
Fuel Credit	-	-0.36
Total Operating Cost	0.71	0.35
Reclamation		
Degassing & Clay Treating	0.40	-

Residue & Replacement	Oil for Reuse		Oil for Fuel	
	Outside	In-Plant	Outside	In-Plant
Residue Disposal or Treatment	0.60	0.10	0.60	0.10
Fuel Credit	-	-0.04	-	-0.04
Oil Replacement	0.15	0.15	1.50	1.50
Net Processing Cost	1.86	1.32	2.45	1.91

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PART 10:

PCB ANALYSIS

PCBA-102 UPDATE

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ABSTRACT

The further development of the PCBA-102 for characterizing Aroclors in dielectric fluids and samples taken from spill sites is described. As a result of findings obtained on inter-utility studies managed by the Brown & Caldwell (RP-1263-23), several specific changes have been incorporated into the operation of and training for the PCBA-102. The major changes incorporated were modifications of the software and implementation of several hardware upgrades. The instructions for use of the PCBA-102 have been rewritten to provide additional clarity. An operational videotape is also available to facilitate the break-in period for an uninitiated operator. We believe these modifications have further simplified the use of the instrument and provide assurance that repeatable, accurate data are obtained.

PCBA-102 instruments were subjected to a series of environmental tests to assure they could operate in field thermal and humidity environments and withstand vibrations which may occur in off-road transport of the instrument. Also presented are the results of a detailed three-concentration level, four-Aroclor type, and five-instrument factorial study (in quintuplet) which showed excellent repeatability. The results of interlaboratory testing among three utilities and a service laboratory are presented. Finally, in-house studies of testing and characterization of samples containing mixed Aroclor mixtures are presented.

BACKGROUND

The PCBA-102 portable gas chromatograph for polychlorinated biphenyl analyses was developed by S-CUBED under partial support from the Electric Power Research Institute under Project 1263-9 (Ref. 1,2). The PCBA-102 utilizes a simple sample preparation kit wherein a portion of a liquid sample is added to a mixture of hexane and sulfuric acid, the mixture is shaken, resulting in the extraction of Aroclors into the hexane phase; other impurities are retained in the sulfuric acid layer. After the instrument has been calibrated and ready for sample analysis, a 2-μL portion of the hexane layer is then transferred by means of a 5-μL syringe into the instrument. For soils, they are treated with methanol to extract Aroclors and then a portion of the methanol mixture is subjected to the oil preparation kit procedure to partition Aroclors into the hexane layer. The resulting gas chromatogram is stored in the on-board minicomputer and is compared with Aroclor standards in memory. At the conclusion of the chromatogram (about 15 minutes), pattern recognition and quantitation subroutines are pwrformed and the instrument prints out information on (1) the Aroclor type found, (2) its concentration in ppm, and (3) a measure of the closeness of the sample chromatogram shape to that of Aroclor standards stored in memory. This measure is called MATCH, which is calculated using a multidimension statistical procedure (MATCH values range from 0 to 1000, with values above 800 being an acceptable comparison). In EPRI Contract 1263-23,

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Brown & Caldwell conducted a field inter-utility study to determine the applicability of the PCBA-102 (Ref. 3). One of the primary difficulties experienced was the fact that the field operators, who were unfamiliar with the use of gas chromatographs had a tendency to tighten down on the septum nut to the point that the septum would be extruded into the gas chromatographic column, thereby severely restricting gas flow. The extrusion of the septum material was by far the biggest cause for failures in the field. Other problems arose from inexperienced operators in injecting a microliter chromatographic syringe into the instrument.

As a result of the operational problems encountered during the Brown & Caldwell project, several specific changes have been incorporated into the operation and training for the PCBA-102. In addition, several upgrades were incorporated to eliminate other potential problems we saw, and to facilitate the manufacture of PCBA-102. These changes a discussed below in sections on:

- Software modification,
- Hardware upgrade,
- Operators manual modifications, and
- A videotape demonstration on the use and care of the PCBA-102.

SOFTWARE MODIFICATIONS

The pattern recognition code was rewritten from the original combination of assembly and PASCAL languages into C language. The peak detection scheme was also modified to incorporate the peak detection by the increasing tangent search, based on a second derivative test with decreased sensitivity for noise, as described by Woerlee and Mol (Ref. 4). By use of a C-emulator station, a computer program was prepared to assist in preparation of individual instrument standard tables, thereby reducing the time to prepare a new PCBA unit for use.

Software was also modified to provide additional information during the analysis, stating when the data were being acquired, when the program was going through the MATCH and Aroclor calculation steps, and, finally, providing the results.

HARDWARE UPGRADES

Several modifications to the hardware were incorporated to facilitate the operation of the PCBA-102 and permit the instrument to meet specific mechanical test requirements. The column inlet tube was tapered to facilitate insertion of the syringes into the instrument. Several mechanical connections were strengthened, the column coils were restrained mechanically, and the oven's thermal insulation was made secure mechanically.

The PCBA-102 has passed the following specific environmental tests in accordance with test protocol MIL SPEC-STD-810C:

- High Temperature - Test Method 501.1; 24-hour exposure to 40°C.
- Low Temperature - Test Method 502.1; 24-hour exposure to -10°C.
- Humidity - Test Method 507.1; 24-hour exposure to 90% relative humidity maintained at 40°C. Temperature cycling is not a part of this test.

- Vibration - Test Method 514.2, Procedure X; test was performed at three different levels, including the high-level vibration test, the magnitude of maximum displacement in a vibration table was one inch with a force of 1.5g. The vibration was done in three axis from 5 to 200 to 5 hz in seven cycles of 12-minutes each.

After the tests, the instrument was then powered up and Aroclor 1260 was found with MATCHs from 975 to 989 at concentrations within ±10%. It is believed this vibration test clearly exceeds that that would be expected to be found in an off-road truck environment.

OPERATORS MANUAL MODIFICATIONS

Considerable more information was provided to the operator in the instruction manual regarding proper procedures for filling and using 5-μL syringes with the PCB extracts. The operator is encouraged to practice this step for the first several runs until he has built up confidence that he can inject a repeatable amount of PCB extract into the PCBA-102. The entire operation of cleaning the syringe, transferring a representative aliquot to the desired level in the syringe, and, finally, injection into the gas chromatograph is relatively straightforward; however, as was observed by Brown & Caldwell, this process does take some finesse, particularly with people who tend to be "all thumbs." Other aids described are: (1) techniques to assure that only the hexane layer is removed for analysis and not the sulfuric acid layer, (2) the use of a Chaney adaptor to present the amount of fluid in the syringe, (3) a special section on how to analyze both low and high PCB contractions in dielectric fluids and soils, (4) use of standard Aroclor solutions prepared in isooctane to assure their long-term integrity, and (5) a special troubleshooting section, including normal and problem computer messages.

Because the instrument is optimized to run samples having concentrations in the range of 10- to 1000-ppm Aroclor, it is important that samples having high Aroclor concentrations are diluted prior to injection into the instrument. Using the normal procedure, samples above 2,000 or 3,000 ppm tend to saturate the detector and the instrument will be inoperable for as long as eight hours to a day. To this end, S-CUBED recommends using a screening procedure, such as CLOR-N-OIL™, to estimate the actual concentration range and then dilute the sample, if appropriate. An operational videotape has been prepared to assist operators in observing how one is suppose to use the device. As a result of the revised instructions and the videotape, the sample preparation and insertion into the instrument has been improved. We believe these modifications have simplified the use of the instrument and have provided assurance that repeatable, accurate data are obtained.

REPEATABILITY OF DIFFERENT INSTRUMENTS

The precision and accuracy of the PCBA-102 was studied by using five different instruments and quintriplicate analysis of 100-, 250-, and 500-ppm standards of Aroclors 1016, 1242, 1254, and 1260. The results of this three-concentration level, four-Aroclor type, five-instrument and five-replicate (300 analyses) study are shown are Table 1. It is seen that excellent accuracy (comparison of the averages found and that taken) and MATCH of the standards were obtained. The repeatability of the concentration measurements (e.g., pooled standard deviation) was generally less than ten percent relative, which was higher than the average error. Mean MATCH values were all greater than 950. The MATCH pooled standard deviation was generally greater for the early eluting Aroclors 1016 and 1242 than for Aroclors 1254 and 1260. The latter two Aroclors take considerably more time to elute and have broader peaks; hence, the error associated with measurements associated

with retention time is considerably less. Nevertheless, these data show the precision and accuracy capable of achieving with the PCBA-102 when analyzing known standards.

Table 1. ANALYSIS OF STANDARDS

		Concentration Taken (ppm)					
		100		250		500	
Aroclor	Measurement	Value Found	Pooled SD	Value Found	Pooled SD	Value Found	Pooled SD
1016	Concentration	96	5.5	247	11.3	484	12.7
	Match	976	24.6	970	24.6	964	21.7
1242	Concentration	97	6.6	245	26.4	489	23.3
	Match	969	31.2	956	23.4	967	32.3
1254	Concentration	97	7.0	256	12.8	513	26.2
	Match	983	9.8	983	6.6	983	5.5
1260	Concentration	100	3.5	250	16.4	462	23.1
	Match	990	2.5	991	4.5	989	9.1

An interlaboratory study of the use of an applicability of the PCBA-102 was undertaken through analyses by Pacific Gas & Electric, San Diego Gas & Electric, Los Angeles Power & Light, and Myriad. In general, these users found the instrument to meet their needs; however, some concerns were raised because the instrument, as it is currently designed cannot report the results of Aroclor mixtures. To that end, S-CUBED is conducting studies to determine the magnitude of errors that might arise through the analysis of binary Aroclor mixtures. The results of studies of the 15 binary mixtures obtained at concentration levels of 10, 50 and 100 ppm for the three Aroclor types 1242, 1254, and 1260, will be presented.

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CHROMATOGRAPHIC ANALYSIS OF PCBs IN TRANSFORMER OILS: A MINI-ROUND ROBIN

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and

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An article appeared in Electric Light and Power magazine in December 1986 reporting that Capillary Column Chromatography/Mass Spectroscopy (CC/MS) for analysis of PCBs in transformer oils was more accurate than Packed Column Gas Chromatography/Electron Capture Detector (PC/ECD) and Capillary Column/Electron Capture Detector (CC/ECD). It was said that these commonly relied upon ECD techniques had a tendency to give false high readings. It is shown in this paper that, when properly done, all three techniques give comparable results.

This work was initiated because a number of people and organizations expressed concern over the published claim of systematically incorrect PCB analyses. Included among these were electric utility PCB managers, the United States Environmental Protection Agency, members of ASTM Committee D27, who contributed to the development of ASTM Standard Method D4059 (Analysis of Polychlorinated Biphenyls in Insulating Liquids by Gas Chromatography), and Doble Engineering Company. It was suggested that EPRI investigate the matter. EPRI then contracted with General Electric in Pittsfield, MA to investigate the relationship between the three methods.

General Electric arranged with three nationally-known laboratories to run a series of tests. The participants were:

Midwest Research Institute - Rob Scuderi
Radian Corporation - Maureen Kilpatrick and Alan Nichols
Twin City Testing - Fred DeRoos

Four samples were obtained, and aliquots sent to each laboratory. Each one was requested to analyze the samples by three methods:

PC/ECD - Packed Column/Electron Capture Detector
CC/ECD - Capillary Column/Electron Capture Detector
CC/MS - Capillary Column/Mass Spectrometer

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The samples were:

A - 430 µg/g Aroclor 1254* in aged oil
B - 1110 µg/g Aroclor 1242* in new oil
C - 2350 µg/g Aroclor 1254* in aged oil
D - 4170 µg/g Aroclor 1242* in aged oil

* = average of all results from three labs and three methods

For comparison of laboratories and methods, the results were normalized so that 1.00 represented the average (for all laboratories and all methods) on each sample. The average values, R_{aver}, and standard deviation, σ_n, are shown in the following tables:

NORMALIZED RESULTS - PCB ANALYSIS
AVERAGED BY METHOD

Method	$R = {}^{1}X/{}^{1}X_{aver}$ All Samples	
PC/ECD	R_{aver}	0.97
	σ_n	0.09
CC/ECD	R_{aver}	1.08
	σ_n	0.07
CC/MS	R_{aver}	0.95
	σ_n	0.19
All Methods	R_{aver}	1.00
	σ_n	0.13

NORMALIZED RESULTS - PCB ANALYSIS
AVERAGED BY LABORATORY

Laboratory	$R = {}^{1}X/{}^{1}X_{aver}$ All Samples	
W	R_{aver}	0.96
	σ_n	0.14
Y	R_{aver}	1.11
	σ_n	0.16
Z	R_{aver}	0.93
	σ_n	0.11
All Labs	R_{aver}	1.00
	σ_n	0.13

Finally, the following observations were made:

- Results from Lab Y are somewhat higher.
- Results from Method CC/ECD are somewhat higher.
- Results from Method CC/MS are somewhat more variable.
- Differences between Methods and Labs are less than 20%.

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We concluded that the ECD techniques show no significant systematic difference from MS when proper procedures are used for each, and therefore, the ECD methods do not result in falsely high values.

INCREASING THE ACCURACY OF THE GC/ECD AND GC/HECD QUANTITATION OF PCBs USING A NEW PCB ANALYTICAL STANDARD

Steve T. Turner and Robert Scuderi

INTRODUCTION

Polychlorinated biphenyls (PCBs) present a difficult challenge to the analytical chemist. There are 209 forms of the PCB molecule and each form (isomer) is different in its effect on the common analytical detection systems presently available. This difference in effect by each isomer on the analytical detection systems, combined with the wide variety of isomers often encountered in PCB contaminated samples, can make the accurate measurement of PCBs a difficult task. To address this problem, the possibility of a new analytical standard mixture is investigated.

BACKGROUND

The Toxic Substances Control Act of 1976 requires the phasing-out of all material or equipment contaminated with PCBs above a certain level. This regulation has resulted in the need for the analysis of a large number of samples from PCB destruction/removal processes. Midwest Research Institute (MRI) has been providing technical assistance to the U.S. Environmental Protection Agency's Office of Toxic Substances in support of their effort to monitor these destruction/removal processes since 1980. Part of MRI's responsibilities have included the analysis of pre- and post-process samples and by-products from mobile PCB incinerators and alternative PCB destruction/removal processes to determine if the regulatory limit for PCB content has been achieved.

The analytical method most frequently used to measure the PCB content in destruction/removal process samples is gas chromatography (GC) equipped with a chlorine sensitive detection system. Chlorine detection systems

include the electron capture detector (ECD) and the Hall electrolytic conductivity detector (HECD). Most PCB destruction/removal processes require the analysis of samples containing a variety of PCBs. The feed samples from these processes are typically contaminated with PCBs as Aroclors. The processed samples may be contaminated with PCBs as individual isomers.

If the identity of the sample's PCB "character" can be adequately identified, as is the case when a single, nondegraded Aroclor is the primary analyte, then the sample's PCB content is most accurately measured using an external Aroclor analytical standard. This measurement can be made by comparing the sample's chromatographic pattern versus the chromatographic pattern and concentration of a known Aroclor (i.e., fingerprinting). However, when the sample's PCB "character" cannot be easily identified, the PCB measurement process becomes more complicated. Examples of this are: samples containing mixtures of Aroclors, partially degraded Aroclors (occuring either singly or in combination with other degraded Aroclors or nondegraded Aroclors) and samples which contain individual PCB isomers which do not present an Aroclor chrometographic pattern.

The accurate measurement of the PCB content of these complex samples presents a unique challenge to the analytical chemist. One method seeking to provide at least a partial solution to this problem has been the use of a multi-isomer PCB analytical standard referred to as the Dye Color Manufacturers Association (DCMA) standard. The DCMA standard is a 10-PCB isomer external standard containing a series of single isomers representing each of the 10 possible homolog series of PCBs. The DCMA standard isomers are used to establish retention time windows which are compared to the sample chromatograms. The establishment of the windows is arbitrary, however, one common method is by setting the windows at a midpoint retention time between the DCMA isomers as they appear in the DCMA standard chromatogram. PCB concentration in the sample is then calculated by comparing the sample's peak response (integrated area) to the response observed for the DCMA standard isomer in the window where the sample peak occurs. The DCMA external standard method measures PCB sample concentrations by allocating sample chrometographic responses to 10 "homolog" windows.

Post-process samples frequently have chrometographic peaks in the mono- and di-PCB retention time region. Evaluations of the DCMA analytical standard

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have been conducted by GC/ECD analyis to quantitate a solution of the 15 mono- and di-PCB isomers. This evaluation resulted in accuracies ranging from 10% to 1500% for the individual isomers. The reason for such a large accuracy range is primarily due to the variability of the PCB isomers responses to the ECD detector. This range of accuracy can be predicted by previously published literature values(1) for the 209 PCB isomer's relative response factors (RRFs) for ECD detectors. The published values also indicate the variation of the RRF is significant within each of the 10 PCB homolog series.

OBJECTIVE

The objective was to determine if an alternate PCB isomer external standard could be predicted from the literature values to more accurately measure the PCB content of samples. Guidelines were established prior to the investigation which balance the need for increased accuracy with the need to optimize the utility of the resulting standard. These guidelines were:

- The new standard will primarily be tailored for ECD analysis but also be applicable to HECD analysis.
- The new standard should also be easy to use and contain a minimum number of isomers.
- The standard isomers should be commercially available and not require time-consuming and costly synthesis.
- The new standard should be amenable to changes in GC temperature programming parameters in order to reduce the overall analysis time for each sample.
- The statistical analysis of the RRFs (literature values for ECD, estimated values for HECD) and RRTs will be performed on an unweighted distribution for all 209 PCB isomers.

The purpose of this last guideline is three-fold: (1) to maintain a predictable performance for the standard and preclude tailoring the standard to a certain subset of samples which would then decrease the standards accuracy for a different sample set, (2) to recognize the improbability of

accurately predicting PCB isomer abundance for a very large set of sample types or destruction/removal processes, and (3) to apply the standard to a significant variety of environmental samples where the PCB isomer abundances very greatly. Relative response factors for the HECD analysis were estimated by using the homolog number for each PCB isomer. The HECD pyrolysis chamber was assumed to be 100% efficient in its ability to cleave the chlorine-carbon bond. Any decrease in the pyrolytic efficiency of the HECD pyrolysis chamber to cleave the bond would tend to result in a decrease in the RRF/HECD variation between homologous series.

RESULTS

Published RRF values for ECD analysis, RRT values(1) for a specified GC liquid phase and estimated RRF values for HECD analysis were evaluated. It was concluded that a PCB standard containing seven isomers could achieve the goals that had been established. The isomers used in the new standard are IUPAC (international union of Pure and Applied Chemists) Nos. 1, 10, 24, 69, 187, 198, and 209.

The 7 isomers in the projected standard were chosen so that their individual retention times would successively begin and end calibration windows, thereby helping to facilitate its use as well as allow temperature programming to occur without invalidating the statistical study.

The estimated relative standard deviation for accuracy (calculated by using the weighted mean of each calibration window based on the total number of isomers present) for ECD analysis is + / -33% and for HECD analysis is + / - 16%. This accuracy cannot be greatly improved without the inclusion of an excessive number of additional isomers.

Comparison of the literature values(2,3) for the PCB isomer abundances in the 7-isomer standard windows for Aroclors 1016, 1242, 1248, 1254, and 1260 indicate a possible accuracy for the quantitation of these Aroclors by either ECD analysis or HECD analysis of from 90% to 110%. This may indicate the 7-isomer standard can provide good accuracy for complex Aroclor mixtures.

SUMMARY

The DCMA standard is shown to be inaccurate for the quantitation of frequently encountered PCB by-products from PCB destruction/removal processes. Literature review and subsequent statistical analysis indicated a new 7-PCB standard mixture to be theoretically more accurate for non-Aroclor PCB quantitation by GC/ECD and GC/HECD analysis. Additional analysis of the 7-isomer standard mixture for use as an Aroclor standard for complex Aroclor mixtures is promising. GC/ECD and GC/HECD analysis of the potential standard is currently being performed. Full experimental results will be detailed.

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3. Albro, Phillip W., Jean T. Corbett, and Joanna L. Schroeder. "Quantitative Characterization of Polychlorinated Biphenyl Mixtures (Aroclors 1248, 1254, and 1260) by Gas Chromatography Using Capillary Columns," Journal of Chromatography 205:103-111 (1981).

MONS 020371

PCB System Inventory: To Predict PCB Contamination in Electrical Equipment

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ABSTRACT

Electric utilities often identify a system inventory as the preferred method to organize an oil sampling program to test for PCB concentration. A full-scale inventory is implemented over a three to four month period (depending on the size of the service area, number of transformers, etc.) by a project manager, temporary supervisor, and data entry and field teams. The data provide current information to update utility maps and records (cards and computer information), billings for pole contacts and maintenance requirements. Most importantly, the data provide a snapshot of geographic conditions which describe environmental and human health and safety exposure risks. The number of equipment in specific categories of exposure risk can be prioritized for a systematic program of sampling, testing and removal, if warranted. The author discusses planning, implementation, costs, and benefits of data to other areas of system operation.

INTRODUCTION

The decision to implement a PCB system inventory to identify potential PCB (polychlorinated biphenyl) exposure risk recognizes that the burden of PCB regulations is not diminishing and action is necessary to identify those electrical units which pose the greatest threat to the environment, human health and the financial status of a utility. The ultimate result is the ability to rank equipment for either sampling and then removal or removal prior to sampling. A PCB system inventory should represent approximately .05 of one percent of a utility's annual budget, and is likely to lead to a yearly budget commitment over several years representing one percent annually.

The intent of a PCB system inventory is to clarify best PCB management practices for a utility to serve the needs of the customer base, to respond to government regulation of PCBs, to reduce liability exposure and to support cost-effective planning and implementation of a PCB control program. The inventory is designed to systematically collect information to build a data base which identifies and environmentally characterizes all fluid-filled equipment in an electrical service area pertaining to PCB exposure risk. Needs for further information gathering activities, such as sampling, or the cost-effectiveness of removal can be assessed using this data. Data analysis produces categories of equipment which can be targeted as "suspect PCB," because the serial numbers closely match other equipment with lab test results in specific ranges of PCB contamination. In this way, a PCB inventory

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provides the data to accurately quantify probable PCB contamination in a service area. Targeted equipment can be removed first which reduces liability by orders of magnitude at the onset of a PCB removal program.

IMPLEMENTATION

Evaluating the potential exposure risks of unknown PCB levels in all types of electrical equipment found in a distribution network is a cumbersome task, at best. This task is streamlined by utilizing a baseline which captures the data necessary to make informed and economically sound decisions about sampling effort, equipment management, removal and disposal. The baseline is composed of data from the following sources: the history of PCB lab test results; the identification of imminent hazards; the characterization of pathways to food or feed, water and commercial ventilation systems; and the record of the distance of imminent hazards from electrical equipment. These data elements are likely to be new areas of information development within a utility and constitute the backbone of the field inventory. This baseline responds to the Environmental Protection Agency regulation of PCBs.

From the entire data base, a utility can determine the most operationally efficient and cost-effective strategy to decrease exposure risks and liability from inadvertent release of PCBs to the environment. Several strategies for equipment management are available to utilities such as removal of equipment based on unit-specific test results, removal of equipment based on serial number inference from the test results history, and retrofilling units.

A system inventory can be completed either through full-scale implementation of the field survey or in a phased approach through surveying equipment around specific imminent hazards such as schools, restaurants, and large buildings. Both methods have merits to qualify them.

For utilities with maps and records severely out-of-date, a full-scale system inventory allows a great quantity of information to be collected which, if recorded promptly, can bring records to a high level of accuracy in as short a time period as a few months. This method can require field crews to record information about equipment not directly pertaining to PCB control, such as telephone and TV cable unauthorized contacts verification, which helps to support the commitment to budget for and implement the inventory. Revenue generated from this verification can be substantial. Also, for utilities performing in-house maintenance on electrical equipment, the full-scale inventory allows the environmental considerations of the data base to carry more weight. This is important especially if oil was reused among transformers making the serial number inference less dependable.

The phased method of inventorying is useful to utilities with fairly accurate maps and records, because a substantial portion of work can be performed in the office prior to going out in the field. The mass of data generated during phased inventories can be turned over fairly quickly to facilitate the equipment management decision process, and then the data can be filed. Either approach is aided by a history of lab test results, but the phased approach especially is enhanced by using the test results history to justify removal of equipment based on inferences about specific serial number categories of equipment manufacturers. This method is applicable to systems which do not have excessive oil cross-contamination of units.

In either case, but particularly with a full-scale inventory, the mass of data collected will be enormous. This necessitates planning for computer access. Several computer applications can be used, but a PC with adequate storage

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capacity is likely the most versatile and powerful medium. Sorting capability is more important than statistical evaluation, so a software program such as DBASE is adequate and easily learned by data entry personnel.

It is essential to coordinate the field inventory with the maps and records division, because incorporating the data with existing records is crucial. Also, the maps and records system is a necessary element in setting up the inventory work schedule and strategy. It is important to note that some utilities may have made the decision not to maintain current maps and written information about equipment locations and types to save operating costs. A credible and successful PCB management program influences all areas of operations and maintenance in a utility. Without a data base, the program is crippled administratively and every spill is a crisis. As PCB regulations are enforced more stringently every year, current records are increasingly essential to utility operations.

Despite the analytical conclusion that cleaning up spills as they occur is cheaper in the longrun than a concerted removal program, liability exposure risk considerations have prompted several utilities to characterize the equipment on their respective systems immediately. Small utilities with less than 5,000 transformers have opted to simply test everything on the system. Large utilities with 20,000 to 1,000,000 transformers have begun to reduce liability substantially by making inferences about manufacturer serial numbers based on the history of PCB lab test results, and are removing equipment from imminent hazard areas prior to sampling based on those inferences.

Results of a full-scale inventory at Grant County PUD, a phased inventory at Snohomish County PUD, both of Washington, and complete equipment sampling and testing at Salem Electric in Oregon show that General Electric and Westinghouse transformers are the likely manufacturers to test out greater than 500 parts per million PCB. One plant for each manufacturer (Sharon, PA - Westinghouse; Pittsfield, MA - General Electric) produced the units which can be targeted by serial number inference as contaminated. For both plants, this contamination problem appears to drop off beginning in 1972 or 1973. Standard transformers of all types from the serial number beginning with "PF" (approximately 1974) often test out greater than 100 parts per million PCB.

As an example, implementation of a full-scale PCB Inventory for a system with 100,000 transformers will cost $300,000, and requires 40 workers to complete the project in four months. The number of workers can be reduced by lengthening the project time, but the cost will remain roughly the same. This type of inventory involves a project manager, two crew supervisors, five data entry and forms controllers, and 16 two-man crews. College students can be used for the field survey, data control and crew supervision. The skills required of the survey crews include training in natural resources and/or environmental management and/or environmental engineering, the ability to read Township and Range maps, First Aid qualifications, the ability to record information accurately and the desire to work outdoors in changing conditions. Crew rotation allows the observational skills of individuals to be kept at a high level and transmitted throughout the crews, thereby increasing the accuracy of information.

The data collected (See: Figure 1, Geographic Description of Equipment Location form) is scored (given points) according to specific parameters, to be discussed, which result in the ability to rank transformers for sampling or removal. The crews collect the data elements, as shown in Table 1, for the purposes of uniquely identifying each unit of electrical equipment, each geographic location, and the exposure risks of each site. The site is given points according to a set scale, and the point total, called the Transformer

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Exposure Risk Score (TERS), allows units to be prioritized for sampling or removal over a predictable time period.

In the high density category, three points are allocated for access, high traffic, shopping, and/or parking lots near a transformer. For distance, if an imminent hazard such as water or a restaurant is within 100 feet of a unit, it receives 3 points.

Imminent hazards are defined as raw food/feed processing, wholesale/retail food/feed, water, commercial ventilation systems, open storage of food/feed, human health and safety, and hospital/health care facilities. All the hazards receive five points except for the last two categories which receive four points. EPA does not specifically regulate water exposure as it does food and feed facilities and commercial ventilation systems except for the containment of water in the case of extinguishing fire. However, in light of spill cleanup problems associated with water contamination and the difficulty of disposing of contaminated water, the water imminent hazard carries equal weight with food and feed or commercial ventilation exposures.

Pathways are defined as the ability of transformer oil to be transferred to an imminent hazard. This point criteria is the most subjective in the field survey and scoring process, but continual communication about what is being seen in the field allows the supervisors and project manager to maintain control over this evaluation. The prime methods of transfer are contamination by actual contact with an imminent hazard and transfer by the food chain or machinery.

Other data elements which can be added to the TERS include a point allocation for equipment age (sliding scale), a stained unit (no current leak visible, current leakers reported immediately), burn barrels, fire wood, garbage cans and mail boxes. Burn barrels and fire wood receive three points because of the potential for dioxin development through burning PCB-contaminated wood or refuse.

Educational areas receive three points. Residential and agricultural areas receive two points. Recreation, airport, industrial and undeveloped areas all receive one point. The reason these areas receive a general point allocation is that no imminent hazard may be present within 100 feet or near a pathway, but the point allocation reflects human or animal use of the area. Commercial areas are not given a general point allocation, because imminent hazards and pathways are typically present.

Equipment used in the field includes high-powered spotting scopes at a cost of approximately $500 for the scope and carrying case, tripods, distribution maps, tape measures, notebooks, field report forms and name tags for identification. Each crew is assigned to a designated Section, Township and Range. One crew member sets up the spotting scope and tripod to read the equipment nameplate, and the other crew member records information on the transformer location form noting the following: map location and road name, whether the transformer is on the map correctly, area and ground condition of the site, water type, distance of the transformer from buildings or imminent hazards and any other additional observations. Missing unit numbers and TV cable and telephone contacts are also noted.

This level of activity is more important to a utility which has inaccurate written records of actual equipment in the field. The spotting scopes used with a tripod are highly effective in reading nameplates. If written records are accurate, the total cost of implementing the inventory will be much less if the spotting scopes are not used.

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CONCLUSION

Deciding the level of inventory effort to commit to may be a difficult task for utilities, but several considerations may streamline the decision process. The inventory will produce valuable information and results for all areas of utility operations from customer requests made to survey crews to identification of equipment hazards. The physical status of the electrical system is evaluated. The maps and records are updated. Leakers are identified. The PCB program manager becomes familiar with potential environmental hazards which can be addressed immediately or in subsequent budget and labor planning.

All the data collected, of course, will be useless unless the utility is committed to act. When this commitment has been made, a system inventory of some scale will be necessary and valuable. For a PCB program manager, a "can-do" attitude is impossible to maintain without a description of what and how much is in the system needing attention. A PCB system inventory provides greater predictability to the mammoth struggle to control PCB exposure risk and allows utilities to drive their PCB programs rather than be driven by outside entities. Those utilities opting to remove equipment identified by a PCB inventory may be unable to quantify the payback in reducing liability risk, but one catastrophic failure by equipment which was not removed but could have been identified for removal is proof enough that system inventories for the cost are an excellent investment.

ACKNOWLEDGMENTS

Thanks to Deneen Dorsey, now employed by General Electric in Sacramento, California, for her invaluable assistance, perseverance and ideas during the implementation of the Grant County PUD system inventory in 1985.

REFERENCE

1. PCB Equipment, Operations and Management Reference Manual. Washington, D.C.: National Rural Electric Cooperative Association, NRECA Research Project 82-7, March 1983.

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Geographic Description of Equipment Location

☐ URBAN ☐ RURAL

Map Location and Grid _____ Verified on Map ☐ Yes ☐ No

EQUIPMENT INFORMATION

DISTRICT NUMBER		#		#		#		
TYPE	CABLE #	Pole Mount	Submersible	Pole Mount	Submersible	Pole Mount	Submersible	
		Pad Mount	Vault	Pad Mount	Vault	Pad Mount	Vault	
	VAULT #	Unit Rating		Unit Rating		Unit Rating		
PCB LABEL ON UNIT		☐ Yes ☐ No		☐ Yes ☐ No		☐ Yes ☐ No		
NON - PCB LABEL ON UNIT		☐ Yes ☐ No		☐ Yes ☐ No		☐ Yes ☐ No		
OIL LEAKING		☐ Yes ☐ No		☐ Yes ☐ No		☐ Yes ☐ No		
NAME PLATE DATA		Serial No		Serial No.		Serial No.		
☐ NO NAME PLATE		Oil Type		Oil Type		Oil Type		
		Manufac		Manufac		Manufac		
TANK CONDITION		☐ Bulged		☐ Bulged		☐ Bulged		
		☐ Damaged		☐ Damaged		☐ Damaged		
		☐ Oil Spot on Tank		☐ Oil Spot on Tank		☐ Oil Spot on Tank		
		☐ OLD Unit		☐ OLD Unit		☐ OLD Unit		
		☐ NEW Unit		☐ NEW Unit		☐ NEW Unit		
		☐ Satisfactory		☐ Satisfactory		☐ Satisfactory		

Road Location _____ Business Name.

☐ Highway ☐ Residential ☐ Intersection
☐ County Rd. ☐ Driveway ☐ Other
☐ City Street ☐ Alley

DENSITY
☐ Access ☐ Parking ☐ Sidewalk
☐ High Traffic ☐ Shopping

IMMINENT HAZARDS/EXPOSURE

☐ Raw Food/Food Processing ☐ Water ☐ Ventilation System ☐ Open Storage
☐ Wholesale/Retail Food/Food ☐ Human Health & Safety ☐ Hospital/Health Care

AREA CONDITION		GROUND CONDITION	WATER TYPE
☐ COMMERCIAL	☐ RECREATIONAL	☐ IRRIGATED	☐ WETLANDS ☐ DRAINAGE DITCH
☐ Retail Outlet	☐ INDUSTRIAL	☐ RESIDENTIAL FOOD GARDEN	☐ POND ☐ STORM SEWER
☐ Dairy	☐ AIRPORT	☐ FARM CROPS	☐ LAKE ☐ RESERVOIR
☐ Meat	☐ UNDEVELOPED	☐ PASTURE	☐ CREEK ☐ SALT
☐ Feed Grain	☐ OTHER	☐ GRAVEL	☐ RIVER
☐ Grocery		☐ PAVEMENT	☐ IRRIGATION DITCH
☐ Warehouse		☐ GRASS/LAWN	☐ POOL
☐ Restaurant		☐ SAND/DIRT	☐ PATHWAY
☐ EDUCATIONAL		☐ UNDISTURBED	Describe _____
☐ RESIDENTIAL		☐ LANDSCAPED	
☐ AGRICULTURAL	☐ MAILBOX		
☐ Farm ☐ Orchard	☐ GARBAGE		
☐ Ranch ☐ Vineyard			

COMMENTS

☐ District Numbers Missing on Equipment
☐ Couldn't locate equipment on Distribution Map
☐ Equipment on pole has different District Number
☐ See Distribution Map for Detailed Comments

Distance from equipment also to nearest facility (House, Bldg) or IMMINENT HAZARD: _____

DEVICE on Private Property
PHOTO TAKEN Roll - _____ Frame - _____
Cable/Telephone Contacts to POLE: ☐ Yes ☐ No

Crew Member Signature _____ Date _____ Reviewed By _____ Date _____

Figure 1. Geographic Description of Equipment Location field report form. Shaded areas indicate imminent hazards.

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Table 1

DATA ELEMENTS FOR PCB SYSTEM INVENTORY

Electrical equipment identification:

- Utility number
- Type of equipment
- Type of installation
- kVA size
- Manufacturer
- Serial number
- Year of manufacture
- Leaks
- Presence of PCB labels
- Equipment condition
- Height
- Nameplate fluid type
- Fluid capacity

Geographic location identification:

- Section, Township, Range
- Road address
- Business or plat name
- Map or drawing number
- Geographic grid number
- Vault number
- Pole location

Exposure risk categories:

- Density of use - 3 points
- Imminent hazards - 4 or 5 points
- Pathways - 5 points
- Distance - 3 points

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FIELD DETERMINATION OF PCB IN GROUNDCOVER

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ABSTRACT

A reliable and quick method to determine if groundcover contains chlorides equivalent to that in 50 or more mg/Kg of PCB contaminated solid has been developed. The method is based on a solvent extraction technique and a subsequent testing of the solvent extract with the commercially available "Clor-N-Oil 50" PCB kit. The test procedure was adjusted to give a positive response to soils contaminated with 50 mg/kg of Aroclor 1242. The method has been successfully applied to soils of different types. It requires however, minimum reagent and sample temperatures of 15°C, and pre-drying of the solids if water content is greater than 30 g/kg. The use of this test method after a PCB spill occurrence will reduce the growth rate of PCB contaminated solids stored in Ontario Hydro locations.

Introduction

Polychlorinated biphenyls have been used in North America in the manufacture of a wide variety of products such as paints, plastics and lubricants. Their most important use however has been in the electrical industry where they have been used as major components of dielectric fluids in power transformers and power factor correction capacitors. Because of concerns relating to environmental pollution and possible health hazards, the production of PCBs and their use in manufacturing industries was stopped , while their use by electrical utilities has been drastically restricted. In Canada, the use of PCBs is restricted to PCBs in service in closed systems purchased prior to 1980. In Ontario, PCBs and PCB contaminated equipment no longer in service must be stored in approved waste storage facilities.

Onterio Hydro, with a large PCB inventory, has undertaken several programs to find solutions to the problems associated with the management of PCBs. Specifically, research progrems have been carried out to evaluate the feasibility of retrofilling askarel filled power transformers, to design and build a mobile unit to decontaminate PCB contaminated insulating oil, to develop methods to decontaminate PCB contaminated soil and other materials, and to develop a quick and reliable method to determine the degree of contamination of groundcover following a PCB spill.

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The absence of an approved PCB destruction facility for treating solid wastes in the Province of Ontario and the lack of an efficient method to accurately determine on-site the degree of contamination of groundcover after a PCB spill have produced a large volume of PCB solid wastes at Ontario Hydro facilities. To reduce such inventory growth, Ontario Hydro developed a quick and reliable method to determine if groundcover contains chlorides equivalent to that in 50 or more mg/kg of polychlorinated biphenyls contaminated solid/1/.

Test Method Description

The test method consists of extracting the PCB from the soil sample with an appropriate solvent and analyzing the solvent extract with the commercial "Clor-N-Oil 50" test kit. Representative samples of groundcover from a spill location are weighed to the nearest 0.1 g. Stoddard solvent meeting specification CAN-2-3.8 M and equivalent to 70 percent by weight of the solid is added to the soil and the mixture is vigorously shaken for one minute. After allowing the mixture to settle for two minutes a 5 ml aliquot of the supernatant solvent extract is added to Tube 1 (with black cap) of the "Clor-N-Oil" test kit. Steps two to five in the "Clor-N-Oil" instruction sheet are then followed/2/.

The colours developed with solvent extracts from soils contaminated with PCBs vary in some degree from those shown on the "Clor-N-Oil" bulletin. Solvent extract from soil containing 50 mg/Kg or more of the common transformer Aroclor 1260 will produce a deep yellow to colourless solution. Any tinge of pink, mauve or blue developed indicates 50 mg/Kg or less of Aroclor 1260 in the soil. If however the suspected PCB soil contaminant is capacitor Aroclors 1242 or 1016, or if the type of PCBs is not known, then yellow to colourless or yellow with a tinge of pink indicates 50 or more mg/Kg of PCBs; while a blue, mauve or pink without a dominant yellow indicates less than 50 mg/Kg of PCBs.

Test Results and Discussion

The test method has been applied to PCB contaminated soil of different types with different PCBs and moisture contents. Some of the results obtained with the PCB screening test using Aroclor 1260 are summarized in Table 1. Although, as observed in the table, the solvent/soil ratio gives positive results when testing soil contaminated with Aroclor 1260 at 40 mg/Kg, this ratio was maintained to get a safe range in case the contaminating PCB was one containing less chlorine, such as Aroclor 1242.

Sampling is a critical step in analyzing PCB soil contamination since the solid is not homogeneous and the contaminant is not uniformly distributed in the soil. To minimize the error in obtaining a good representation of the contaminated soil, it is desired to have the largest sample size within the practical limits of the analytical technique. Our experiments show that soil samples between 250 and 350 g give reliable and consistent results, while significantly smaller sample sizes tend to give inconsistent and erroneous results; and larger soil samples are impractical. Therefore sample sizes between 250 and 350 g soil are recommended for this test method.

The results show that consistent responses were obtained with different types of soils, pasture, clay, gravel, sand or a combination of them. This is in good agreement with the results obtained in a previous study/3/ which showed that from 95 to 99% of the original content of PCB in clay, sand or gravel can be

extracted by this solvent at ambient temperature (~20°C). This difference in the PCB extraction efficiency of the solvent is not significant enough to change the kit's response.

As expected however, and as observed in the table, excessive moisture in the soil will affect the colour indication. The presence of significantly greater than 30 g/Kg of water may give "false negative" results because of a reduction of the PCB extraction efficiency of the solvent. Water in excess of 30 g/Kg in the soil should be removed from the sample by careful drying at a moderate temperature to obtain a more accurate result.

The presence of inorganic chloride compounds such as sodium chloride and calcium chloride in the soil will not give "false positive" PCB results. The low solubility of these salts in the PCB extracting solvent prevents their removal from the soil in significant amounts to interfere in the PCB determination.

Storage of the kits at low temperatures (-18°C) and allowing them to return to ambient conditions (25°C) prior to their use does not affect the kit's performance. However the use of PCB kit at low temperatures (10°C or lower) gave inconsistent results and is not recommended. Moreover, the PCB extraction efficiency of the solvent is reduced at lower temperatures and may produce incorrect negative results.

Conclusions

A reliable field test method to determine if groundcover contains 50 or more mg/Kg of PCB has been developed. The method is based on the extraction of PCB from any type of soil with a stoddard solvent meeting the specification CAN-2-3.8M and analysis of the solvent extract by means of the "Clor-N-Oil 50" PCB commercial kit. Water content in the soil lower than 30 g/Kg and a minimum test and reagent temperature of about 15°C are required to ensure accurate results. The test method is designed to give a positive response to any soil containing chlorine in organic compounds equivalent to a minimum of 50 mg/Kg of Aroclor 1242. With more highly chlorinated PCBs such as Aroclor 1254 or Aroclor 1260 a safe "false positive" indication will be obtained with soils containing between approximtely 35 and 50 mg/Kg PCB. When determining PCB levels in contaminated soils, false "positive results" will represent only a small fraction of all tests carried out.

References

1. Ferrie, J.S., "Field Determination of PCB in Groundcover," Ontario Hydro Research Report 83-286-H (1986).

2. Dexsil Chemical Corporation, "Instructions for CLOR-N-OIL 50 PCB Screening Kit".

3. Janis, W.J., "Removal of PCB from Contaminated Soils and Associated Materials - Part I," Ontario Hydro Research Report 83-429-K (1983).

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Table I

Screening Test Results For
PCB Contaminated Soils At Different Conditions

Sample Number	Soil Type	Dry Soil Weight (g)	PCB Content A-1260 (mg/Kg)	Water in Soil (g/Kg)	Solvent/Soil Ratio (g/g)	Colour Developed	PCB Level Indicated (mg/Kg)
1	Pasture	115.5	40	13	0.7	Yellow	> 50
2	Pasture	115.5	40	13	0.8	Yellow with pink tinge	> 50 (borderline)
3	Pasture	115.5	40	13	0.9	Yellow with deeper pink then #2	< 50 (borderline)
4	Pasture	115.5	40	13	1.0	Yellow with deeper pink then #3	< 50
5	Pasture	323.7	40	30	0.7	Yellow with pink tinge	50 (borderline)
6	Pasture	323.7	40	50	0.7	Deeper pink then in #5	< 50
7	Pasture	323.7	40	100	0.7	Purple	< 50
8	Pasture	323.7	40	100	0.7	Purple	< 50
9	Pasture	258.1	50	30	0.7	Yellow	> 50
10	Pasture	258.1	50	50	0.7	Pink-Purple	< 50
11	Pasture	218.7	60	100	0.7	Yellow with pink tinge	50 (borderline)
12	Pasture	183.1	70	100	0.7	Pink-Purple	50 (borderline)
13	Clay	258.1	50	25	0.7	Yellow	> 50
14	Clay	258.1	50	25	0.7	Yellow	> 50
15	Clay	323.7	50	25	0.7	Yellow with pink tinge	50 (borderline)
16	Gravel	134.3	35	14	0.7	Yellow	> 50
17	Gravel	258.1	50	14	0.7	Yellow	> 50
18	Gravel	323.7	40	14	0.7	Yellow with pink tinge	50 (borderline)

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APPENDIX

PCB Seminar

10/06/87-10/09/87
Hyatt Regency
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List of Attendees

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