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PHYSICAL PROPERTIES OF CONTAMINATED TRICHLOROETHYLENE AND 1,1,1-TRICHLOROETHANE

R. D. Holt

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R. D. Holt, Project Leader

Project Team:
C. R. Cooperrider
F. J. Kamler

Technical Communications
Kansas City Division

Allied-Signal Aerospace Company

AlliedSignal

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The specific gravity, volume change, dielectric constant, dissipation factor, boiling point, and nonvolatile residue carryover during distillation was measured for various contamination levels of rosin in trichloroethylene and 1,1,1-trichloroethane. Solvent stabilizers and the vapor pressure of solvents were examined. The effects of unknown contamination in solvents from manufacturing departments were measured. The theoretical effects of oil contamination on the boiling point are discussed.

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Kansas City Division
P.O. Box 419159
Kansas City, Missouri
64141-6159

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SUMMARY

The physical properties of trichloroethylene (TCE) and 1,1,1-trichloroethane (TCA) contaminated with rosin were characterized. The specific gravity, dielectric constant, dissipation factor, boiling point, and nonvolatile residue carryover during distillation were measured for various contamination levels of rosin in TCE and TCA.

The boiling point of the solvents is affected more by the solvent stabilizers than by rosin at rosin contamination levels of less than 30 percent by weight. In the 35 and greater percentage contamination range, rosin has the dominant effect on the solution boiling point. Specific gravity changes sufficiently with contamination so that it can be used to measure the amount of rosin in the solvent. The dielectric constant of rosin-contaminated TCE was discovered to be very constant over the entire range of contamination levels, 0 to 50 percent by weight. The dielectric constant of pure and contaminated TCA is significantly different from that of TCE. This difference can be used to easily and quickly identify an unknown solvent type for solvent waste recycling.

The data obtained on this project were essential to support the design of the new vapor-containing cleaning system; the system will meet the demands of new environmental and health restrictions and maintain the high level of cleaning needed on electronic assemblies. These data eliminated the unknowns in the area of contamination carryover during distillation and in physical property changes that could be monitored to indicate solvent contamination level.

The effects of unknown contamination in solvents from manufacturing departments were measured. This included measuring the boiling point, nonvolatile content, nonvolatile carryover during distillation, dissipation factor, and dielectric constant.

The effects of oil contamination on the boiling point were theoretically examined. Laboratory analysis of solvents contaminated with oils is an important effort that should be completed to support the cleaning of machined parts. No future work in characterization of contaminated solvents is planned as part of this project.

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DISCUSSION

SCOPE AND PURPOSE

The purpose of this project was to determine the physical properties of contaminated trichloroethylene (TCE) and 1,1,1-trichloroethane (TCA). This project was in support of the design of the new vapor-containing cleaning system to meet the demands of new environmental and health restrictions and maintain the high level of cleaning needed on electronic assemblies.

The scope of this project was limited to laboratory evaluation of rosin-contaminated solvents and a literature search for the effects of other contamination types. Two types of soldering flux were the only rosin sources investigated. The primary focus of this project was to provide data accurate enough to support the design of the new vapor-containing cleaning system. This work was accomplished during September and October 1988.

ACTIVITY

Background

The majority of electronic assemblies built at Allied-Signal Inc., Kansas City Division (KCD), are required by specification to be cleaned with chlorinated or fluorinated (chlorofluorocarbon) solvents. Evaluation of solvent cleaning has shown that TCE and TCA, which are chlorinated solvents, are the best solvents for removing flux and other organic contaminants found on electronic and mechanical assemblies. The coming severe restrictions or banning of fluorinated solvents (CFCs),[1-4] because of their detrimental effects on the ozone layer, make their use as cleaning agents less desirable. The toxicity and lowering of exposure limits on chlorinated solvents will restrict their use and require controlled cleaning techniques.

The need to continue to use these solvents to maintain adequate product cleanliness has prompted the design and development of a vapor-containing cleaning system. The initial goal for the cleaning system is to maintain the distilled solvent at a technical grade purity level. The ability to maintain the solvent at any specified quality level will be limited by the physical properties of the solvents and by the monitoring methods. The physical properties of contaminated solvents must be known before a cleaning system that distills and recycles the solvent can be designed and built.

Theoretical analysis can estimate the amount of change in some properties of solvents with varying contamination levels, but the validity of any theoretical model must be established with laboratory data. Laboratory tests were performed at KCD to experimentally determine how the boiling point, specific gravity, volume, nonvolatile residue carried over during distillation, dielectric constant, and dissipation factor change with different percent-by-weight levels of solder flux (rosin)-contaminated solvent.

Pure Solvent and Contaminant Properties

The typical properties of TCE and TCA are given in Table 1. Specifications for cleaning at KCD typically require reagent grade chlorinated solvents for spray cleaning applications and technical grade solvents for vapor-degreasing applications. The technical grade solvent specifications are similar to reagent grade solvents (Table 2).

The TCE used in all of the laboratory experiments in this report was from one 5-gallon can of technical grade solvent. The TCA used in all the laboratory experiments was from one lot of technical grade solvent supplied in 1-gallon glass containers.

Solder flux is the major contaminant on electronic assemblies. Flux is mainly rosin with isopropyl alcohol as a solvent carrier. Rosin is a solid separated from pine tree sap by distillation. The color of pure rosin is primarily a result of oxidation which turns it from water white clear to a dark brown.[7] Rosin is 80-to 90-percent abeitic acid, with 10- to 15-percent pimaric acid. The boiling points of these resin acids are 250 and 282°C, respectively.[12] These resin acids and their isomers easily dissolve in chlorinated solvents, and this is the main reason why chlorinated solvents are so effective in removing rosin.

For soldering applications at KCD, flux rosin must meet three military specifications: MIL-F-14256, QQS-571, and LLL-R-626. The typical properties of this rosin are given in Table 3.[7] The rosin used in the experiments meets MIL-F-14256 specification as types R and RMA. The solvent was removed by elevating the flux temperature to 80 to 90°C for 16 to 48 hours (90°C is well below the boiling point of the flux and rosin).

Solvent Stabilizers

Patents reveal that various solvent manufacturers use different stabilizers (Table 4). It is the purpose of stabilizers to keep the solvent from becoming acidic and from subsequent decomposition. The presence of these stabilizers would be expected to alter some of the physical properties of the solvent, especially the boiling point. The boiling point can be depressed or elevated according to Raoult's Law if the contaminant and solvent mixtures behave as ideal solutions.

Table 1. Typical Properties of Trichloroethylene and 1,1,1-Trichloroethane

Properties	Trichloroethylene	1,1,1-Trichloroethane
Chemical Formula	C_2HCl_3	$C_2H_3Cl_3$
Molecular Weight	131.4	133.4
Boiling Point at 760 mm Hg	189°F 87°C	165°F 74°C
Freezing Point	-124°F -86.7°C	-34°F -37°C
Specific Gravity at 25°C	1.456	1.300 to 1.325
Pounds per Gallon at 25°C	12.11	10.97
Vapor Density (air = 1.00)	4.53	4.55
Specific Heat (1 Btu/lb°F = 1 cal/g°C)	0.22 Btu/lb°F	0.258 Btu/lb°F
Heat of Vaporization at Boiling Point		
cal/g	56.4	56.7
Btu/lb	101.6	102
Solubility (g/100 g) at 25°C, H_2O in Solvent	0.04	0.05
Flash Point		
Tag Open Cup, ASTM Method D-1310	none	none
Tag Closed Cup, ASTM Method D-56	none	none
Flammable Limits (volume % of solvent in air) at 25°C		
Lower Limit	8.0	7.5
Upper Limit	10.0	15

Gas chromatograms were made of as-received solvent, distilled solvent, and undistilled solvent (that is, solvent remaining in the flask after half of it was distilled). (See Table 5 and Figures 1 and 2.) The chromatograms identified most of the stabilizers in the solvents used in this study. Some stabilizers boiled out of solution quickly while others remained; this change in stabilizer concentration affects the boiling point of the solvent.

Table 2. Specifications for Reagent Grade and Technical Grade Solvent

Property	Specification
Trichloroethylene, Reagent Grade, American Chemical Society Specifications 1987	
Color (APHA)	Not More Than 10
Density (g/mL) at 25°C	Not Less Than 1.455 or More than 1.460
Boiling Range	Entirely Within 1.0°C Range Including 87.1°C ±0.1°C
Residue After Evaporation	Not More Than 0.001%
Tritratable Acid	Not More Than 0.0001 meq/g
Tritratable Base	Not More Than 0.0003 meq/g
Water (H_2O)	Not More Than 0.02%
Heavy Metals (as Pb)	Not More Than 1 ppm
Free Halogens	To Pass Test--See Specification
Trichloroethylene, Technical Grade, Mil Spec. O-T-634C	
Specific Gravity at 25°C	1.450 to 1.460
Distillation Range:	
Initial Boiling Point Minimum	86.0°C
Minimum 95% Distilled, Maximum	87.5°C
Dry Point, Maximum	90.0°C
Acidity (as HCl), Weight Percent, Maximum	0.01 Regular Grade 0.001 Vapor Degreasing Grade
Alkalinity (as NaOH), Percent Weight, Maximum	0.015
Water, ppm, Maximum	100
Appearance	Clear and Free of Suspended Matter
Color, APHA,	15
Nonvolatile residue, Weight Percent, Maximum	0.002 Regular Grade 0.010 Vapor Degreasing Grade
Acid Acceptance (as NaOH), Weight Percent, Minimum See Mil. Spec. for Details	0.160
Trichloroethane, Pure Grade	
Nonvolatile Content, ASTM-D-2109	0.002% Maximum
Water Content, ASTM-D-1364	0.05% Maximum
Distillation Range, ASTM-D-1078	72 to 88°C
Inhibitor Presence, ASTM-D-2943	To Pass Test With No Reaction

Table 2 Continued. Specifications for Reagent Grade and Technical Grade Solvent

Property	Specification
Trichloroethane, Technical Grade, Mil. Spec. O-T-620C	
Appearance	Clear and Free of Suspended Matter
Color (Maximum)	15 Platinum Cobalt Units
Distillation Range:	
Initial Boiling Point, Minimum	70°C
Dry Point, Maximum	88°C
Odor	Characteristic Mild Sweet No Residual Odor
Water, ppm, Maximum	100
Acidity (as HCl), Weight Percent, Maximum	0.001
Alkalinity (as NaOH), Percent Weight, Maximum	0.015
Nonvolatile Matter, Percent, Maximum	0.0101
Corrosion of Aluminum Alloy	No Pitting, Etching, or Discoloration
Specific Gravity at 25°C. See Mil. Spec. for Details.	1.284 to 1.324

Past studies at KCD have shown that stabilizers for TCE contain butylene oxide and cyclohexene oxide for vapor degreasing and solvent recycling. Cyclohexene oxide tends to stay in the sump where the stabilizer is needed the most, whereas most of the butylene oxide is easily carried over into the distillate during distillation.

Specific Gravity

Specific gravity measurements were made on TCE and TCE at various rosin-contamination levels between 0 and 50 percent by weight. The measurements were made at 25°C following ASTM D 2111-85, method B, using hydrometers 129H through 133H. The data are presented in Table 6 and graphed in Figure 3. The change in specific gravity with rosin in solution fits a straight line in the 0- to 30-percent range for TCE and TCA. Using a simple least squares fit, the equations describing this change are

Table 3. Typical Properties of Rosin*

Property	Specification	
Specific Gravity		
22°C	1.07 to 1.09	
210°C	0.93	
Composition	80- to 90-Percent Abietic Acid, 10- to 15- Percent Pimaric Acid, Remainder Is Isometric Resin Acids of Abietic Acid	
Softening Point, Minimum	70°C	ASTM E 28
Boiling Temperature		
250°C	Abeitic Acid	
282°C	Pimaric Acid	
Acid Number, Minimum	160	ASTM D 465
Saponification Number, Minimum	166	ASTM D 464
Insoluble Matter in Toluene	0.05 Percent by weight	ASTM D 269
Volume Resistivity	$>10^{13}$ at 0°C $\sim 10^{13}$ at 50°C $\sim 10^{9}$ at 150°C	
Viscosity at 110°C	15 Ns/m^2 (150 Poise)	

*Data from military specifications MIL-F-14256, QQS-571, LLL-R-626, and reference 7.

$$Sg\ (\text{solution}) = m\ W\% + b\ , \tag{1}$$

where

m = -0.4738 and b = 1.4505 for TCE,

m = -0.2791 and b = 1.3124 for TCA, and

W% (<30%) is the weight percent of rosin in the solution.

According to this least squares fit equation, the specific gravities of TCE and TCA at a zero rosin concentration are 1.4505 and 1.3124, respectively. This is very close to the measured values of 1.4520 and 1.3130. These clean solvent specific gravity values are within the technical grade requirements.

The ranges of specific gravity for technical grade solvents are 1.450 to 1.460 for TCE and 1.284 to 1.324 for TCA. The specific gravity could be used to measure the amount of contamination in the sump to within ±1.2 weight-percent of rosin contamination of TCE and within ±0.8 weight-percent of rosin contamination of TCA.

Table 4. Stabilizer Packages of Trichloroethylene, 1,1,1-Trichloroethane, and Perchloroethylene

Solvent	Stabilizer Package	Patent
Trichloroethylene	Pyridine, p-Tertiary-butylcatechol, Diisopropylamine	2906782,1959, Sicedison SPA. IT.
Trichloroethylene	Butylene Oxide, Pyridine, Ethyl Acetate, Isobutyl Alcohol	2887516, 1957, Sicedison SPA. IT.
Trichloroethylene	Propargyl Alcohol Pyra-ole	2803676, 1957, Dow Chemical Co.
Trichloroethylene	1. Glycidol or Glycerin 2. p-Methoxy Phenol, Ditert-butyl, 4-Methyl Phenol p-Tert-butylphenol or Thymol 3. Diisoprylamine 4. Epichlorohydrin or Butylene Oxide	3852367, 1974, Central Glass Co., JP.
Trichloroethylene	Substitute 1,3-Dioxane Products, Acetaldehyde Dimethyl, Hydrozone	4466903, 1984, PPG Industries, Inc.
Trichloroethylene	1. Isobutyl Alcohol 2. Epichlorohydrin, Butylene Oxide, Diisopropylamine Oxide, or Cyclohexene Oxide	3188355, 1965, Dexter Corp.
Trichloroethylene	Alkoxykanol, n-Methyl Pyrrole, Butylene Oxide, and/or Epichlorohydrin	3269953, 1966, Imperical Chem. Ind.
Trichloroethylene	1. Triethylamine, Dimethylaniline, or Diisopropylamine 2. Butylene Oxide or Styrene Oxide 3. Trimethyl-Amonium Acetate	3314892, 1967, Candian Ind. LTD

Table 4 Continued. Stabilizer Packages of Trichloroethylene, 1,1,1-Trichloroethane, and Perchloroethylene

Solvent	Stabilizer Package	Patent
Trichloroethylene	Ethyl Acetate, Acetonitrile, or Pytazine n-Methyl Pyrrole	4368338, 1983, Dow Chemical Co.
Trichloroethylene	1. Cyclohexene oxide 2. Butylene oxide 3. n-propanol and two unknown compounds	Lab Analysis, Ref. 39. Sample from PPG Industries, Inc.
Trichloroethylene	1. Butylene oxide 2. Epichlorohydrin 3. Ethyl acetate 4. n-propylamine and three unknown compounds	Lab Analysis, Ref. 39. Sample From Dow Chemical Co.
1,1,1-Trichloroethane	1,4-Dioxane, Butylene Oxide, 3-Butyn-1-ol, Ethyl, or Isopropyl Nitrate	4394284, 1963, Dow Chemical Co.
1,1,1-Trichloroethane	1,4-Dioxane, Nitromethane, 1,2-Butylene Oxide, 1,2-Epoxy Hexane	916129, 1963, Dow Chemical Co.
1,1,1-Trichloroethane	1,4-Dioxane, Sec-butyl Alcohol, 1,2-Butylene Oxide, 1,2-Epoxy Hexane	916129, 1963, Dow Chemical Co.
1,1,1-Trichloroethane	1,4-Dioxane, Nitromethane, 1,2-Butylene Oxide	Lab Analysis, Ref. 39. Sample from Dow Chemical Co. Like Pat.# 916129.
Perchloroethylene	Tert-butyl Glycidyl Ether	Lab Analysis, Ref. 39 Sample from Dow Chemical Co.
Perchloroethylene	1-(1-methyl-1H-pyrrol-2yl)- Ethanone and Cyclohexene	Lab Analysis, Ref. 39. Sample from PPG Industries, Inc.

Table 5. Gas Cromatographic Measurements of Solvent Stabilizers and Impurities

Stabilizer, Impurities, or Solvent	Percent in Solution*		
	As-Received	Distilled	Undistilled
Trichloroethylene			
1. Butylene Oxide	0.50	0.78	0.22
2. 1,1,1-Trichloroethane	0.022	0.089	0.015
3. 1,1,2-Trichloroethane	0.099	0.046	0.15
4. 1,1,2-Trichloroethylene	96.24	95.41	98.89
5. Unknowns	3.14	3.67	0.72
Sum (Numbers 1-5)	100.001	99.995	99.995
1,1,1-Trichloroethane			
1. Cyclohexane	0.0049	0.0088	0.0046
2. Butylene Oxide	0.32	0.38	0.25
3. 1,3-Dioxolane	0.84	0.95	0.75
4. 1,1,2-Trichloroethylene	0.0087	0.0074	0.013
5. Secondary Butanol	0.97	1.12	0.80
6. 1,4-Dioxane	0.91	0.32	1.50
7. 1,2-Dichloroethane	0.12	0.11	0.13
8. Nitromethane	0.57	0.77	0.34
9. Nitroethane	0.0047	0.0027	0.0063
10. 1,1,1-Trichloroethane	96.25	96.34	96.21
Sum (Numbers 1-10)	99.9983	100.0089	100.0039

* Average from duplicate measurements.

The rosin contamination level could be determined to within a 0.5-percent accuracy if the clean solvent specific gravity is known. Other contaminants in the sump can affect the accuracy of rosin weight estimation, but specific gravity measurements should still provide a good estimate of the rosin contamination level.

The change in specific gravity of a pure substance with temperature can be calculated by using the following equation:

$$Sg(t) = a\,(t-25°C) + Sg(25°C)\ , \qquad (2)$$

where a is the thermal coefficient of density.' Pure TCE and TCA have a thermal coefficient of density of 0.001649 and 0.001657, respectively. The thermal coefficient of density of

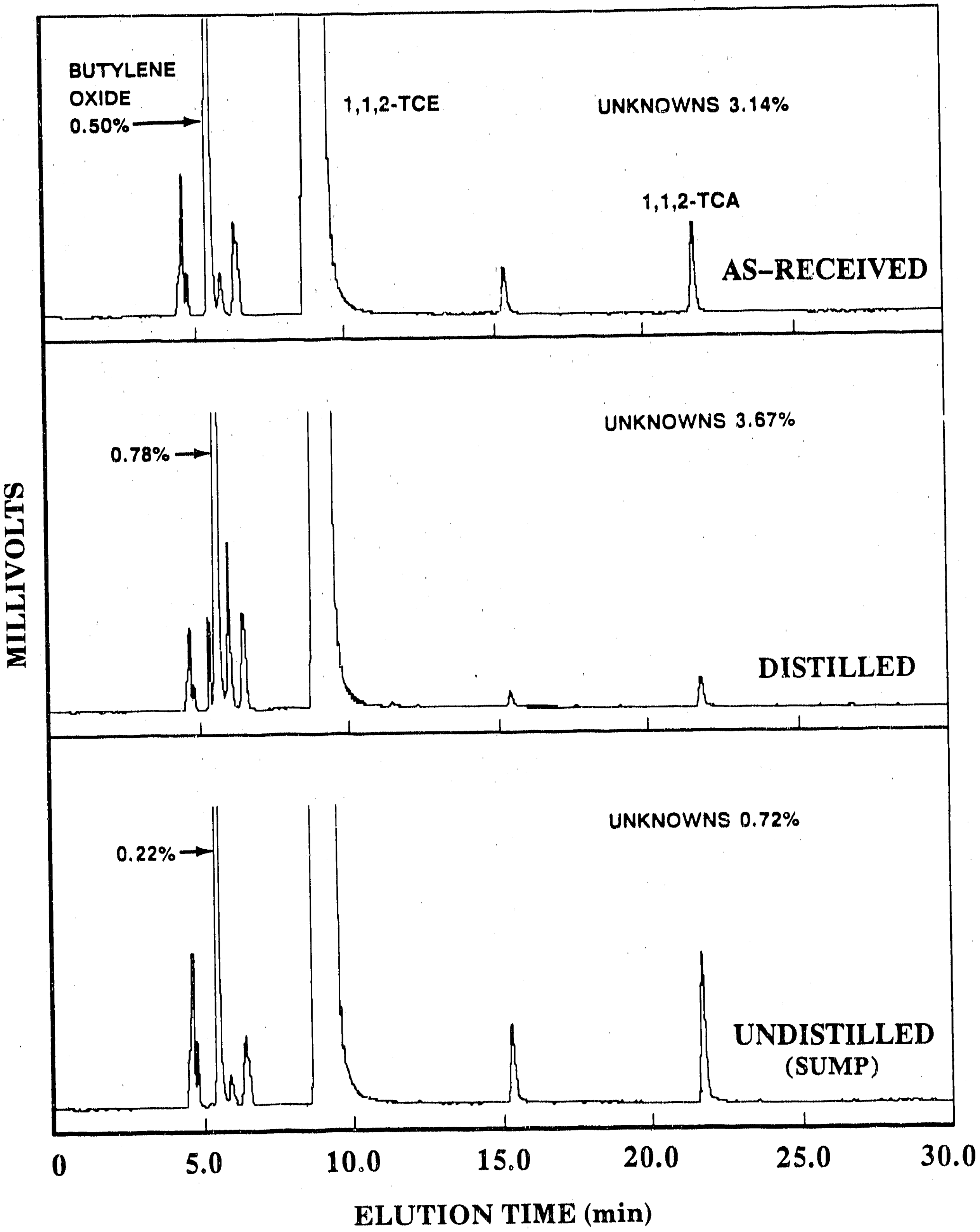

Figure 1. Gas Chromatogram of Trichloroethylene As-Received, Distilled, and Undistilled

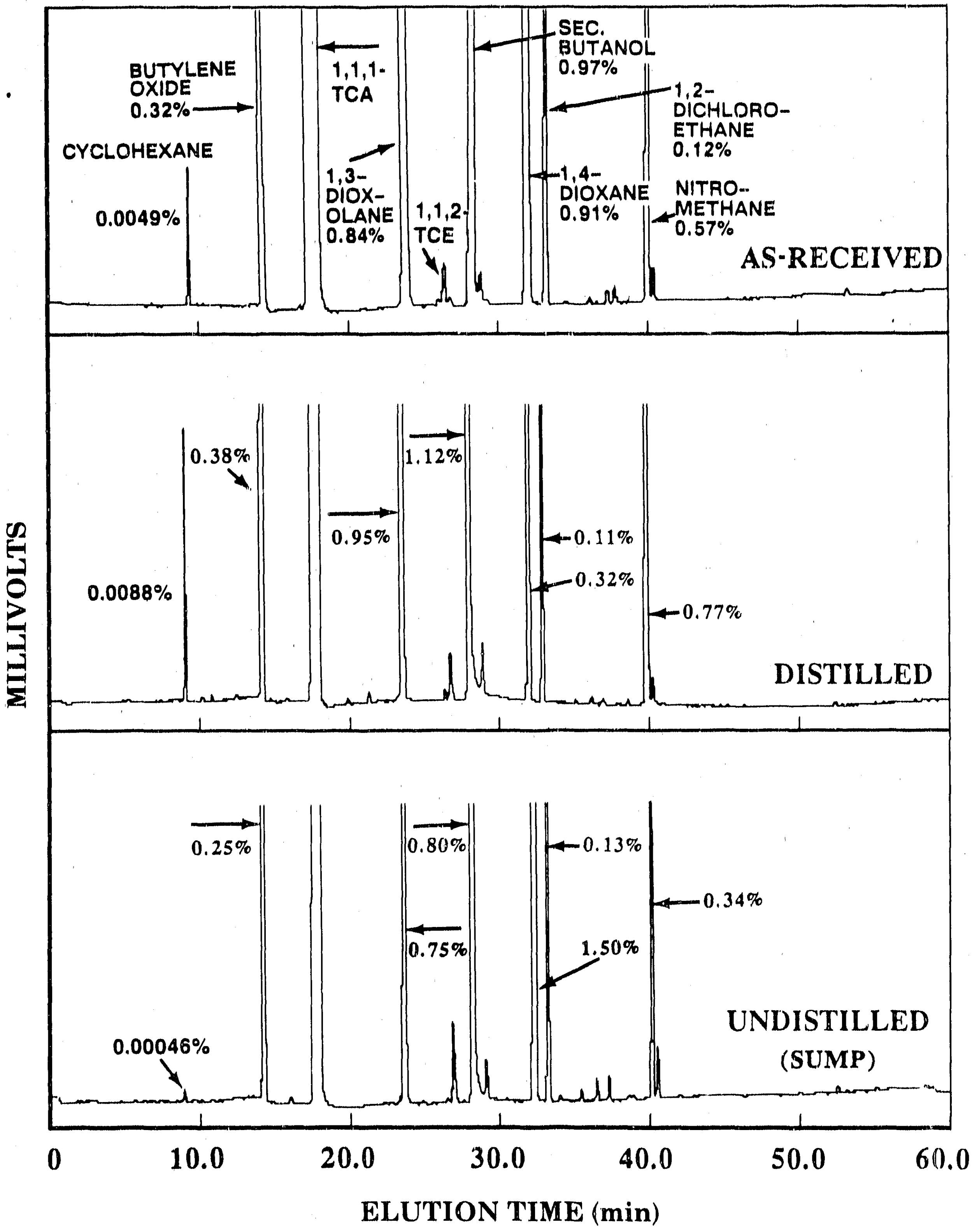

Figure 2. Gas Chromatogram of 1,1,1-Trichloroethane As-Received, Distilled, and Undistilled

Table 6. Specific Gravity and Volume Change of TCE and TCA at Various Contamination Levels by Rosin at 25°C

Rosin Contamination Level (percent by weight)	TCE			TCA		
	Specific Gravity	Rosin* Source	Volume Change (percent)	Specific Gravity	Rosin* Source	Volume Change (percent)
0**	1.4520#	--	0	1.3130##	--	0
5.0	1.4245	197	7.30	1.2973	A	6.54
10.0	1.4012	197	15.14	1.2825	A	13.75
15.0	1.3772	197	24.04	1.2686	A	21.76
20.0	1.3519	197	34.26	1.2602	A	30.24
25.0	1.3298	1544	45.59	1.2408	B	41.09
30.0	1.3101	1544	58.33	1.2274	B	52.82
30.0	1.3132	197	57.96	1.2309	A	52.39
40.0	1.2663	1544	91.11	1.2042	B	81.73
50.0	1.2437	197	133.5	1.2036	A	118.2

*This column indicates the flux from which the rosin was obtained. The alcohol carrier solvent was removed by evaporation at 80 to 90°C.
**Indicates as-received technical grade solvent.
#Average of four measurements.
##Average of six measurements.

The percent of volume change values were calculated from the measured changes in specific gravity by the following equation which is derived in the text.

$$\%\ \text{Volume Change} = \left[\frac{1}{1 - W\%}\right]\left[\frac{Sg\ (0\%)}{Sg\ (W\%)}\right] - 1$$

rosin is -0.000798 based on the two data points from Table 3. A rigorous equation relating the individual coefficient densities to that of a mixture was not found. Comparison of the coefficients suggests that the coefficient for a solution would be less than that of pure solvent for TCE or TCA.

Volume Change

The change in volume with percent-by-weight contamination can be calculated from the specific gravity measurements. The initial volume of the solvent, Vi, is given by

$$Vi = \frac{Sw}{Sg(0\%)}, \qquad (3)$$

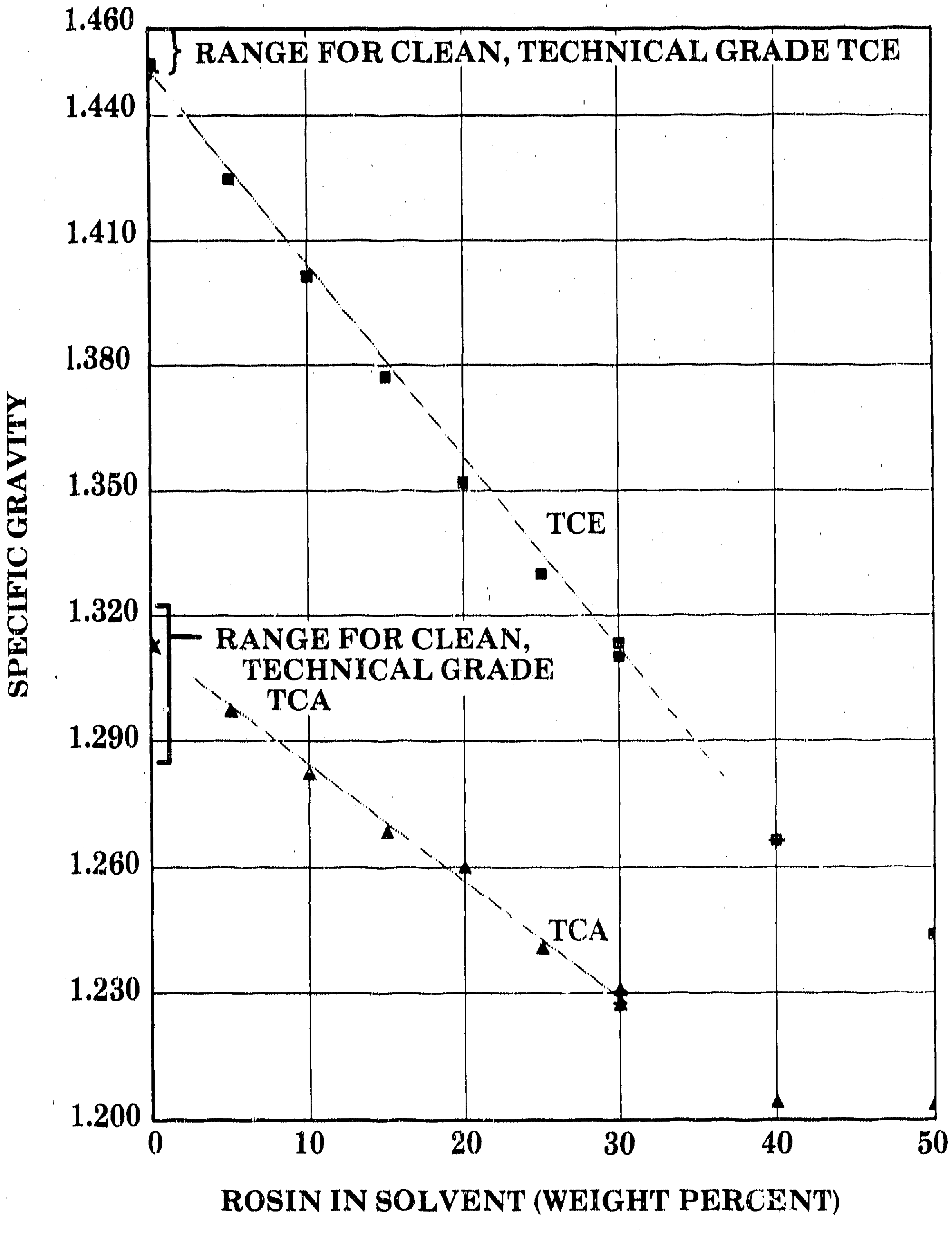

Figure 3. Specific Gravity Versus Rosin Contamination Levels for Trichloroethylene and 1,1,1-Trichloroethane at 25°C

where Sw is the weight of the pure solvent and Sg(0%) is the specific gravity of the solvent with 0 percent, by weight, contamination. The volume at some contamination level W% is given by

$$V(W\%) = \frac{[Sw+Rw]}{Sg(W\%)} , \tag{4}$$

where Rw is the weight of the rosin in solution. The contamination level W% is given by

$$W\% = \frac{Rw}{[Sw+Rw]} . \tag{5}$$

The percent volume caused by a contamination of the solvent by rosin is then given by

$$\begin{aligned} dV(W\%) &= \frac{[V(W\%)-Vi]}{Vi} \\ &= \left[\frac{[Sw+Rw]}{Sg(W\%)} - \frac{Sw}{Sg(0\%)}\right] \frac{Sg(0\%)}{Sw} \\ &= \left[\frac{1}{1 - W\%}\right]\left[\frac{Sg(0\%)}{Sg(W\%)}\right] - 1 . \end{aligned} \tag{6}$$

This equation is as accurate as the equation modeling the specific gravity, which is a function of weight percent. The results of these calculations are given in Table 6 and graphed in Figure 4.

Dielectric Constant

The dielectric constant, K, of a material is a measure of the way in which a medium responds to an electric field. It is easiest to think of the dielectric constant in terms of the capacitance increase caused by filling a parallel plate capacitor with a material of dielectric constant K. The capacitance of a parallel plate capacitor is given by

$$C = \frac{K\epsilon A}{d} , \tag{7}$$

where ϵ is the permittivity of free space, A is the area of the plates, d is the distance between plates, and fringe effects are neglected.

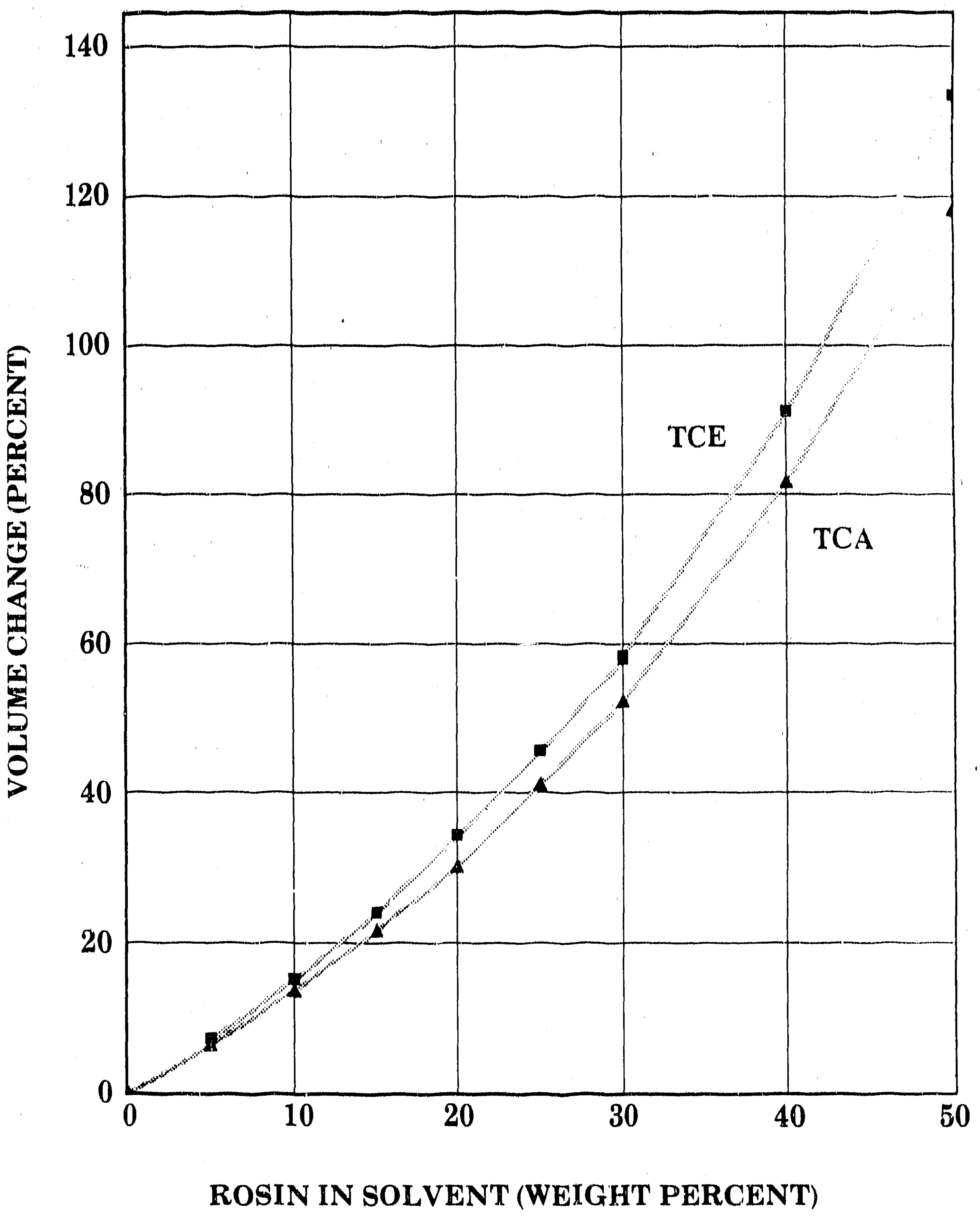

Figure 4. Volume Change Versus Rosin Contamination Levels for Trichloroethylene and 1,1,1-Trichloroethane at 25°C

The dielectric constants of contaminated solutions were measured by using a guarded-ring parallel-plate capacitor and making capacitance measurements. Capacitance measurements were made using a capacitance test cell and an automatic capacitance bridge. The test cell had a micrometer adjustment with 0.001-inch markings for the parallel plate spacing and had a guard ring to eliminate fringe effects. The parallel plate was 2.0 inches in diameter with a 0.2-inch-wide circumferential guard ring. The capacitance bridge was accurate to ±1 percent for the capacitance values measured. All measurements were made at room temperature. Capacitance measurements were made at 10 kHz, 100 kHz, and 1 MHz, with a few measurements at 1 kHz. Capacitance measurements were made at spacings of 0.050, 0.100, 0.150, 0.200, and 0.250 inch between the parallel plates. The measurements can be found in Appendix A of this report. The dielectric constant was calculated by using the equation

$$K = \frac{C_e}{C_a} , \tag{8}$$

where C_e is capacitance of the test cell filled with the solution and Ca is the capacitance of the test cell filled only with air. The results of the calculations are summarized in Table 7 and graphed in Figure 5.

The dielectric constant of reagent grade TCA is 6.44, about 0.7 below the measured dielectric value of technical grade TCA. The dielectric constant of reagent grade TCE is 4.42, very close to the measured dielectric value of technical grade TCE. The difference in the values of TCA may be caused by the stabilizers in TCA and the impurities in the technical grade solvent.

Measurements at 1 MHz and the 0.050-inch spacing were slightly higher than other measurements and were not included in the averages presented in Table 7. The dielectric constant measurements at any rosin contamination level of TCE or TCA were essentially constant from 1 kHz to 1 MHz for all spacings of the parallel plates. The dielectric constant measurements for TCA were constant from 1 kHz to 100 kHz but increased slightly at 1 MHz for all plate spacings. The difference in the solution's dielectric constant between using rosin from the two fluxes was less than 2 percent (see the 30-percent by weight contamination level data in Table 7).

The change in dielectric constant of TCE and TCA is not significant enough to provide an accurate determination of the rosin concentration in solvent for small changes in contamination level. It is remarkable how consistent the dielectric constant of TCE remained over the large range of rosin contamination levels. This consistency could be used to quickly and inexpensively distinguish between contaminated solutions of TCE

Table 7. Dielectric Constant of Rosin-Contaminated Solvents

Contamination Level As-Received (percent by weight)	Dielectric Constant (Average of Measured Values)			
	TCE		TCA	
	1 kHz to 100 kHz	1 MHz	1 kHz to 100 kHz	1 MHz
0.00 (a)	3.4684	3.4755	7.1840	7.2374
0.00 (b)	3.4950	3.5012	7.2203	7.2699
0.05	3.4682	3.4749	7.2127	7.2634
0.1	3.4690	3.4768	7.2182	7.2686
0.5	3.4731	3.4805	7.1946	7.2434
1.0	3.4735	3.4816	7.1676	7.2034
3.0	3.4861	3.4930	7.0559	7.1075
5.0	3.4969	3.5049	6.8940	6.9407
10	3.5290	3.5358	6.6728	6.7160
15	3.5484	3.5549	6.4213	6.4586
20	3.5926	3.5998	6.3555	6.3860
25*	3.6134	3.6175	6.0561	6.0815
30*	3.6259	3.6281	5.8508	5.8795
30	3.5810	3.5876	5.7741	5.8002
40*	3.6396	3.6395	5.5325	5.5183
50*	3.6126	3.6138	4.7426	4.6570

(a) Solution measured on October 11, 1988.

(b) Solution measured on November 5, 1988; same lot and container as in (a). The capacitance measuring bridge was recalibrated between the two measurements; this may account for the difference in measured values of identical solutions.

* Solutions with asterisk were contaminated with rosin from Flux B. All other solutions were contaminated with rosin from Flux A.

and TCA. The usual method of distinguishing contaminated TCE and TCA solutions requires time-consuming laboratory analysis.

Dissipation Factor

Dissipation factor is a measure of the power loss when a material is used as a dielectric in a capacitor. The losses are caused by microscopic mechanical effects resulting from the forces caused by the varying electrical field and from resistive losses in the dielectric. The dissipation factor, sometimes called the loss tangent, is defined as the ratio of the real impedance divided by the complex impedance of the dielectric.[10]

The dissipation factor was measured using the same equipment and procedure as was used for measuring the dielectric constant. The data are in Appendix B and are presented graphically in Figure 6.

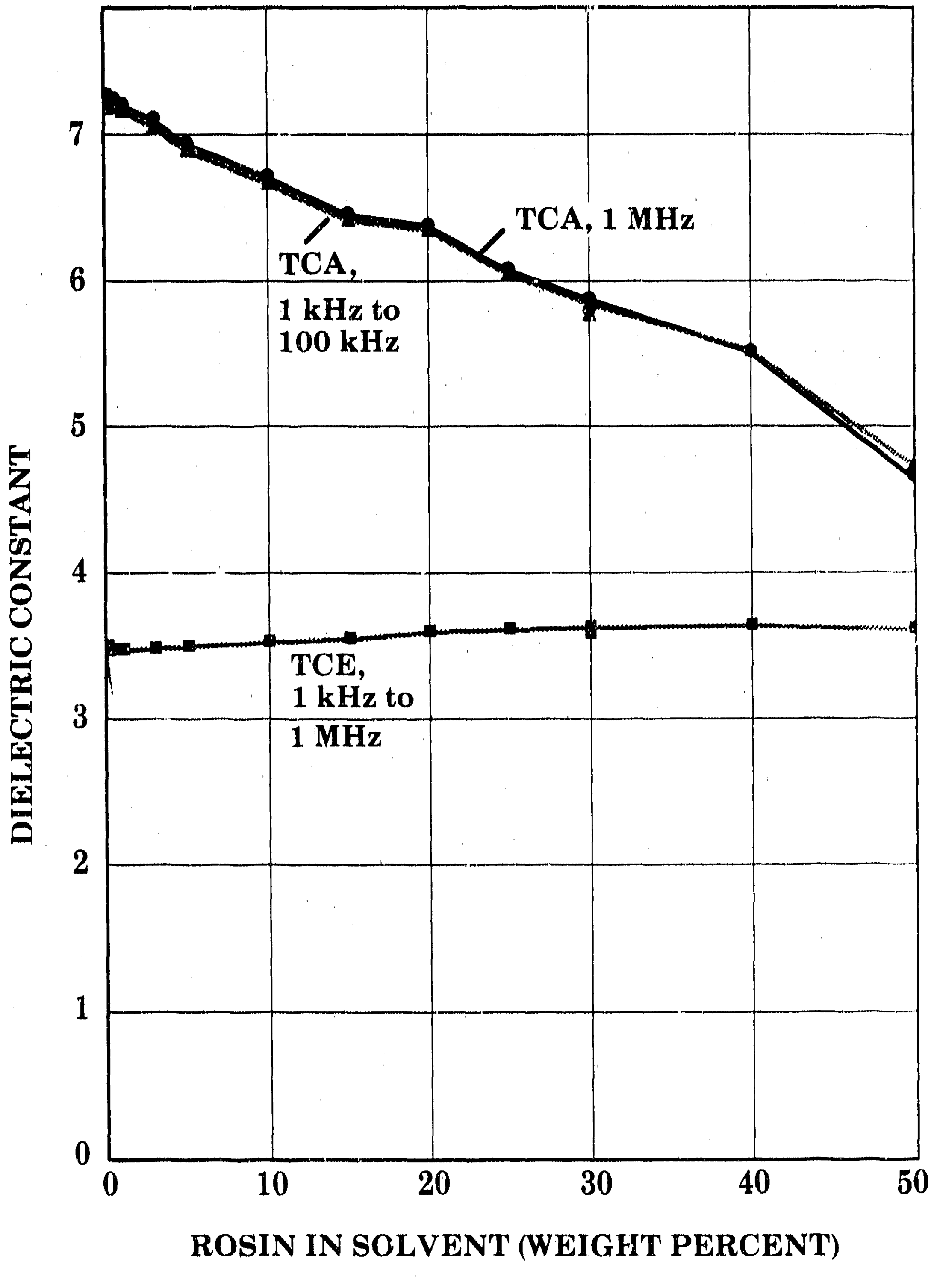

Figure 5. Dielectric Constant Versus Rosin Contamination Levels for Trichloroethylene and 1,1,1-Trichloroethane at 25°C

The dissipation factor of pure and contaminated TCE and TCA varied inversely proportional to frequency. At any frequency, the dissipation factor did change significantly in the 0- to 5-percent by weight concentration level of rosin (see Figure 6). These data show that by measuring the dissipation factor one may monitor the very low level concentration of rosin in TCA. Unfortunately, the rapid change in dissipation factor for TCE does not occur until after the 3-percent contamination level is reached. Combined measurement of the dissipation factor and dielectric constant may be the easiest and quickest way to monitor the contamination level at various concentration levels.

Boiling Point

For the purposes of this discussion, the boiling point is the measured temperature of the vapor zone as stated in ASTM D 1078-86, "Distillation Range of Volatile Organic Liquids," or it can be the measured temperature in the vapor zone (or in the solution) for some other described laboratory setup. According to the technical grade specifications (Table 2), the solvent boiling point for TCE must be 86°C minimum (186.8°F), and for TCA, the boiling point must be 70°C minimum (158°F). There is no specification upper limit on the initial boiling temperature of technical grade TCE or TCA, but there is a distillation range specified and it will be used to specify the boiling point limits.

The boiling point of any solvent would be expected to change with contamination according to Raoult's law for ideal solutions and with deviations from Raoult's law for nonideal solutions. In an ideal solution, the intermolecular attractions between solvent molecules and solute molecules are the same as the intermolecular attractions between molecules of pure solute or pure solvent.[11] Only by experimental methods can one readily determine if some contamination will interact with the solvent as an ideal solution and how the boiling point will be altered by that contamination.

Boiling Point, Oil Contamination

A brochure on vapor degreasing by Dow Chemical gives boiling points of oil-contaminated solvents in a 0- to 80-percent by weight contamination level. The graphs and data were apparently based on an earlier published paper which was a theoretical analysis and were not based on actual measurements.[12] The oils considered in the theoretical analysis were a mineral lard oil, a sulfochlorinated-lard oil, and a sulfurized-lard oil. The theoretical data from 0- to 30-percent contamination are given in Table 8 and Figures 7 and 8.

The assumption made in the theoretical model is that the mixtures of solvent and oils behave as ideal solutions. If this assumption for oils and solvents is erroneous, all the

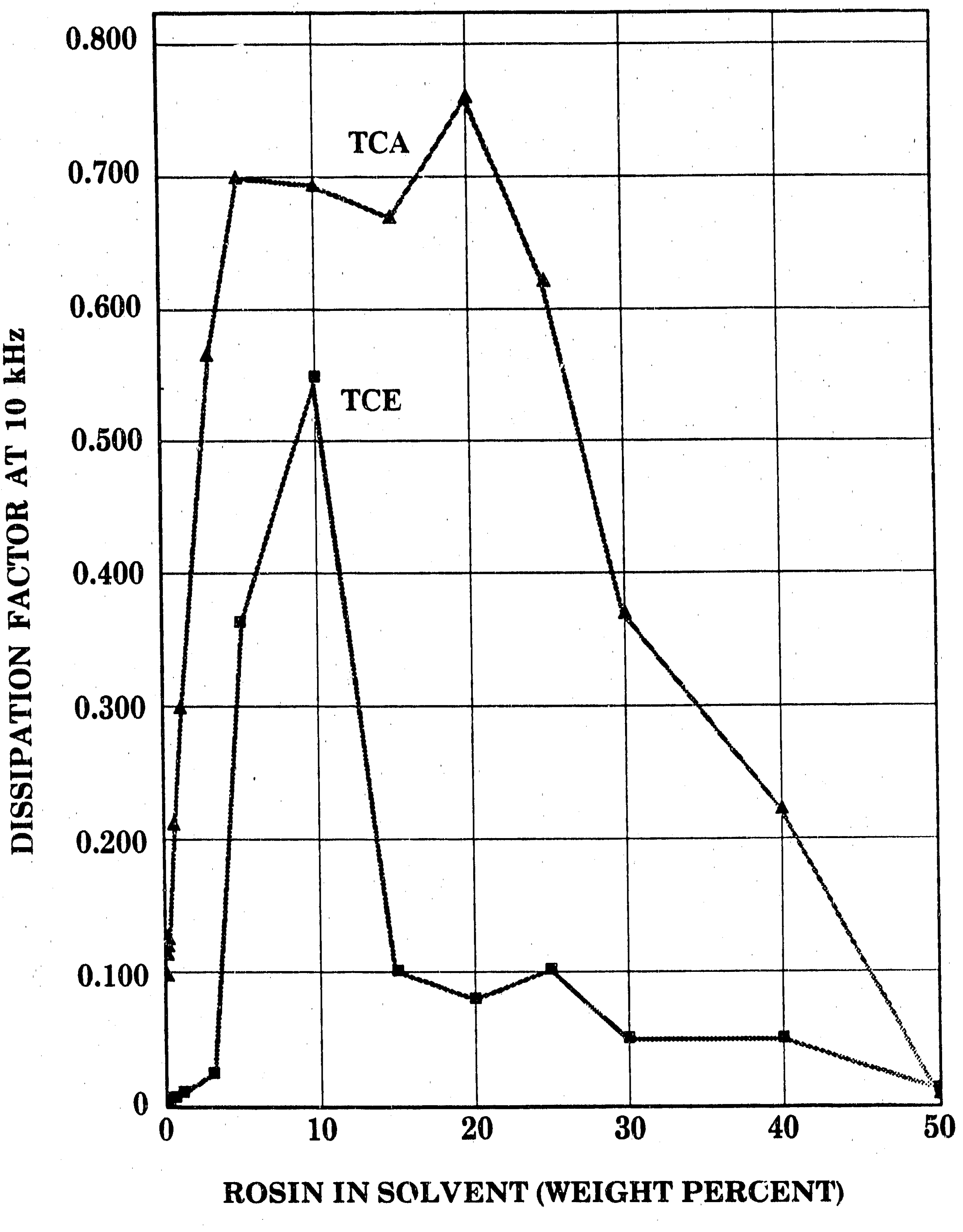

Figure 6. Dissipation Factor Versus Rosin Contamination Levels for Trichloroethylene and 1,1,1-Trichloroethane

Table 8. Theoretical Boiling Temperature for 5- to 30-Percent Oil-Contamination Levels of Trichloroethylene and 1,1,1-Trichloroethane

Solvent	Contamination (percent by volume)	Calculated Boiling Point (°C)* Oil A	Oil B	Oil C
Trichloroethylene	5	87.4	87.4	87.3
	10	87.9	88.0	87.8
	15	88.5	88.6	88.3
	20	89.1	89.3	88.9
	30	90.6	90.9	90.2
1,1,1-Trichloroethane	5	75.3	75.4	75.3
	10	75.9	76.0	75.8
	15	76.6	76.7	76.4
	20	77.3	77.4	77.1
	30	79.0	79.2	78.6

*Contaminants:

Oil A is a mineral-lard oil.
Oil B is a sulfochlorinated-lard oil.
Oil C is a sulfurized mineral oil.

conclusions are invalid. In addition, the theoretical analysis did not include the effects of the solvent stabilizers on the boiling point temperature.

Boiling Point, Stabilizers in Solution

Solvent stabilizers are expected to affect the boiling point of a solvent. During distillation, the concentration of stabilizer components in the solvent would change unless the mixture is a true azeotrope. Boiling point temperature measurements were made of the solution according to ASTM D 1078. ASTM D 1078 requires that the measuring thermometer be in the vapor zone above the boiling solution and not in the solution. The data can be found in Appendix B and are graphed in Figures 9 and 10. The approximately 1°C difference in temperature measurements between the 100-mL and 250-mL solutions is attributed to the change in height of the thermometer above the boiling solvent.

Boiling Point, Rosin Contamination

The electronic assembly manufacturing departments at KCD would have solder flux, not oil, as the major contaminant in a

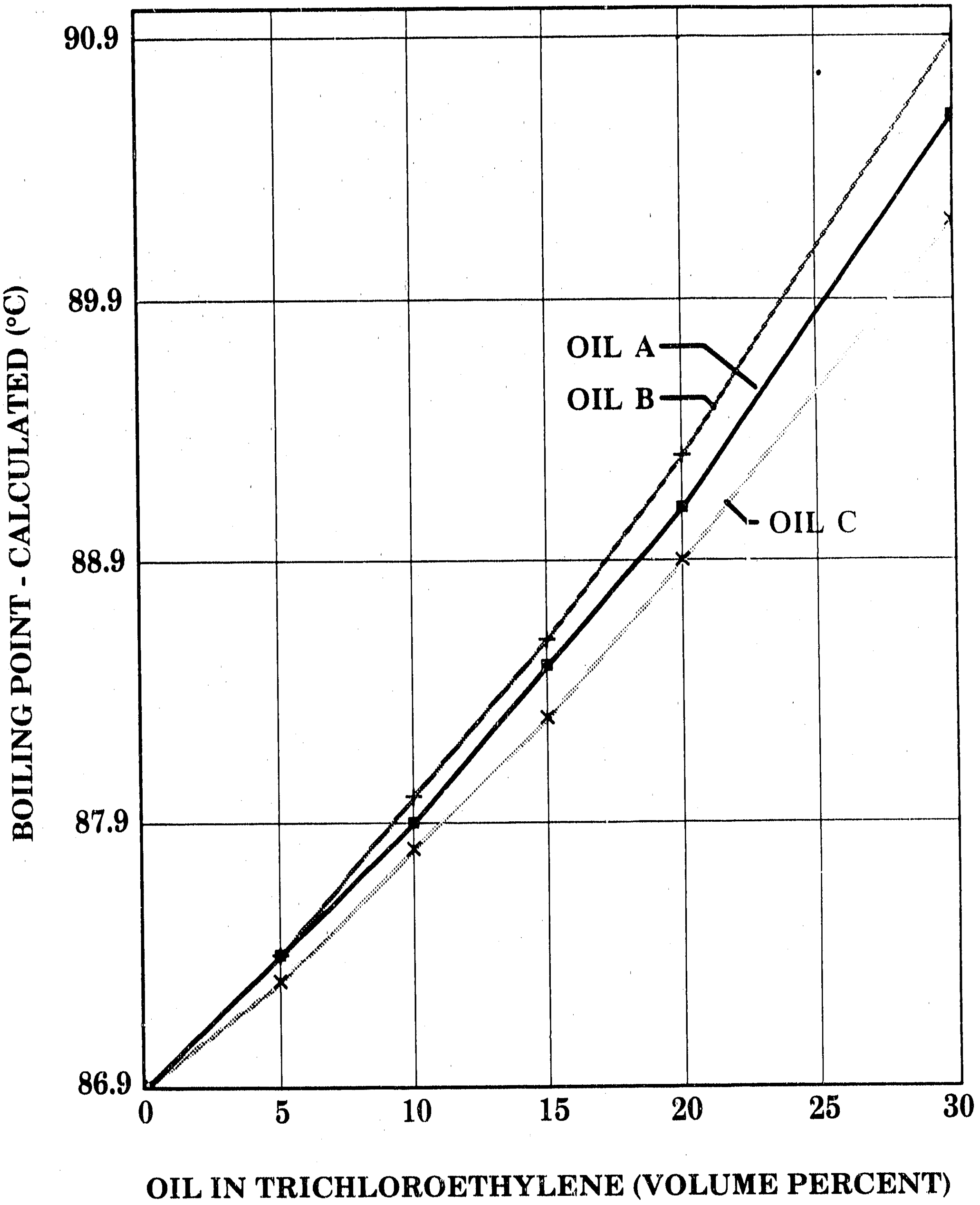

Figure 7. Theoretical Boiling Point Versus Oil Contamination Levels for Trichloroethylene

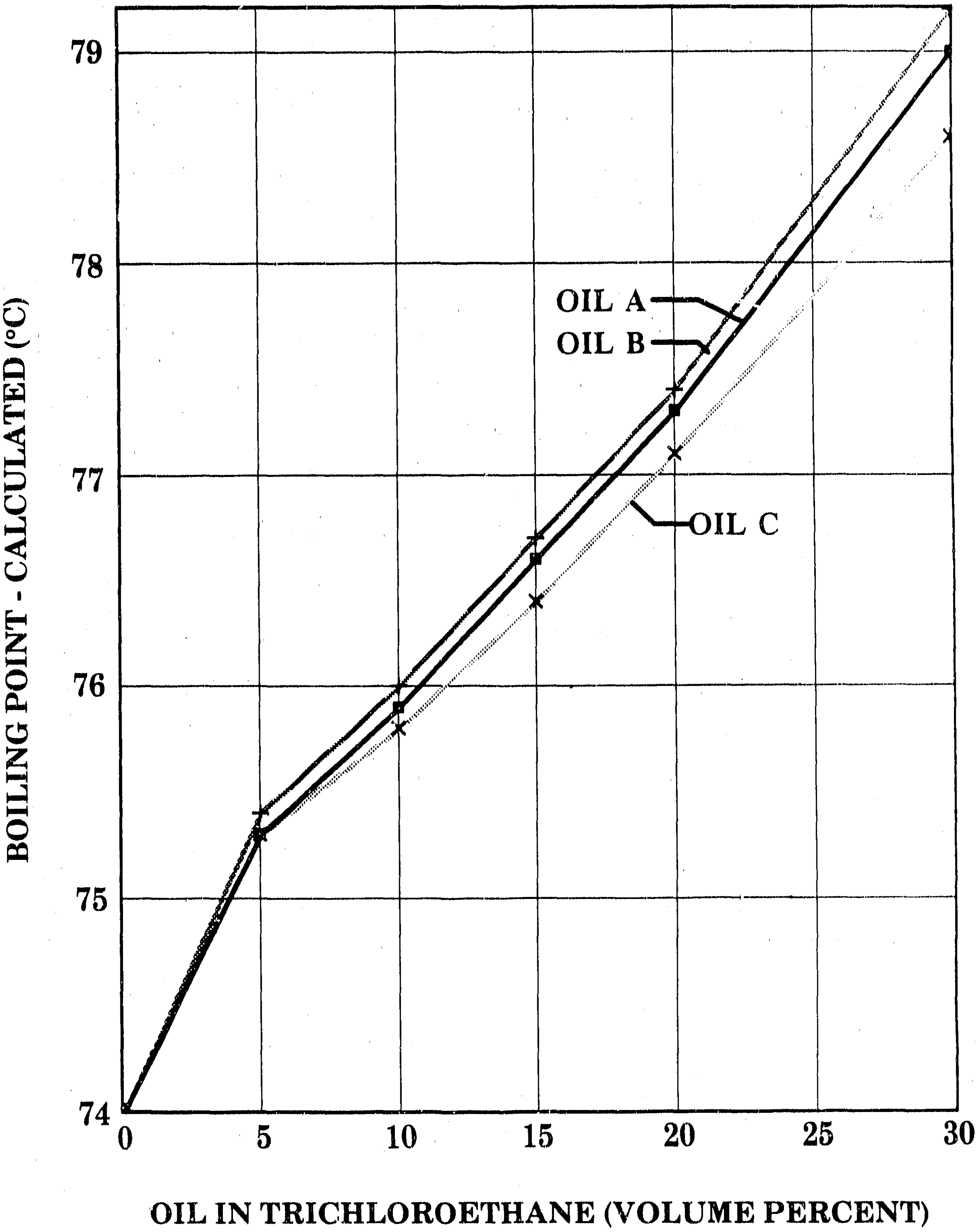

Figure 8. Theoretical Boiling Point Versus Oil Contamination Levels for 1,1,1-Trichloroethane

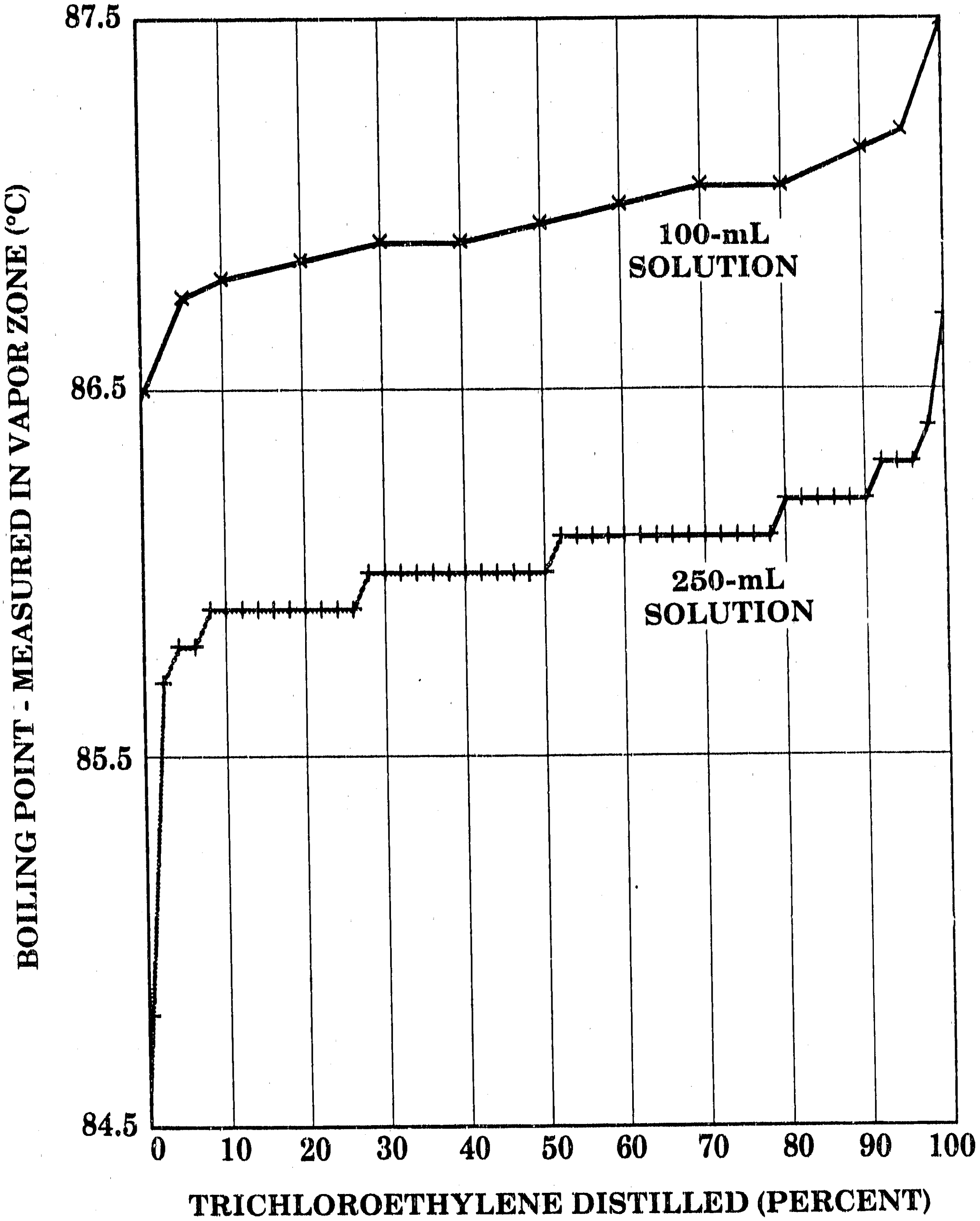

Figure 9. Boiling Point Change During Distillation of As-Received Trichloroethylene

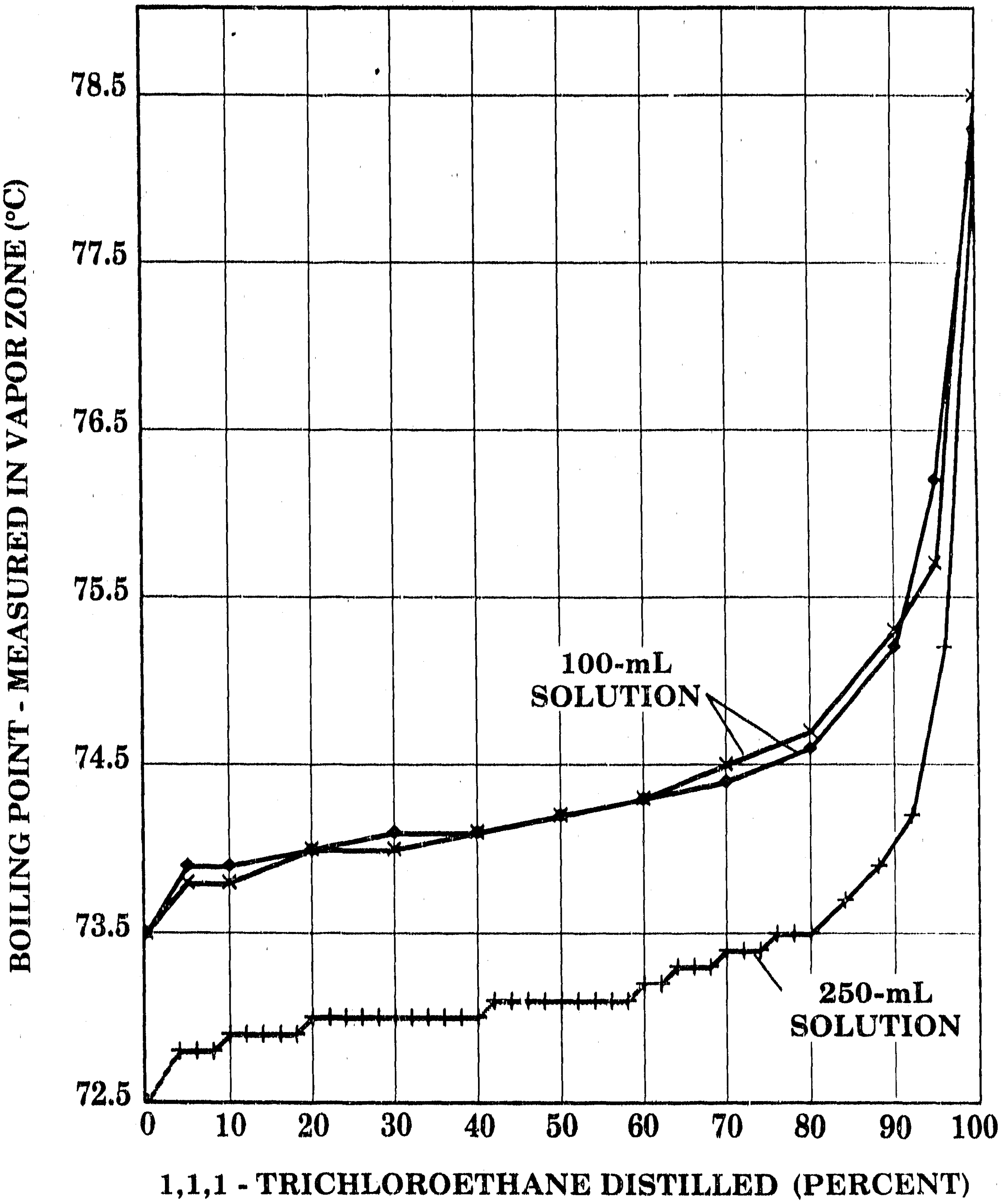

Figure 10. Boiling Point Change During Distillation of As-Received 1,1,1-Trichloroethane

distillation sump. This would be typical for most electronic cleaning applications at KCD. Flux is mainly rosin (80 to 90% abeitic acid, with 10 to 15% pimaric acid) diluted with alcohol.[13] The boiling points of these acids are 250 and 282°C, respectively, which is much higher than either solvent; consequently,[8] the rosin may be considered as a nonvolatile solute.

Two soldering fluxes were the sources of rosin for the boiling point contamination studies. The flux was placed in an oven at 80 to 90°C for 16 to 48 hours to drive off isopropyl alcohol, which was the solvent carrier. Solder flux A is 36-percent rosin by weight, and solder flux B is purchased at KCD under a specification requiring flux with 50-percent rosin by weight.

Solder fluxes also contain activators to aid in the removal of oxides from metal surfaces. The type of activator, amount of activator, and equivalent chlorine ion content of flux activators for many fluxes can be found in a report on solder fluxes and perhaps in flux material safety data sheets. For flux A, the activator is triethanolamine hydrochloride, and for flux B, the activator is dimethylamine hydrochloride. The activators produce an equivalent chlorine content in these fluxes at 0.03 and 0.16 percent by weight of the rosin for fluxes A and B, respectively.

In preparation of the contaminated solutions, rosin was observed to dissolve easily in TCE and TCA at rosin weight levels of 20 percent and less. The rosin dissolved more quickly in TCE than TCA at all contamination levels. At higher levels, the solutions had to set overnight, and at the 40- and 50-percent levels, the solution had to be heated to get all the rosin to dissolve. Once the rosin dissolved, the rosin did not readily come out of solution at 40-percent and lower concentration levels. At the 50-percent level, there was a small amount of thick syrup-like residue on the glass walls after cooling to room temperature.

Initial efforts to measure the boiling point of rosin-contaminated solutions followed the ASTM D 1078 procedure, which measures the temperature in the solvent vapors above the boiling solution. This method gave inconsistent results. The ASTM D 1078 procedure is designed for measuring the boiling points of pure chemicals and has severe limitations if applied to contaminated solutions.

The vapor pressure of a solution with nonvolatile and nonideal solute as the contamination varies according to how many solvent molecules per unit volume are in solution. This is because the boiling temperature and vapor pressure of the solvent in a solution is altered by the molar fraction of the solute in solution. The greater the quantity of nonvolatile solute, the lower the molar fraction and the lower the vapor pressure at a given temperature. To reach a boiling point, the temperature of the solution must increase to the point where the vapor pressure of the diluted solution equals the atmospheric pressure. To measure the boiling point of contaminated solutions, the temperature probe must be in the solution, not above it as is the common laboratory practice for pure chemicals.

Rosin is a nonvolatile contamination that is a nonideal solute in TCE and TCA. A modified ASTM method (Figure 11) was used to measure the boiling points of the contaminated solutions. As the solvent is distilled, the contamination level of the solution increases. The contamination level at any point can be calculated from the known amount of rosin in solution, the initial amount of solvent in the flask, and the amount of solvent distilled. The solution volume was 500 mL initially. The temperature was measured with a thermocouple in the solution as the solvent was being distilled. The thermocouple was 120 mm lower than the mercury thermometer and at about the 50 mL of solution level. The temperature measurements are given in Appendix B and are graphed in Figures 12 and 13. Only the initial concentration and twice initial concentration temperature measurements are graphed in Figures 12 and 13.

In all tests, the boiling point of as-received material started lower than the boiling point of pure solvent. This was caused by the presence of stabilizers in the solution, which also did not behave as true azeotropes. Some stabilizers boiled out of solution quickly while others remained. This is evident from the gas chromatograms (see Figures 1 and 2 and Table 5). These stabilizers were the main influence altering the solvent boiling point if the rosin-contamination level was at less than 30-percent by weight. Above 35-percent by weight rosin contamination, the boiling point was significantly affected by the rosin in solution. Based on these data, the temperature of the boiling solution could be used to determine the rosin contamination level in the 35- to 50-percent by weight contamination range; corrections for atmospheric pressure changes would need to be made to any temperature measurement.

Boiling Point, Atmospheric Effects

The boiling point of any solution is directly related to the vapor pressure of the solution and the atmospheric pressure. As the atmospheric pressure changes, the boiling point will also change. The change in boiling point, dT, caused by a change in atmospheric pressure from standard pressure (760 mm Hg), is given by the equation

$$dT = L(273.3+T)(760-P) \ , \qquad (9)$$

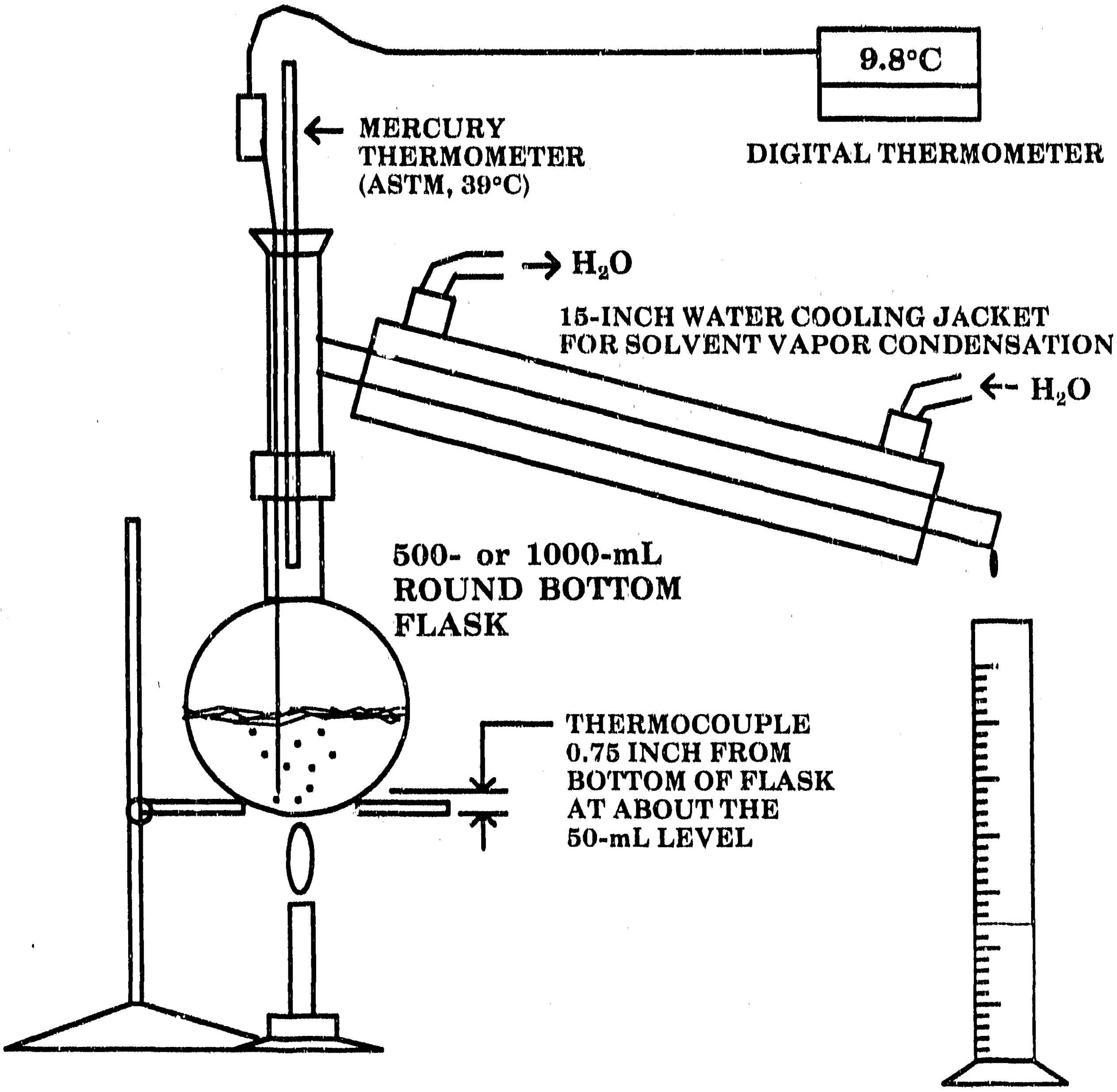

Figure 11. Laboratory Apparatus for Boiling Point Measurements of Contaminated Solutions

where L is the gas constant for the solvent (about 0.00012), T is the observed boiling point in degrees Celsius, and P is the atmospheric pressure in mm Hg at which T was measured.' The change in boiling point with pressure is sometimes more simply given as

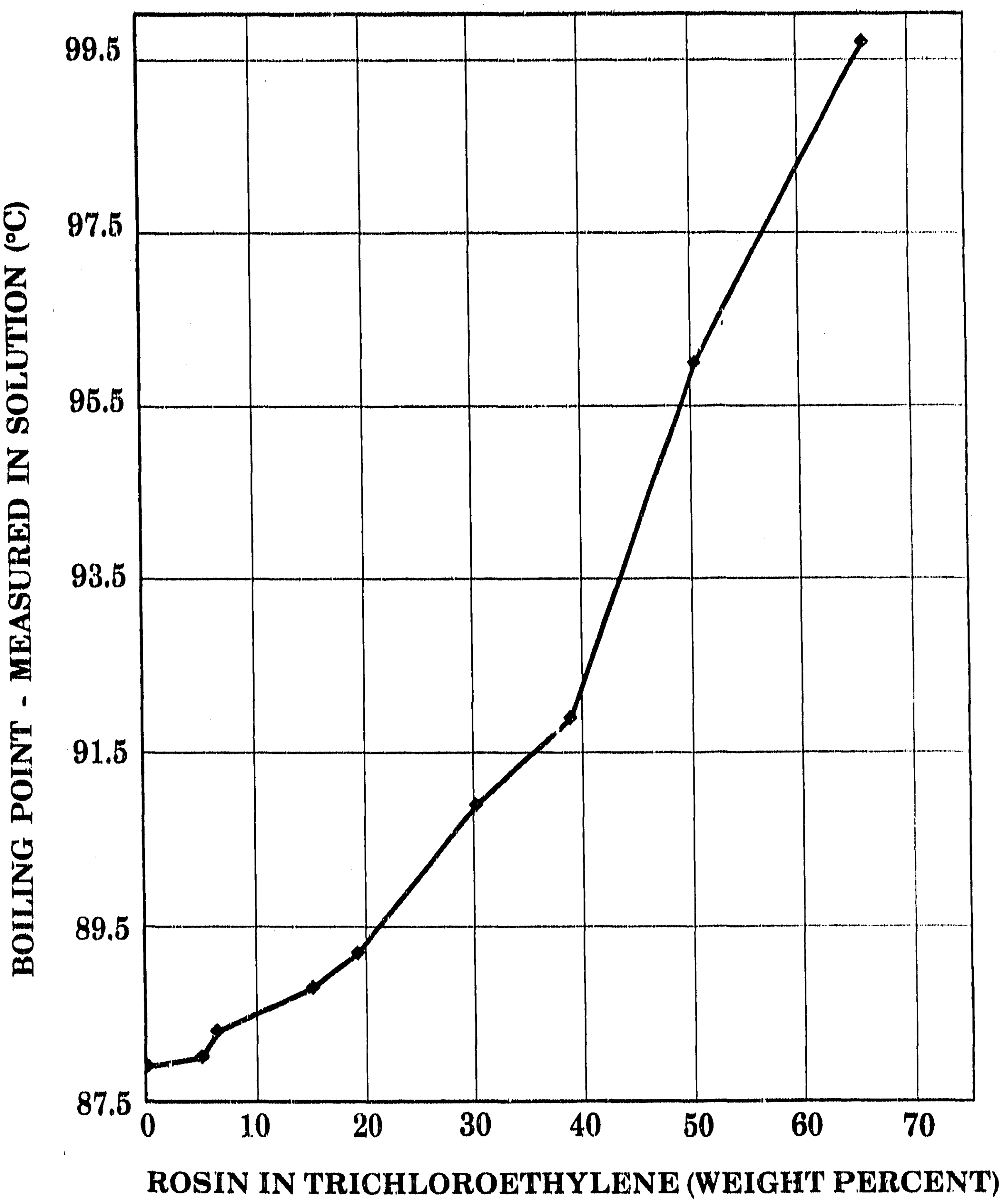

Figure 12. Boiling Point Versus Rosin Contamination Levels for Trichloroethylene

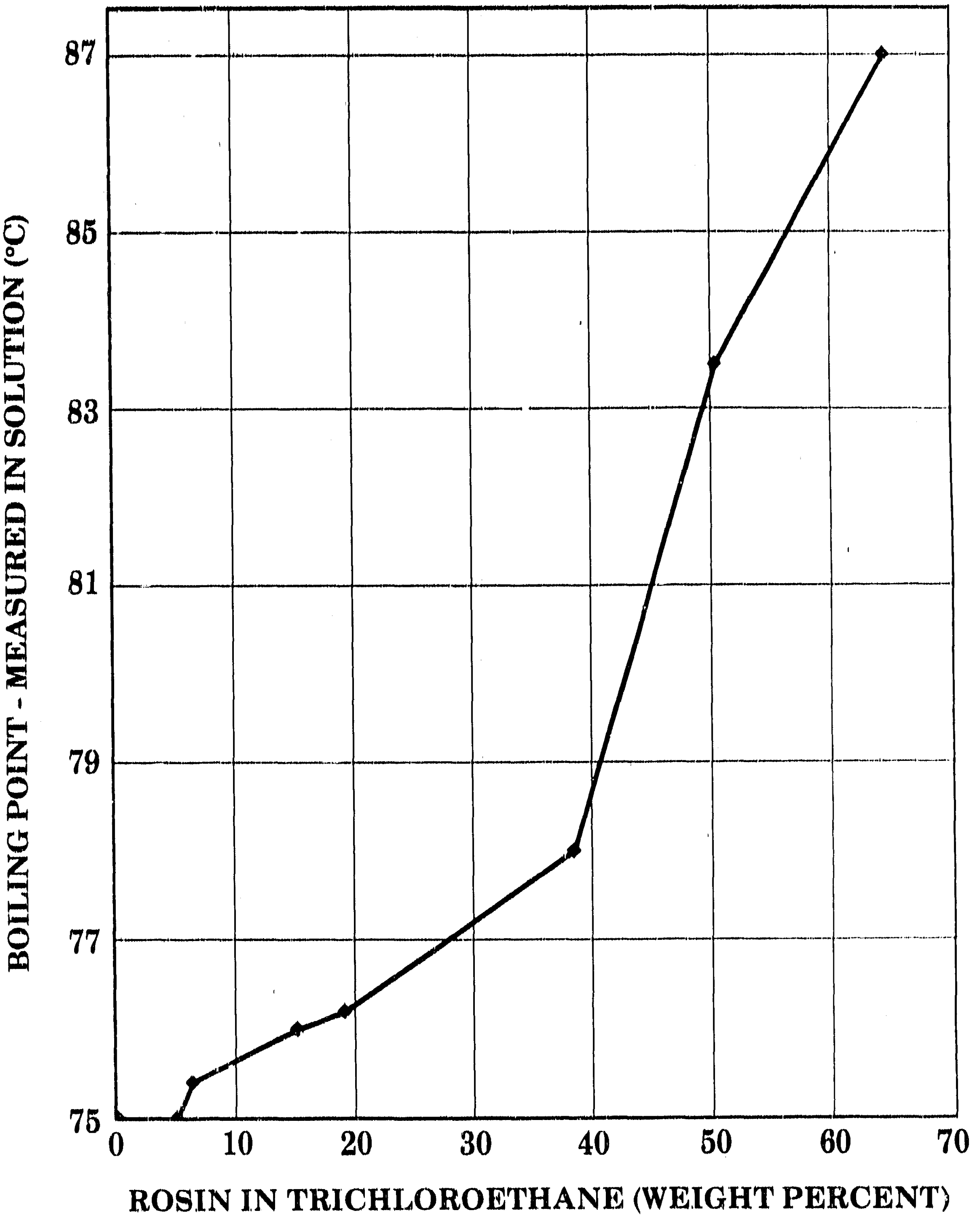

Figure 13. Boiling Point Versus Rosin Contamination Levels for 1,1,1-Trichloroethane

$$\frac{dT}{dP} = Z\,(760-P)\ , \qquad (10)$$

where Z is 0.044 for TCE and 0.043 for TCA.' Based on these equations, one would expect daily pressure variations to cause a ±0.3 to 0.6°C variation in the boiling point. The boiling point data in the Appendices have been corrected for atmospheric pressure change and normalized to 760 mm Hg.

Nonvolatile Residue

Laboratory experiments measured the amount of nonvolatile residue carried over in the distillation of various rosin contamination levels of TCE and TCA. The laboratory apparatus was arranged as illustrated in Figure 11. A 500-mL measure of contaminated solution was placed in the boiling flask, and another 50-mL measure of as-received solvent was added to the flask. The solution was distilled at a slow rate (as recommended in ASTM D 1078) until 100 mL of solvent were collected. An additional 100 mL of as-received solvent were added and the process was repeated. Maintaining the total amount of solvent in the flask at 500 ±50 mL kept the percent by weight of contamination within about 10 percent of the original value. A total of 200 mL, minimum, of distilled solvent was collected for nonvolatile residue carryover measurements.

The 200-mL or greater solutions were evaporated and weighed. The procedure followed was generally in conformance with ASTM D 2109-85, "Nonvolatile Matter in Halogenated Organic Solvents and Their Mixtures," except for the quantity of solution used. The amounts carried over in the laboratory distillation process were very small and always within technical grade specifications (see Table 9 and Figure 14). The as-received TCE and TCA had nonvolatile contents of 0.0138 and 0.00178 percent by weight, respectively; these values are above the specification limits as given in Table 2.

Contaminated solvents were also collected from sumps of vapor degreasers and waste collection cans from spray booths at KCD. These solvents were contaminated from 0.3 to 14 percent, by weight, with various unknown oils, rosins, and other substances. A solution volume of 500 mL was placed in the boiling flask, and 250 mL of solvent were distilled for nonvolatile residue measurements. Distilling 250 mL of solution, without adding solution, increased the final contamination level to approximately twice the initial value. The results of the nonvolatile weight measurements are given in Table 10 and illustrated in Figure 15. The carryover of nonvolatile residue in each case was less than 0.001 percent by weight. The distillation process adequately separates the solvent from the contamination.

Table 9. Nonvolatile Carryover During Distillation of Contaminated Trichloroethylene and 1,1,1-Trichloroethane

Rosin Contamination Level in Distilled Solution (percent by weight)	TCE		TCA		
	Nonvolatile Residue (percent by weight)	Evaporated Solution Size (grams)	Nonvolatile Residue (percent by weight)	Evaporated Solution Size (grams)	Rosin Source
As-Received	0.0138	338.73	0.00177	242.08	none
As-Received Distilled	0.00004	287.66	0.00001	127.60	none
5	0.00004	286.75	0.00008	259.22	A
15	0.00014	286.50	0.00012	260.96	A
30	0.00052	286.73	0.00042	259.94	A
50	0.00052	286.20	0.00023	257.95	A

Vapor Pressure

The quantity of solvent in the air is limited by the maximum amount of solvent that can remain in a vapor form at the air temperature. The maximum amount of solvent vapor in air is determined by the ideal gas law and the vapor pressure of the gas at that temperature. The ideal gas law applied to partial pressures is

$$P = \frac{nRT(k)}{V}, \tag{11}$$

where P is partial pressure caused by the gas, n is the number of moles of the gas sample, R is the universal gas constant, T(k) is the temperature in kelvins, and V is volume. The solvent in weight per volume, D, is given by

$$\begin{aligned} D &= \frac{m\ n}{V} \\ &= \frac{m\ P(T)}{[R\ T]}, \end{aligned} \tag{12}$$

where m is the mole weight of the solvent and P(T) is the vapor pressure as a function of the temperature. P(T) has been experimentally determined and can be given in graph form or by Antoine's equation.

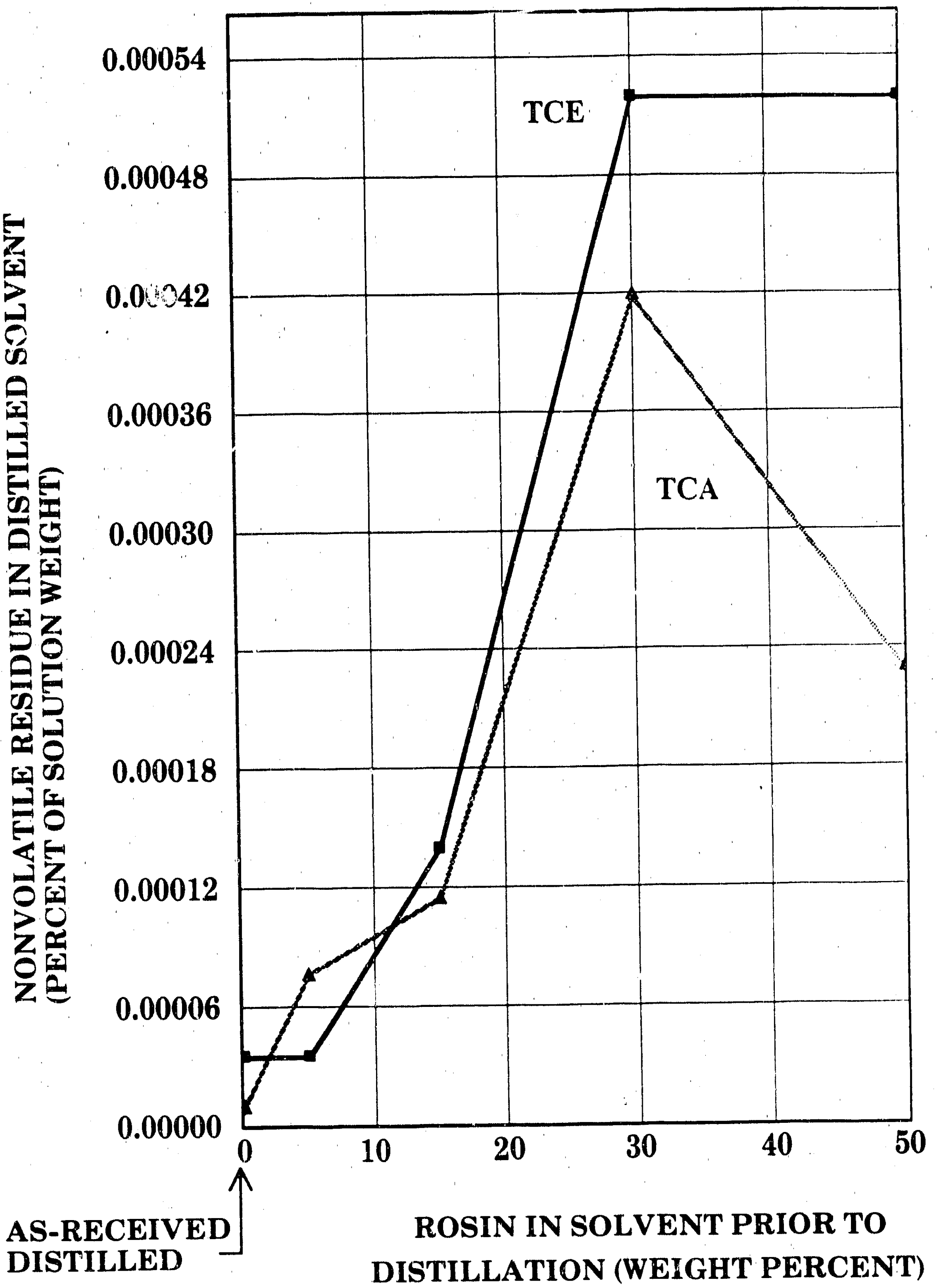

Figure 14. Nonvolatile Residue Carried Over in the Distillation Process of Technical Grade Trichloroethylene and 1,1,1-Trichloroethane

Table 10. Nonvolatile Carryover During Distillation of Contaminated Solutions From Various Manufacturing Departments Compared With Vendor Chemicals

Source and Date of Contaminated Solution	Solvent	Nonvolatile Residue (percent by weight)	Evaporated Solution Size (grams)	Predistilled Contamination Source Level (percent by weight)		Equipment Description
				Initial	Final	
Sump, Distilled	Prelete (TCA)	0.00045	112.18	1.0	2.1	Vapor Degreaser
Can, Distilled 10-27-88	TCE	0.00028	359.47	0.32	0.63	Spray Booth
Can, Distilled 10-31-88	TCE	0.00012	341.43	0.41	0.81	Spray Booth Control
Sump, Distilled 11-5-88	TCA	<0.00001	326.74	0.15	0.30	Vapor Degreaser
Can, Distilled 10-27-88	TCE	<0.00001	323.40	3.6	7.2	Spray Booth
Sump, Distilled	TCE	<0.00001	338.35	0.09	0.19	Vapor Degreaser
Wand 10-27-88	TCE	0.00096	349.26	---	---	
Sump, Distilled	TCE	0.00014	352.82	0.5	1.0	Vapor Degreaser
Wand 10-31-88	TCE	0.00018	374.75	0.5	1.0	Vapor Degreaser
As-Received Solvents						
Technical Grade TCE, 5-gallon can						
As-Received		0.00138	338.73			
As-Received Distilled		0.00004	287.66			
Technical Grade TCA, 1-gallon glass jar						
As-Received		0.00178	242.08			
As-Received Distilled		<0.00001	127.60			
Prelete, 5-gallon can						
As-Received		0.00128	202.40			
As-Received Distilled		0.00129	124.38			

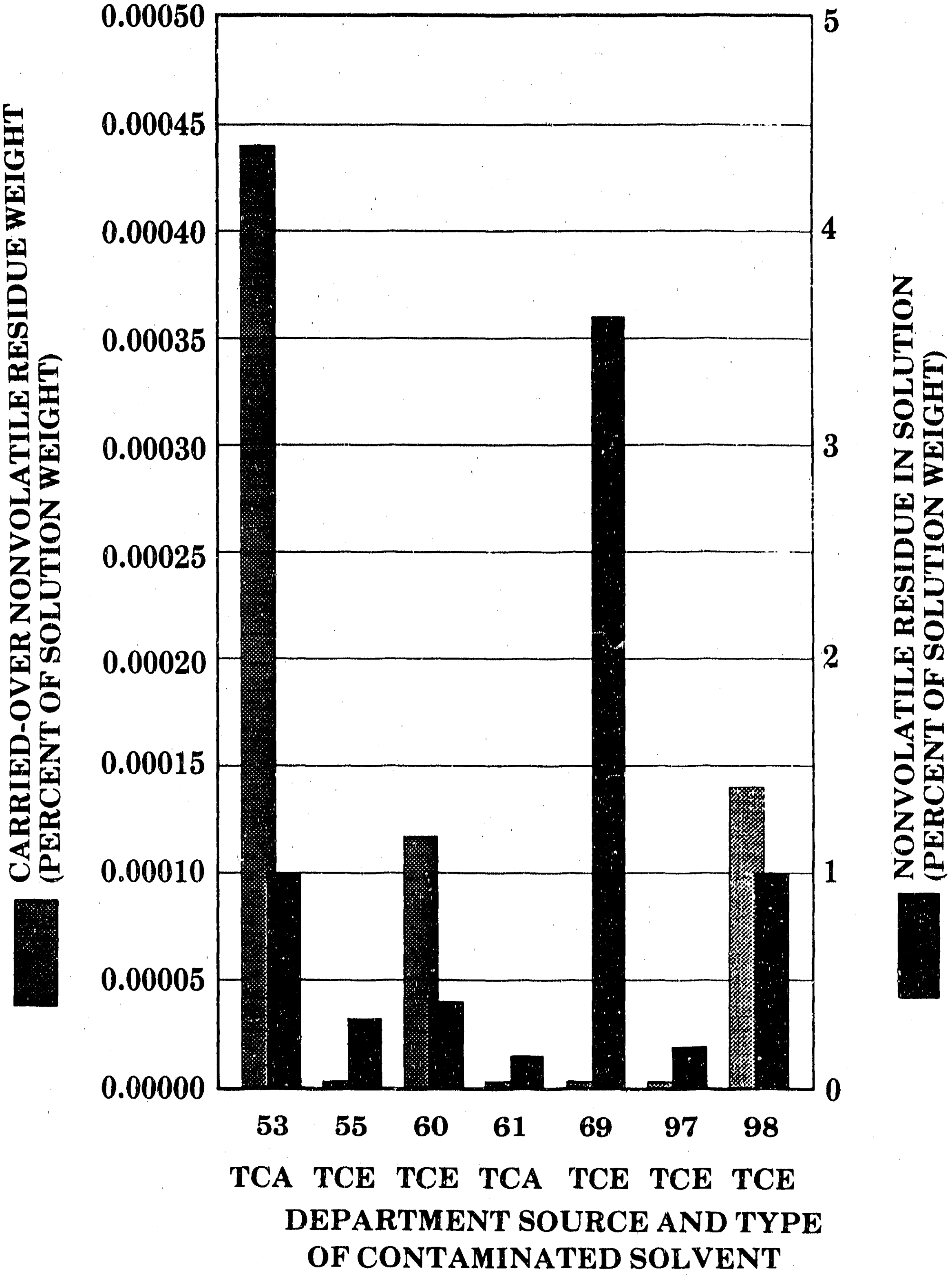

Figure 15. Nonvolatile Residue Carried Over in the Distillation Process of Contaminated Solvent From Various Departments

Antoine's equation is

$$\mathrm{Log10}[P(T)] = A - \frac{B}{[C+T(c)]} , \tag{13}$$

where A, B, and C are the Antoine constants for the solvent, T(c) is the temperature in degrees Celsius, and P(T) is pressure in millimeters of mercury. The Antoine constants for TCE and TCA are given in Table 11. To avoid the confusion of temperature being in both kelvins and Celsius, the substitution of $T(c) = T(k) - 273.3$ will be used. So we have

$$\mathrm{Log10}[P(T)] = A - \frac{B}{[C+T(k)-273.3]} . \tag{14}$$

The amount of solvent per volume as a function of temperature is then

$$D = \frac{m\ 10^{\left[A - \frac{B}{C + T(k) - 273.3}\right]}}{R\ T(k)} , \tag{15}$$

where m(TCE) = 0.29128 lb/mol, m(TCA) = 0.29348 lb/mol, R = 2.2024 ft^3·mmHg/mol·K, and D is in $\mathrm{lb/ft}^3$. The results of calculations showing how the solvent vapor density varies with the temperature are given in Table 12.

Fire Safety

The solvents TCE and TCA have no flash point or fire point as determined by standard tests. However, these solvents do have flammable limits in air and have a flammable range when high concentrations are mixed in air (see Table 1) and exposed to a high-energy source. At 100°C the flammable limits in air of TCE increase to the 52-percent concentration level.[14] For this reason, only electrical equipment approved for use in hazardous locations is recommended for use in closed tanks, in areas where high vapor concentrations may occur because of a spill or other mishap, or in other locations where high concentrations of solvent vapor may accumulate.[15]

Lab experiments performed show that TCE and TCA will continue to burn as long as they are exposed to an open flame. TCE and TCA give off chlorine and other toxic gases when burning. The presence of rosin did not appear to increase the fire hazard of these solvents. A simple experiment exposing 30- and 50-percent solutions to open flame produced reactions similar to those of

Table 11. Antoine Constants for Trichloroethylene and 1,1,1-Trichloroethane

Constant	Trichloroethylene*	1,1,1-Trichloroethane**
A	7.11886	6.92654
B	1367.05	1215.94
C	235.809	226.503

*Applies from -86 to 86°C.
**Applies from -30 to 74°C.

uncontaminated solutions; one would expect this from the distillation experiments that show very little rosin goes into vapor. At very high (greater than 90-percent) rosin concentrations, the rosin can be ignited and will continue to burn with the small amount of solvent present.

Health Safety

Health and safety data of fluorinated, chlorinated, and a few other solvents are given in Table 13.[14,15] Other general properties of TCE and TCA were given in Table 1.[14] It is apparent from Table 13 that if one can smell the solvent, there is too much in the air. OSHA has proposed a reduction in the threshold limit value-time weighted average (TLV-TWA) values for some of these solvents, and further reductions could make using them more difficult and less desirable.[16]

A white paper issued by the Halogenated Solvents Industry Alliance (HSIA) brought out recent findings that laboratory rats metabolize solvents in a different manner than humans, and, therefore, the conclusions of the toxicology studies implicating TCE and TCA are invalid.[17,18] Independent of these findings, the solvents' known toxicity and the hazardous waste produced from cleaning with them requires that improvements be made in their use. Restrictions on these solvents by government agencies are not likely to be removed. For these reasons, a vapor-containing cleaning system is needed that will minimize atmospheric emissions and exposure of personnel to solvent.

ACCOMPLISHMENTS

The physical properties of contaminated trichloroethylene and 1,1,1-trichloroethane were characterized. The specific gravity, volume change, dielectric constant, dissipation factor, boiling point, and nonvolatile residue carryover during distillation were

Table 12. Solvent Density and PPM Level for Various Temperatures of Air

Temperature		Solvent Vapor Density (lb/ft^3)*		Solvent Level**	
°C	°F	TCE	TCA	TCE (%)	TCA (%)
37.7	100	0.056	0.090	22	39
32.2	90	0.045	0.073	18	32
26.7	80	0.036	0.059	14	26
10.0	50	0.017	0.029	6.7	13
6.1	43	0.014	0.024	5.5	10
0	32	0.010	0.018	3.9	7.8
-10	14	0.0058	0.010	2.3	4.3
-20	-4	0.0032	0.0057	1.25	2.5
-30	-22	0.0016	0.0030	0.62	1.3
-40	-40	0.00078	0.0021#	0.31	0.91 (9100 ppm)#
-50	-58	0.00034	--	1300 ppm	--
-60	-76	0.00014	--	550 ppm	--
-70	-94	0.000049	--	193 ppm	--
-80	-112	0.000015	--	59 ppm	--
-86	-123	0.000007	--	28 ppm	--

*Calculated using Antoine's constants from Table 11.

**After removal of condensed solvent and returning to standard temperature and pressure.

#Values at -35°C, just above the freezing point of TCA, which is -37°C.

measured for various contamination levels of rosin in trichloroethylene and 1,1,1-trichloroethane. Solvent stabilizers and the vapor pressure of solvents were also examined. The effects of oil contamination on the boiling point were also examined.

The data obtained in this effort supported the design of the new vapor-containing cleaning system that will meet the demands of new environmental and health restrictions and maintain the high level of cleaning needed on electronic assemblies. These data eliminated the unknowns in the area of contamination carryover and physical property changes that could be monitored to indicate contamination level.

FUTURE WORK

Two practical applications to environmental waste are suggested below. No future work in characterization of contaminated solvents is planned as part of this project at the present time.

Table 13. Health and Safety Data for Solvents

Solvent Odor*

Solvent	Odor Threshold	Slight, Not Unpleasant	Strong, Unpleasant
Trichloroethylene	20-50	100-400	>1000
1,1,1-Trichloroethane (Methyl Chloroform)	100	350-500	>900
Perchloroethylene	5-70	100-200	>280
Methylene Chloride	150-600	250-1000	>1000

OSHA Exposure Limits*

Solvent	8-Hour** Time-Weighted Average	Acceptable Ceiling Concentration	Maximum Acceptable Peak
Trichloroethylene	50 (25)***	200	300 for 5 min in any 2 h
1,1,1-Trichloroethane	350	---	450 for 15 min
Perchloroethylene	25 (50)***	200	300 for 5 min in any 3 h
Methylene Chloride	500	1000	2000 for 5 min in any 2 h
Methylethylketone (MEK)	200	---	---
Isopropyl Alcohol	400	---	---
Genesolv DFX	480	---	---
Genesolv DM	600	---	---
Acetone	750	---	---

ACGIH TLVs ****

	TLV*	
Solvent	Time-Weighted Average**	Short-Term (15 min) Exposure Limit#
Trichloroethylene	50	200
1,1,1-Trichloroethane (Methyl Chloroform)	350	450
Perchloroethylene	50	200
Methylene Chloride	50	--
Methylethylketone (MEK)	200	300
Isopropyl Alcohol	400	500
Genesolv DFX	480	
Genesolv DM	140	
Acetone	750	1000

Table 13 Continued. Health and Safety Data for Solvents

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*All values in parts per million by volume in air.

**Exposure for 8 hours per day for a 40-hour work week.

***Proposed limits by OSHA on June 7, 1988, Federal Register, Volume 53, Number 109, pp 20959-21393.

****ACGIH stands for the American Conference of Governmental Industrial Hygienists. TLV stands for threshold limit value.

#Four times per day exposure limit with one hour, minimum, between exposures.

Laboratory analysis of solvents contaminated with oils is an important effort that should be completed.

Solvent recovery must start with accurately identifying the solvent. If cross-contamination of solvents occurs in a recycling batch, all the solvent could be lost and the still damaged. Measurements of the dielectric constant and dissipation factor of TCE and TCA contaminated with rosin indicate that these properties may be used as a fast and inexpensive indicator of which solvent is in a waste container (see Figures 5 and 6). To a limited degree, the amount of contamination in the solvent may be estimated by measuring the dielectric constant and dissipation factor. Efforts should be dedicated toward using this method for solvent recovery process control.

High weight percent contaminated solvent waste is not recoverable at and above the 90-percent level because of the need to drain the waste from the still. Recycling high weight percent contaminated solvent waste may require an alternate recycling method from what is typically used. Recycling solvent waste is a distillation process that normally does not take the sump to dryness; the residue, still a liquid, is drained and disposed of as hazardous waste.

Over 90-percent of the solvent could be recovered and the residue could be disposed of as nonhazardous waste (assuming the contamination is rosin) if a temperature-controlled heat source heated the residue until it was completely dry. The waste could be boiled dry in a thin disposable liner, removed, and disposed of. This recovery approach, if 95-percent efficient, could reduce the solvent waste to 0.05 times the weight of the contaminant.

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Appendix A

ELECTRICAL PROPERTY MEASUREMENTS OF AS-RECEIVED AND ROSIN-CONTAMINATED TRICHLOROETHYLENE AND 1,1,1-TRICHLOROETHANE

MATERIALS AND PROCEDURE

This work was accomplished during September and October 1988.

SOLVENTS AND ROSIN

The trichloroethylene (TCE) used in all the laboratory experiments was from one 5-gallon can of technical grade solvent. The trichloroethane (TCA) used in all the laboratory experiments was from one lot of technical grade solvent supplied in 1-gallon glass containers.

The rosin used in the experiments was taken from two solder fluxes which meet MIL-F-14256 specification as types R and RMA. The rosin solvent carrier (alcohol) in the flux was removed by elevating the flux temperature to 80 to 90°C for 16 to 48 hours (90°C is well below the boiling point of the flux and rosin).

DIELECTRIC CONSTANT

The dielectric constant of contaminated solutions was measured using a guarded-ring parallel-plate capacitor and making capacitance measurements. Capacitance measurements were made using a capacitance test cell and an automatic capacitance bridge. The test cell had a micrometer adjustment with 0.001-inch markings for the parallel plate spacing and had a guard ring to eliminate fringe effects. The parallel plate was 2.0 inches in diameter with a 0.2-inch wide circumferential guard ring. The capacitance bridge was accurate to ±1 percent for the capacitance values measured and all measurements were made at room temperature. Capacitance measurements were made at 10 kHz, 100 kHz, and 1 MHz, with a few measurements at 1 kHz. Capacitance measurements were made at spacings of 0.050, 0.100, 0.150, 0.200, and 0.250 inch between the parallel plates. The dielectric constant was calculated by using the equation

$$K = \frac{C_\epsilon}{Ca},$$

where C_ϵ is capacitance of the test cell filled with the solution and Ca is the capacitance of the test cell filled only with air.

DISSIPATION FACTOR

Dissipation factor is a measure of the power loss when a material is used as a dielectric in a capacitor. The dissipation factor is also called the loss tangent and is defined as the ratio of the real impedance divided by the complex impedance of the dielectric. The dissipation factor was measured using the same equipment and procedure as was used for measuring the dielectric constant (Tables A-1, A-2, and A-3). The dissipation factor measurements at 1 MHz were not very consistent. Values in parenthesis (Table A-1) were from measurements taken at a later date. Stable readings occurred in the 10- to 100-kHz frequency range.

Table A-1. Dissipation Factor and Capacitance Measurements of As-Received TCE and TCE Contaminated With Rosin

Gap Spacing (inch)	Capacitance (pF)				Dissipation Factor			
	1 kHz	10 kHz	100 kHz	1 MHz	1 kHz	10 kHz	100 kHz	1 MHz
Solution: Air (Measured 11-5-88)								
0.050	21.56	21.58	21.55	21.72	0	0.0005	0.0001	0.0003
0.100	10.95	10.970	10.966	11.049	0	0	0.0003	0
0.150	7.34	7.351	7.344	7.399	0	0.0001	0.0006	0
0.200	5.50	5.518	5.511	5.551	0	0.0002	0.0008	0
0.250	4.39	4.410	4.403	4.435	0	0.0002	0.0012	0
Solution: As-Received Technical Grade TCE (Measured 10-11-88)								
0.050	74.86	74.86	74.86	76.05	0.0062	0.0008	0.0003	0.0016
0.100	38.02	38.03	38.02	38.44	0.0062	0.0010	0.0010	0.0015
0.150	25.44	25.47	25.45	25.70	0.0061	0.0010	0.0003	0.0016
0.200	19.09	19.12	19.10	19.26	0.0060	0.0012	0.0005	0.0027
0.250	15.25	15.280	15.276	15.434	0.0060	0.0007	0.0018	------
Ave. Dielectric Constant			3.468	3.476				
Solution: As-Received Technical Grade TCE (Measured 11-5-88)								
0.050	75.33	75.36	75.34	76.52	0.0062	0.0007	0.0003	0.0006
0.100	38.27	38.29	38.28	38.70	0.0061	0.0009	0.0002	0.0004
0.150	25.63	25.64	25.63	25.87	0.0061	0.0012	0.0003	0.0003
0.200	19.24	19.25	19.25	19.42	0.0060	0.0012	0.0002	0.0005
0.250	15.37	15.404	15.499	15.555	0.0058	0.0005	0.0002	0.0000
Ave. Dielectric Constant			3.495	3.501				
Solution: As-Received--in storage bottle for 25 days (Measured 11-5-88)								
0.050	75.16	75.18	75.16	76.33	0.0162	0.0018	0.0004	0.0020
0.100	38.17	38.19	38.17	38.57	0.0170	0.0020	0.0002	0.0008
0.150	25.59	25.61	25.59	25.83	0.0175	0.0022	0.0004	0.0009
0.200	19.22	19.25	19.23	19.40	0.0170	0.0023	0.0004	0.0009
0.250	15.35	15.390	15.385	15.537	0.0166	0.0016	0.0004	0.0003
Ave. Dielectric Constant			3.488	3.495				

Table A-1 Continued. Dissipation Factor and Capacitance Measurements of As-Received TCE and TCE Contaminated With Rosin

Gap Spacing (inch)	Capacitance (pF) 1 kHz	10 kHz	100 kHz	1 MHz	Dissipation Factor 1 kHz	10 kHz	100 kHz	1 MHz
Solution: 0.05-Percent Rosin from Flux A (Measured 11-5-88)								
0.050	74.69	74.70	74.69	75.84	0.0161	0.0018	0.0003	0.0006
0.100	37.95	37.98	37.96	38.37	0.0161	0.0018	0.0002	0.0004
0.150	25.46	25.46	25.46	25.68	0.0160	0.0020	0.0004	0.0004
0.200	19.12	19.14	19.12	19.28	0.0160	0.0023	0.0003	0.0003
0.250	15.27	15.306	15.300	15.447	0.0160	0.0016	0.0003	0.0000
Ave. Dielectric Constant			3.468	3.475				
Solution: 0.1-Percent Rosin from Flux A (Measured 11-5-88)								
0.050	74.69	74.73	74.71	75.85	0.0241	0.0026	0.0004	0.0006
0.100	37.99	38.01	37.98	38.40	0.0240	0.0028	0.0003	0.0005
0.150	25.44	24.47	25.45	25.69	0.0241	0.0028	0.0004	0.0004
0.200	19.13	19.14	19.12	19.29	0.0240	0.0030	0.0005	0.0005
0.250	15.28	15.312	15.307	15.454	0.0240	0.0024	0.0004	0.0001
Ave. Dielectric Constant			3.469	3.477				
Solution: 0.5-Percent Rosin from Flux A (Measured 11-5-88)								
0.050	74.82	74.84	74.82	75.97	---	---	---	---
0.100	38.04	38.06	38.04	38.45	0.0657	0.0070	0.0007	0.0005
0.150	25.48	25.50	25.48	25.72	0.0656	0.0070	0.0009	0.0005
0.200	19.13	19.16	19.14	19.31	0.0656	0.0072	0.0008	0.0006
0.250	15.29	15.323	15.319	15.466	0.0656	0.0064	0.0008	0.0000
Ave. Dielectric Constant			3.473	3.481				
Solution: 1.0-Percent Rosin from Flux A (Measured 11-5-88)								
0.050	74.77	74.79	74.77	75.93	0.1061	0.0108	0.0010	0.0006
0.100	38.07	38.08	38.07	38.49	0.1060	0.0109	0.0013	0.0005
0.150	25.48	25.51	25.49	25.73	0.1059	0.0110	0.0011	0.0005
0.200	19.16	19.17	19.14	19.32	0.1056	0.0112	0.0012	0.0006
0.250	15.30	15.333	15.329	15.476	0.1057	0.0105	0.0012	0.0000
Ave. Dielectric Constant			3.474	3.482				

Table A-1 Continued. Dissipation Factor and Capacitance Measurements of As-Received TCE and TCE Contaminated With Rosin

Gap Spacing (inch)	Capacitance (pF)				Dissipation Factor			
	1 kHz	10 kHz	100 kHz	1 MHz	1 kHz	10 kHz	100 kHz	1 MHz
Solution: 3.0-Percent Rosin from Flux A (Measured 11-5-88)								
0.050	75.16	75.17	75.15	76.32	0.2419	0.0244	0.0025	0.0009
0.100	38.17	38.19	38.17	38.58	0.2419	0.0246	0.0024	0.0007
0.150	25.57	25.59	25.57	25.81	0.2419	0.0246	0.0025	0.0007
0.200	19.20	19.22	19.21	19.38	0.2418	0.0250	0.0026	0.0007
0.250	15.35	15.376	15.373	15.525	0.2417	0.0241	0.0026	0.0001
Ave. Dielectric Constant			3.486	3.493				
Solution: 5-Percent Rosin from Flux A (Measured 10-11-88)								
0.050	75.47	75.47	75.45	76.65	0.3628	0.0365	0.0039	0.0020
0.100	38.32	38.33	38.33	38.75	0.3628	0.0367	0.0037	0.0016
0.150	25.65	25.66	25.63	25.89	0.3627	0.0367	0.0039	0.0016
0.200	19.26	19.26	19.25	19.43	0.3629	0.0370	0.0039	0.0017
0.250	15.39	15.41	15.41	15.58	0.3632	0.0363	0.0038	0.0011
Ave. Dielectric Constant			3.500	3.505				
Solution: 10-Percent Rosin from Flux A (Measured 10-11-88)*								
0.050	76.15	76.17	76.15	77.42	0.5488	0.0552	0.0056	0.0026 (14)
0.100	38.66	38.67	38.67	39.08	0.5487	0.0555	0.0059	0.0029 (13)
0.150	25.89	25.90	25.88	26.11	0.5485	0.0555	0.0059	0.0041 (14)
0.200	19.44	19.45	19.42	19.61	0.5486	0.0556	0.0058	0.0019 (13)
0.250	15.52	15.54	15.55	15.72	0.5490	0.0549	0.0058	0.0018 (08)
Ave. Dielectric Constant			3.529	3.614				
Solution: 15-Percent Rosin from Flux A (Measured 10-11-88)*								
0.050	---	76.67	76.65	77.94	---	0.1010	0.0105	0.0049 (23)
0.100	---	38.89	38.85	39.39	---	0.1012	0.0103	0.0038 (22)
0.150	---	26.04	26.02	26.30	---	0.1014	0.0103	0.0030 (20)
0.200	---	19.55	19.53	19.70	---	0.1016	0.0104	0.0033 (18)
0.250	---	15.61	15.62	15.79	---	0.1008	0.0105	0.0019 (22)
Ave. Dielectric Constant			3.548	3.555				

Table A-1 Continued. Dissipation Factor and Capacitance Measurements of As-Received TCE and TCE Contaminated With Rosin

Gap Spacing (inch)	Capacitance (pF)				Dissipation Factor			
	1 kHz	10 kHz	100 kHz	1 MHz	1 kHz	10 kHz	100 kHz	1 MHz
Solution: 20-Percent Rosin from Flux A (Measured 10-11-88)*								
0.050	---	77.52	77.49	78.81	---	0.0801	0.0084	0.0043 (23)
0.100	---	39.36	39.34	39.77	---	0.0802	0.0082	0.0032 (23)
0.150	---	26.36	26.35	26.66	---	0.0804	0.0086	0.0074 (23)
0.200	---	19.80	19.78	19.96	---	0.0804	0.0083	0.0032 (19)
0.250	---	15.82	15.83	15.97	---	0.0797	0.0088	0.0083 (21)
Ave. Dielectric Constant			3.593	3.600				
Solution: 25-Percent Rosin from Flux B (Measured 10-11-88)*								
0.050	---	77.99	77.96	79.29	---	0.1020	0.0105	0.0033 (26)
0.100	---	39.59	39.56	40.03	---	0.1023	0.0106	0.0037 (27)
0.150	---	26.51	26.49	26.73	---	0.1026	0.0108	0.0050 (26)
0.200	---	19.92	19.90	20.08	---	0.1029	0.0105	0.0033 (23)
0.250	---	15.91	15.93	16.04	---	0.1034	0.0107	0.0028 (30)
Ave. Dielectric Constant			3.613	3.618				
Solution: 30-Percent Rosin from Flux B (Measured 10-11-88)*								
0.050	---	78.29	78.27	79.59	---	0.0515	0.0056	0.0045 (31)
0.100	---	39.71	39.69	40.12	---	0.0514	0.0054	0.0039 (31)
0.150	---	26.61	26.58	26.84	---	0.0514	0.0053	0.0024 (31)
0.200	---	19.98	19.97	20.14	---	0.0515	0.0053	0.0022 (30)
0.250	---	15.96	15.97	16.08	---	0.0516	0.0053	0.0027 (30)
Ave. Dielectric Constant			3.623	3.628				
Solution: 30-Percent Rosin from Flux B (Measured 10-11-88)*								
0.050	---	77.27	77.25	77.56	---	0.0481	0.0051	0.0045 (43)
0.100	---	39.23	39.21	39.64	---	0.0486	0.0051	0.0028 (40)
0.150	---	26.28	26.26	26.50	---	0.0489	0.0053	0.0052 (33)
0.200	---	19.74	19.71	19.90	---	0.0490	0.0052	0.0032 (32)
0.250	---	15.78	15.78	15.95	---	0.0485	0.0052	0.0022
Ave. Dielectric Constant			3.581	3.588				

Table A-1 Continued. Dissipation Factor and Capacitance Measurements of As-Received TCE and TCE Contaminated With Rosin

Gap Spacing (inch)	Capacitance (pF)				Dissipation Factor			
	1 kHz	10 kHz	100 kHz	1 MHz	1 kHz	10 kHz	100 kHz	1 MHz
Solution: 40-Percent Rosin from Flux B (Measured 10-11-88)								
0.050	78.33	78.34	78.30	79.51	0.4955	0.0501	0.0057	0.0041
0.100	39.86	39.86	39.80	40.23	0.4956	0.0503	0.0057	0.0044
0.150	26.72	26.72	26.69	26.89	0.4854	0.0505	0.0059	0.0037
0.200	20.09	20.10	20.06	20.20	0.4949	0.0506	0.0059	0.0037
0.250	16.08	16.07	16.04	16.16	0.4945	0.0507	0.0060	0.0045
Ave. Dielectric Constant			3.640	3.640				
Solution: 50-Percent Rosin from Flux A (Measured 10-11-88)								
0.050	78.19	78.19	78.16	79.09	0.1270	0.0132	0.0037	0.0224
0.100	39.68	39.69	39.66	39.92	0.1261	0.0131	0.0034	0.0179
0.150	26.56	26.58	26.56	26.74	0.1254	0.0132	0.0035	0.0175
0.200	19.95	19.97	19.95	20.06	0.1249	0.0133	0.0034	0.0170
0.250	15.94	15.973	15.967	16.03	0.1244	0.0126	0.0038	0.0168
Ave. Dielectric Constant			3.613	3.614				

*Measurements in parentheses were taken at a later date.

Table A-2. Dissipation Factor and Capacitance Measurements of As-Received TCA and TCA Contaminated With Rosin

Gap Spacing (inch)	Capacitance (pF)				Dissipation Factor			
	1 kHz	10 kHz	100 kHz	1 MHz	1 kHz	10 kHz	100 kHz	1 MHz
Solution: Air (Capacitance measured 10-11-88; dissipation factor measured 11-5-88)								
0.050	21.56	21.58	21.55	21.72	0	0.0005	0.0001	0.0003
0.100	10.95	10.970	10.966	11.049	0	0	0.0003	0
0.150	7.34	7.351	7.344	7.399	0	0.0001	0.0006	0
0.200	5.50	5.518	5.511	5.551	0	0.0002	0.0008	0
0.250	4.39	4.410	4.403	4.435	0	0.0002	0.0012	0
Solution: As-Received Technical Grade TCA (Measured 10-11-88)								
0.050	---	155.20	155.17	158.40	---	0.0970	0.0118	0.0197
0.100	---	78.75	78.74	80.25	---	0.1141	0.0117	0.0040
0.150	---	52.74	52.72	53.59	---	0.1176	0.0123	0.0068
0.200	---	39.57	39.54	40.10	---	0.1194	0.0121	0.0028
0.250	---	31.60	31.58	32.02	---	0.1202	0.0121	0.0022
Ave. Dielectric Constant			7.184	7.237				
Solution: As-Received Technical Grade TCA (Measured 11-5-88)								
0.050	---	155.86	155.83	160.17	---	0.0912	0.0091	0.0019
0.100	---	79.15	79.12	80.56	---	0.0964	0.0096	0.0012
0.150	---	52.99	52.97	53.77	---	0.0989	0.0098	0.0015
0.200	---	39.78	39.78	40.33	---	0.0989	0.0098	0.0016
0.250	---	31.77	31.76	32.18	---	0.0994	0.0099	0.0015
Ave. Dielectric Constant			7.220	7.270				
Solution: 0.05-Percent Rosin from Flux A (Measured 11-5-88)								
0.050	---	155.55	155.52	159.74	---	0.1202	0.0120	0.0021
0.100	---	79.11	79.08	80.52	---	0.1223	0.0122	0.0015
0.150	---	52.95	52.93	53.72	---	0.1206	0.0122	0.0018
0.200	---	39.74	39.73	40.28	---	0.1209	0.0122	0.0018
0.250	---	31.75	31.72	32.15	---	0.1215	0.0121	0.0015
Ave. Dielectric Constant			7.213	7.263				

Table A-2 Continued. Dissipation Factor and Capacitance Measurements of As-Received TCA and TCA Contaminated With Rosin

Gap Spacing (inch)	Capacitance (pF)				Dissipation Factor			
	1 kHz	10 kHz	100 kHz	1 MHz	1 kHz	10 kHz	100 kHz	1 MHz
Solution: 0.1-Percent Rosin from Flux A (Measured 11-5-88)								
0.050	---	155.80	155.79	160.02	---	0.1249	0.0125	0.0022
0.100	---	79.16	79.10	80.55	---	0.1283	0.0128	0.0015
0.150	---	52.97	52.95	53.75	---	0.1261	0.0127	0.0019
0.200	---	39.78	39.75	40.32	---	0.1261	0.0126	0.0020
0.250	---	31.76	31.76	32.18	---	0.1267	0.0127	0.0017
Ave. Dielectric Constant			7.218	7.269				
Solution: 0.5-Percent Rosin from Flux A (Measured 11-5-88)								
0.050	---	155.25	155.23	159.39	---	0.2146	0.0215	0.0032
0.100	---	78.94	78.89	80.36	---	0.2158	0.0217	0.0028
0.150	---	52.79	52.76	53.55	---	0.2118	0.0213	0.0028
0.200	---	39.64	39.63	40.17	---	0.2098	0.0212	0.0028
0.250	---	31.65	31.64	32.05	---	0.2096	0.0211	0.0026
Ave. Dielectric Constant			7.195	7.243				
Solution: 1-Percent Rosin from Flux A (Measured 11-5-88)								
0.050	---	154.87	155.84	159.06	---	0.3023	0.0303	0.0041
0.100	---	78.54	78.51	80.03	---	0.3000	0.0301	0.0039
0.150	---	52.59	52.58	53.41	---	0.2992	0.0301	0.0040
0.200	---	39.48	39.46	40.00	---	0.2986	0.0300	0.0035
0.250	---	31.55	31.52	31.93	---	0.2982	0.0301	0.0043
Ave. Dielectric Constant			7.168	7.203				
Solution: 3-Percent Rosin from Flux A (Measured 11-5-88)								
0.050	---	154.49	152.44	156.53	---	0.5682	0.0570	0.0072
0.100	---	77.33	77.30	78.72	---	0.5667	0.0569	0.0067
0.150	---	51.79	51.75	52.58	---	0.5661	0.0570	0.0069
0.200	---	38.88	38.86	39.43	---	0.5658	0.0568	0.0067
0.250	---	31.03	31.03	31.47	---	0.5662	0.0569	0.0066
Ave. Dielectric Constant			7.056	7.108				

Table A-2 Continued. Dissipation Factor and Capacitance Measurements of As-Received TCA and TCA Contaminated With Rosin

Gap Spacing (inch)	Capacitance (pF)				Dissipation Factor			
	1 kHz	10 kHz	100 kHz	1 MHz	1 kHz	10 kHz	100 kHz	1 MHz
Solution: 5-Percent Rosin from Flux A (Measured 10-11-88)								
0.050	---	148.79	148.76	152.79	---	0.7006	0.0703	0.0095
0.100	---	75.54	75.52	76.87	---	0.6991	0.0694	0.0069
0.150	---	50.62	50.58	51.34	---	0.6977	0.0685	0.0055
0.200	---	38.00	37.97	38.50	---	0.6970	0.0677	0.0040
0.250	---	30.36	30.31	30.74	---	0.6964	0.0673	0.0034
Ave. Dielectric Constant			6.894	6.940				
Solution: 10-Percent Rosin from Flux A (Measured 10-11-88)								
0.050	---	144.02	143.95	147.75	---	0.6921	0.0670	0.0042
0.100	---	73.16	73.12	74.41	---	0.6919	0.0676	0.0056
0.150	---	48.99	48.94	49.67	---	0.6924	0.0681	0.0063
0.200	---	36.79	36.75	37.25	---	0.6936	0.0698	0.0081
0.250	---	29.37	29.34	29.74	---	0.6949	0.0698	0.0094
Ave. Dielectric Constant			6.673	6.716				
Solution: 15-Percent Rosin from Flux A (Measured 10-11-88)								
0.050	---	138.47	138.33	141.84	---	0.6714	0.0677	0.0093
0.100	---	70.44	70.33	71.53	---	0.6704	0.0667	0.0072
0.150	---	47.18	47.09	47.77	---	0.6696	0.0657	0.0056
0.200	---	35.40	35.35	35.81	---	0.6694	0.0654	0.0060
0.250	---	28.32	28.26	28.62	---	0.6687	0.0650	0.0061
Ave. Dielectric Constant			6.421	6.459				
Solution: 20-Percent Rosin from Flux A (Measured 10-11-88)								
0.050	---	137.40	137.29	140.74	---	0.7617	0.0766	0.0095
0.100	---	69.56	69.50	70.65	---	0.7609	0.0757	0.0075
0.150	---	46.65	46.60	47.25	---	0.7602	0.0749	0.0064
0.200	---	35.04	34.97	35.43	---	0.7598	0.0744	0.0050
0.250	---	28.01	27.97	28.30	---	0.7596	0.0739	0.0044
Ave. Dielectric Constant			6.356	6.386				

Table A-2 Continued. Dissipation Factor and Capacitance Measurements of As-Received TCA and TCA Contaminated With Rosin

Gap Spacing (inch)	Capacitance (pF) 1 kHz	10 kHz	100 kHz	1 MHz	Dissipation Factor 1 kHz	10 kHz	100 kHz	1 MHz
Solution: 25-Percent Rosin from Flux B (Measured 10-11-88)								
0.050	---	130.89	130.89	133.82	---	0.6218	0.0633	0.0143
0.100	---	66.31	66.20	67.26	---	0.6216	0.0624	0.0122
0.150	---	44.4744	44.38	44.98	---	0.6211	0.0618	0.0099
0.200	---	33.38	33.31	33.74	---	0.6214	0.0612	0.0082
0.250	---	26.70	26.65	26.97	---	0.6214	0.0601	0.0036
Ave. Dielectric Constant			6.056	6.082				
Solution: 30-Percent Rosin from Flux B (Measured 10-11-88)								
0.050	---	126.12	125.98	128.95	---	0.3725	0.0376	0.0073
0.100	---	64.15	64.08	65.09	---	0.3710	0.0365	0.0041
0.150	---	42.99	42.94	43.51	---	0.3703	0.0357	0.0031
0.200	---	32.26	32.22	32.60	---	0.3702	0.0353	0.0022
0.250	---	25.79	25.75	26.05	---	0.3701	0.0350	0.0015
Ave. Dielectric Constant			5.851	5.880				
Solution: 30-Percent Rosin from Flux A (Measured 10-11-88)								
0.050	---	124.64	124.50	127.43	---	0.3705	0.0373	0.0066
0.100	---	63.31	63.23	64.21	---	0.3698	0.0361	0.0043
0.150	---	42.43	42.36	42.92	---	0.3700	0.0353	0.0031
0.200	---	31.84	31.78	32.16	---	0.3710	0.0351	0.0022
0.250	---	25.43	25.38	25.70	---	0.3730	0.0347	0.0009
Ave. Dielectric Constant			5.774	5.800				
Solution: 40-Percent Rosin from Flux B (Measured 11-5-88)								
0.050	---	119.29	119.13	121.68	---	0.2224	0.0241	0.0187
0.100	---	60.66	60.58	61.04	---	0.2233	0.0304	0.0710
0.150	---	40.64	40.57	40.82	---	0.2232	0.0298	0.0720
0.200	---	30.52	30.46	30.56	---	0.2235	0.0315	0.0725
0.250	---	24.40	24.35	24.51	---	0.2236	0.0279	0.0580
Ave. Dielectric Constant			5.533	5.518				

Table A-2 Continued. Dissipation Factor and Capacitance Measurements of As-Received TCA and TCA Contaminated With Rosin

Gap Spacing (inch)	Capacitance (pF)				Dissipation Factor			
	1 kHz	10 kHz	100 kHz	1 MHz	1 kHz	10 kHz	100 kHz	1 MHz
Solution: 50-Percent Rosin from Flux A (Measured 11-5-88)								
0.050	102.50	102.50	102.20	102.12	0.0777	0.0089	0.0084	0.0284
0.100	52.02	52.04	51.88	51.53	0.0785	0.0090	0.0083	0.0264
0.150	34.84	34.84	34.73	34.43	0.0782	0.0091	0.0083	0.0260
0.200	26.15	26.16	26.06	25.85	0.0780	0.0093	0.0083	0.0265
0.250	20.88	20.90	20.82	20.64	0.0780	0.0094	0.0083	0.0270
Ave. Dielectric Constant			4.743	4.657				

Table A-3. Dissipation Factor and Capacitance Measurements of Solutions With Unknown Contamination Type

Solution Source: Spray Booth
Suspected Major Contamination: Rosin
Nonvolatile Contamination: 0.32% by Weight
Solvent: TCE
Sample collected on 10-27-88
Measured on 11-5-88

Gap Spacing (inch)	Capacitance (pF)				Dissipation Factor			
	1 kHz	10 kHz	100 kHz	1 MHz	1 kHz	10 kHz	100 kHz	1 MHz
0.050	---	73.68	73.64	74.76	---	0.1340	0.0134	0.0018
0.100	---	37.45	37.42	37.81	---	0.1357	0.0136	0.0018
0.150	---	25.10	25.07	25.30	---	0.1368	0.0139	0.0017
0.200	---	18.86	18.83	19.00	---	0.1373	0.0140	0.0018
0.250	---	15.062	15.069	15.218	---	0.1368	0.0139	0.0012
Ave. Dielectric Constant			3.418	3.428				

Solution Source: Vapor Degreaser
Suspected Major Contamination: Oil
Nonvolatile Contamination: 0.15% by Weight
Solvent: TCA
Sample collected on 11-5-88
Measured on 11-5-88

Gap Spacing (inch)	Capacitance (pF)				Dissipation Factor			
	1 kHz	10 kHz	100 kHz	1 MHz	1 kHz	10 kHz	100 kHz	1 MHz
0.050	---	141.53	141.14	144.67	---	0.5430	0.0550	0.0062
0.100	---	71.89	71.73	72.99	---	0.5436	0.0549	0.0061
0.150	---	48.12	48.01	48.73	---	0.5409	0.0547	0.0064
0.200	---	36.13	36.04	36.55	---	0.5375	0.0544	0.0061
0.250	---	28.84	28.79	29.14	---	0.5362	0.0540	0.0052
Ave. Dielectric Constant			6.549	6.602				

Solution Source: Spray Booth
Suspected Major Contamination: Rosin
Nonvolatile Contamination: 3.6% by Weight
Solvent: TCE
Sample collected on 10-30-88
Measured on 11-5-88
Note: There was a large amount of alcohol floating on the TCE in the waste container.

Gap Spacing (inch)	Capacitance (pF)				Dissipation Factor			
	1 kHz	10 kHz	100 kHz	1 MHz	1 kHz	10 kHz	100 kHz	1 MHz
0.050	---	---	77.66	78.85	---	---	0.0941	0.0107
0.100	---	---	39.43	39.87	---	---	0.0943	0.0109
0.150	---	---	26.41	26.68	---	---	0.0944	0.0111
0.200	---	---	19.82	20.00	---	---	0.0944	0.0104
0.250	---	---	15.857	15.98	---	---	0.0943	0.0107
Ave. Dielectric Constant			3.418	3.428				

Table A-3 Continued. Dissipation Factor and Capacitance Measurements of Solutions With Unknown Contamination Type

Gap Spacing (inch)	Capacitance (pF)				Dissipation Factor			
	1 kHz	10 kHz	100 kHz	1 MHz	1 kHz	10 kHz	100 kHz	1 MHz
Solution Source: Vapor Degreaser					Solvent: TCA			
Suspected Major Contamination: Oils					Sample collected on 10-27-88			
Nonvolatile Contamination: 0.1% by Weight					Measured on 11-5-88			
0.050	74.18	74.21	74.19	75.36	0.0686	0.0070	0.0007	0.0006
0.100	37.70	37.72	37.71	38.14	0.0684	0.0072	0.0009	0.0005
0.150	25.25	25.28	25.27	25.51	0.0682	0.0073	0.0009	0.0004
0.200	18.97	18.98	18.97	19.14	0.0682	0.0074	0.0008	0.0005
0.250	15.15	15.184	15.180	15.336	0.0680	0.0068	0.0010	0.0000
Ave. Dielectric Constant			6.5485	6.602				
Solution Source: Vapor Degreaser					Solvent: TCE			
Suspected Major Contamination: is					Sample collected on 10-30-88			
Nonvolatile Contamination: 0.5% by Weight					Measured on 11-5-88			
0.050	73.39	73.43	73.41	74.53	0.1796	0.0182	0.0019	0.0008
0.100	37.34	37.36	37.34	37.75	0.1791	0.0183	0.0020	0.0008
0.150	24.98	25.00	24.99	25.23	0.1786	0.0183	0.0020	0.0006
0.200	18.76	18.78	18.76	18.93	0.1784	0.0185	0.0020	0.0006
0.250	15.00	15.027	15.025	15.175	0.1783	0.0178	0.0021	0.0001
Ave. Dielectric Constant			3.406	3.418				

Appendix B

BOILING POINT MEASUREMENTS OF AS-RECEIVED AND ROSIN-CONTAMINATED TRICHLOROETHYLENE AND 1,1,1-TRICHLOROETHANE

MATERIALS AND PROCEDURE

This work was accomplished during September and October 1988, with supplemental laboratory tests in January 1989.

SOLVENTS AND ROSIN

The trichloroethylene (TCE) used in all the laboratory experiments was from one 5-gallon can of technical grade solvent. The trichloroethane (TCA) used in all the laboratory experiments was from one lot of technical grade solvent supplied in 1-gallon glass containers.

The rosin used as contamination in the experiments was taken from two solder fluxes which meet MIL-F-14256 specification as types R and RMA. The rosin solvent carrier (alcohol) in the flux was removed by elevating the flux temperature to 80 to 90°C for 16 to 48 hours (90°C is well below the boiling point of the flux and rosin).

BOILING POINT MEASUREMENTS

For the purposes of this report, the boiling point is either the measured temperature of the vapor zone as stated in ASTM D 1078-86, "Distillation Range of Volatile Organic Liquids," or the measured temperature in the vapor zone or in the solution for some other described laboratory setup.

Initially, boiling point temperature measurements were made of the as-received and rosin-contaminated solvents according to ASTM D 1078. ASTM D 1078 requires that the mercury measuring thermometer be placed only in the vapor zone above the boiling solution and not in the solution. Measurements of the boiling point of as-received TCE and TCA according to ASTM D 1078 were made and are found in Tables B-1 and B-2. The ASTM D 1078 method gave inconsistent results for rosin-contaminated solutions. The ASTM D 1078 procedure is designed for measuring the distillation range of volatile organic liquids, that is, pure chemicals, and has severe limitations if applied to contaminated solutions. To measure the boiling point of contaminated solutions, the temperature probe must be in the solution, not above it as is the common laboratory practice for pure chemicals.

A modified ASTM method was used to measure the boiling points of the contaminated solutions and is shown as Figure 11 in the main body of this report. As the solution is distilled, the contamination level of the solution increases. The contamination level at any point can be calculated from the known amount of rosin in solution, the initial amount of solvent in the flask, and the amount of solvent distilled. The following equation was used to calculate the contamination percent by weight.

$$W\% = \frac{Wr}{(Vts-Vds)Sg + Wr} = \frac{1}{\left[(Vts-1.5-Vds)\,\frac{Sg}{Wr}\right] + 1} ,$$

where Wr is the rosin weight, Vts is the total volume of the initial amount of pure solvent, Vds is the amount of distilled solvent, 1.5 mL of solvent is estimated to be condensed on the surfaces of the glass ware, and Sg is the specific gravity of the pure solvent. Vds and Sg are measured values. Wr and Vts can be calculated by knowing the specific gravity of a solution as some weight percent (W%) of rosin contamination by the following equation. The total volume of contaminated solvent is 250 mL in this example.

$$Wt = [250\ \text{mL}]\ Sg(5\%) ,$$

$$= 356.13\ \text{g} ;$$

$$Wr = 0.05\ Wt ,$$

$$= 17.806\ \text{g} ; \text{ and}$$

$$Vts = \frac{Wt - Wr}{Sg(0\%)} ,$$

$$= 233.0\ \text{mL} .$$

The temperature was measured with a thermocouple in the solution as the solvent was being distilled. The thermocouple was 120 mm lower than the mercury thermometer and at about the 50-mL of solution level. The data in all the tables (B-1 through B-8) have been corrected to 760 mm Hg pressure. In Tables B-4 through B-7, the boiling point temperature change caused by contamination was calculated by substracting the boiling point of as-received solvent from the boiling point of contaminated solvent for the same amount of distilled solvent.

Table B-1. Boiling Point Measurements of As-Received and Rosin-Contaminated TCE According to ASTM D 1078 (100-mL Solution)

	Solution Boiling Point (°C)								
Amount Distilled (mL)	As-Received TCE		Rosin in TCE (percent by weight)						
	Test 1	Test 2	5# A*	10# A*	15 A*	20 B*	25 B*	30 B*	30# A*
First Drop	86.6	86.4	85.6	84.7	84.0	84.5	84.2	84.5	81.5
5	86.8	86.7	86.3	86.1	85.6	85.6	85.6	85.5	84.5
10	86.8	86.8	86.4	86.4	86.0	86.0	86.1	86.0	84.9
20	86.9	86.8	86.7	86.5	86.3	86.4	86.4	86.3	86.1
30	86.9	86.9	86.8	86.8	86.6	86.6	86.6	86.5	86.6
40	86.9	86.9	86.9	86.8	86.8	86.6	87.1	87.7	87.0
50	87.0	86.9	86.9	86.9	87.0	86.8	87.2	92.5	87.9
60	87.1	86.9	87.0	87.0	87.0	87.2	94.0	95.5	89.6
70	87.1	87.0	87.0	87.2	87.4	92.3	96.0	—	—
80	87.1	87.0	87.1	87.6	—	—	—	—	—
90	87.2	87.1	87.9	90.0	—	—	—	—	—
95	87.6	87.2	90.8	—	—	—	—	—	—
Dry	87.6	87.4	—	—	—	—	—	—	—
Solvent Recovered (mL)	98.5	98.5							

#Average of two measurements.
*Indicates rosin source, types A and B solder flux.

Table B-2. Boiling Point Measurements of As-Received and Rosin-Contaminated TCA According to ASTM D 1078 (100-mL Solution)

	Solution Boiling Point (°C)								
			Rosin in TCA (percent by weight)						
Amount Distilled (mL)	As-Received TCA Test 1	As-Received TCA Test 2	5# A*	10# A*	15 A*	20 A*	25 B*	30 B*	30# A*
First Drop	73.5	73.5	72.9	72.2	72.1	72.8	84.2	84.2	71.4
5	73.8	73.8	73.4	73.3	73.1	73.4	75.6	75.5	72.5
10	73.8	73.9	73.7	73.7	73.3	73.6	76.1	76.0	72.9
20	74.0	74.0	73.9	73.8	73.6	73.9	76.4	76.4	73.2
30	74.0	74.0	74.0	74.0	73.8	74.0	76.6	76.6	73.5
40	74.1	74.1	74.1	74.1	74.0	74.2	77.1	77.7	74.1
50	74.2	74.2	74.2	74.2	74.1	74.4	77.2	91.5	75.3
60	74.3	74.3	74.3	74.4	74.4	74.8	94.0	93.9	83.7
70	74.5	74.4	74.5	74.7	75.1	76.6	96.0	--	--
80	74.7	74.6	74.8	75.6	80.0	--	--	--	--
90	75.3	75.2	77.0	--	--	--	--	--	--
95	78.5	76.1	--	--	--	--	--	--	--
Dry	98.5	78.8	--	--	--	--	--	--	--
Solvent Recovered (mL)	98.5	98.5							

#Average of two measurements.
*Indicates rosin source, Types A and B solder flux.

Table B-3. Boiling Point Measurements of As-Received TCE and TCA Using 250 mL of Solution

Amount Distilled (mL)	Boiling Point (°C) TCE In Vapor	Boiling Point (°C) TCA In Vapor
First Drop	85.7	73.6
5	86.6	----
10	86.7	73.9
15	86.7	73.9
20	86.8	73.9
25	86.8	74.0
30	86.8	74.0
35	86.8	74.0
40	86.8	74.0
45	86.8	74.0
50	86.8	74.1
55	86.8	74.1
60	86.8	74.1
65	86.8	74.1
70	86.9	74.1
75	86.9	74.1
80	86.9	74.1
85	86.9	74.1
90	86.9	74.1
95	86.9	74.1
100	86.9	74.1
105	86.9	74.2
110	86.9	74.2
115	86.9	74.2
120	86.9	74.2
125	86.9	74.2
130	87.0	74.2
135	87.0	74.2
140	87.0	74.2
145	87.0	74.2
150	87.0	74.3
155	87.0	74.3
160	87.0	74.4
165	87.0	74.4
170	87.0	74.4

Table B-3 Continued. Boiling Point Measurements of As-Received TCE and TCA Using 250 mL of Solution

Amount Distilled (mL)	Boiling Point (°C)	
	TCE In Vapor	TCA In Vapor
175	87.0	74.5
180	87.0	74.5
185	87.0	74.5
190	87.1	74.6
195	87.1	74.6
200	87.1	74.6
210	87.1	74.8
220	87.1	75.0
230	87.1	75.3
240	87.2	76.3
245	87.3	----
Dry Point	87.6	79.1
Solvent Recovered (mL)	247	247

Table B-4. Boiling Point Measurements of As-Received and Rosin-Contaminated TCE With the Thermometer in the Vapor Zone (250-mL Solution)

Amount Distilled (mL)	Temp. in As-Received Solvent ({C)	Temp. in Contaminated Solvent ({C)		Temp. Change Caused by Contamination ({C)		Contamination Level (percent by weight)
		A*	B*	A*	B*	
First Drop	85.7	85.8	85.6	0.1	-0.1	5.00
5	86.6	86.3	86.3	-0.3	-0.3	5.14
10	86.7	86.3	86.5	-0.4	-0.2	5.25
15	86.7	86.4	86.5	-0.3	-0.2	5.36
20	86.8	86.5	86.6	-0.3	-0.2	5.48
25	86.8	86.5	86.6	-0.3	-0.2	5.61
30	86.8	86.5	86.6	-0.3	-0.2	5.74
35	86.8	86.6	86.7	-0.2	-0.1	5.87
40	86.8	86.6	86.7	-0.2	-0.1	6.00
45	86.8	86.7	86.7	-0.1	-0.1	6.17
50	86.8	86.7	86.7	-0.1	-0.1	6.35
55	86.8	86.7	86.7	-0.1	-0.1	6.50
60	86.8	86.8	86.7	0.0	-0.1	6.67
65	86.8	86.8	86.7	0.0	-0.1	6.86
70	86.9	86.8	86.8	-0.1	-0.1	7.06
75	86.9	86.8	86.8	-0.1	-0.1	7.29
80	86.9	86.8	86.8	-0.1	-0.1	7.49
85	86.9	86.9	86.8	0.0	-0.1	7.72
90	86.9	86.9	86.8	0.0	-0.1	7.98
95	86.9	86.9	86.8	0.0	-0.1	8.24
100	86.9	86.9	86.8	0.0	-0.1	8.53
105	86.9	86.9	86.9	0.0	0.0	8.84
110	86.9	86.9	86.9	0.0	0.0	9.17
115	86.9	86.9	86.9	0.0	0.0	9.52
120	86.9	86.9	86.9	0.0	0.0	9.91
125	86.9	86.9	86.9	0.0	0.0	10.33
130	87.0	86.9	86.9	-0.1	-0.1	10.78
135	87.0	86.9	86.9	-0.1	-0.1	11.28
140	87.0	86.9	86.9	-0.1	-0.1	11.82
145	87.0	87.0	86.9	0.0	-0.1	12.42
150	87.0	87.0	86.9	0.0	-0.1	13.08
155	87.0	87.0	86.9	0.0	-0.1	13.82
160	87.0	87.0	86.9	0.0	-0.1	14.64
165	87.0	87.0	86.9	0.0	-0.1	15.57
170	87.0	87.0	86.9	0.0	-0.1	16.63

Table B-4 Continued. Boiling Point Measurements of As-Received and Rosin-Contaminated TCE With the Thermometer in the Vapor Zone (250-mL Solution)

Amount Distilled (mL)	Temp. in As-Received Solvent ({C)	Temp. in Contaminated Solvent ({C)		Temp. Change Caused by Contamination ({C)		Contamination Level (Percent by Weight)
		A*	B*	A*	B*	
175	87.0	87.0	87.0	0.0	0.0	17.83
180	87.0	87.1	87.0	0.1	0.0	19.23
185	87.0	87.1	87.0	0.1	0.0	20.87
190	87.1	87.1	87.0	0.0	0.0	22.81
195	87.1	87.1	87.0	0.0	0.0	25.15
200	87.1	87.1	87.0	0.0	0.0	28.02
205	87.1	---	---	---	---	---
210	87.1	87.1	87.1	0.0	0.0	36.32
215	87.2	---	---	---	---	---
220	87.3	87.3	87.3	---	---	51.60
225	---	---	87.5	---	---	65.36
228	---	87.5	---	---	---	77.80
230	87.6	---	87.9	---	---	89.10

*A and B denotes the flux from which the rosin was obtained.

Table B-5. Boiling Point Measurements of As-Received and Rosin-Contaminated TCE With the Thermometer in the Solution (250-mL Solution)

Amount Distilled (mL)	Temp. in As-Received Solvent ({C)	Temp. in Contaminated Solvent ({C)		Temp. Change Caused by Contamination ({C)		Contamination Level (percent by weight)
		A*	B*	A*	B*	
First Drop	87.6	87.8	87.9	0.2	0.3	5.00
5	87.8	87.9	88.0	0.1	0.2	5.14
10	87.9	88.1	88.4	0.2	0.2	5.25
15	87.8	88.2	88.4	0.4	0.6	5.36
20	87.9	88.2	88.6	0.3	0.7	5.48
25	87.9	88.2	88.6	0.3	0.7	5.61
30	87.9	88.3	88.6	0.4	0.7	5.74
35	88.0	88.4	88.6	0.4	0.6	5.87
40	88.0	88.4	88.6	0.4	0.6	6.02
45	88.0	88.5	88.7	0.5	0.7	6.17
50	88.0	88.5	88.7	0.5	0.7	6.35
55	88.0	88.5	88.7	0.5	0.7	6.50
60	88.0	88.5	88.8	0.5	0.8	6.67
65	88.0	88.6	88.9	0.6	0.9	6.86
70	88.0	88.8	88.9	0.8	0.9	7.06
75	88.0	88.8	88.9	0.8	0.9	7.29
80	88.0	88.8	89.0	0.8	1.0	7.49
85	88.0	88.8	89.0	0.8	1.0	7.72
90	88.1	89.0	89.0	0.9	0.9	7.98
95	88.2	89.0	89.1	0.8	0.9	8.24
100	88.1	89.1	89.1	1.0	1.0	8.53
105	88.2	89.1	89.1	1.1	0.9	8.84
110	88.0	89.2	89.2	1.2	1.2	9.17
115	88.1	89.1	89.4	1.0	1.3	9.52
120	88.0	89.2	89.3	1.2	1.3	9.91
125	88.1	89.2	89.5	1.1	1.4	10.33
130	88.0	89.3	89.5	1.3	1.5	10.78
135	88.0	89.4	89.4	1.4	1.4	11.28

*A and B denotes the flux from which the rosin was obtained.

Table B-6. Boiling Point Measurements of As-Received and Rosin-Contaminated TCA With the Thermometer in the Vapor Zone (250-mL Solution)

Amount Distilled (mL)	Temp. in As-Received Solvent ({C)	Temp. in Contaminated Solvent ({C)		Temp. Change Caused by Contamination ({C)		Contamination Level (percent by weight)
		A*	B*	A*	B*	
First Drop	73.6	73.1	73.3	-0.5	-0.3	5.0
5	---	73.4	73.6	0.0	---	5.14
10	73.9	73.7	73.8	-0.2	-0.1	5.24
15	73.9	73.8	73.9	-0.1	0.0	5.36
20	73.9	73.8	73.9	-0.1	0.0	5.48
25	74.0	73.8	74.0	-0.2	0.0	5.60
30	74.0	73.9	74.0	-0.1	0.0	5.73
35	74.0	73.9	74.1	-0.1	0.1	5.87
40	74.0	74.0	74.1	0.0	0.1	6.01
45	74.0	74.0	74.1	0.0	0.1	6.16
50	74.1	74.0	74.1	-0.1	0.0	6.32
55	74.1	74.0	74.1	-0.1	0.0	6.48
60	74.1	74.1	74.1	0.0	0.0	6.66
65	74.1	74.1	74.1	0.0	0.0	6.84
70	74.1	74.1	74.1	0.0	0.0	7.04
75	74.1	74.1	74.1	0.0	0.0	7.24
80	74.1	74.1	74.2	0.0	0.1	7.46
85	74.1	74.1	74.2	0.0	0.1	7.69
90	74.1	74.1	74.2	0.0	0.1	7.94
95	74.1	74.2	74.2	0.1	0.1	8.21
100	74.1	74.2	74.2	0.1	0.1	8.49
105	74.2	74.2	74.2	0.0	0.0	8.79
110	74.2	74.2	74.3	0.0	0.1	9.11
115	74.2	74.2	74.3	0.0	0.1	9.46
120	74.2	74.3	74.3	0.1	0.1	9.84
125	74.2	74.3	74.3	0.1	0.1	10.25
130	74.2	74.3	74.3	0.1	0.1	10.69
135	74.2	74.3	74.3	0.1	0.1	11.18
140	74.2	74.4	74.3	0.2	0.1	11.71
145	74.2	74.4	74.4	0.2	0.2	12.29
150	74.3	74.4	74.4	0.1	0.1	12.93
155	74.3	74.5	74.4	0.2	0.1	13.65
160	74.4	74.5	74.5	0.1	0.1	14.44
165	74.4	74.5	74.5	0.1	0.1	15.34
170	74.4	74.6	74.6	0.2	0.2	16.36

Table B-6 Continued. Boiling Point Measurements of As-Received and Rosin-Contaminated TCA With the Thermometer in the Vapor Zone (250-mL Solution)

Amount Distilled (mL.)	Temp. in As-Received Solvent ([C)	Temp. in Contaminated Solvent ([C)		Temp. Change Caused by Contamination ([C)		Contamination Level (percent by weight)
		A*	B*	A*	B*	
175	74.5	74.6	74.6	0.1	0.1	17.52
180	74.5	74.7	74.6	0.2	0.1	18.85
185	74.5	74.7	74.6	0.2	0.1	20.41
190	74.6	74.8	74.7	0.2	0.1	22.25
195	74.6	74.8	74.7	0.2	0.1	24.45
200	74.6	74.9	74.9	0.3	0.3	27.14
210	74.8	75.2	75.2	0.4	0.4	34.78
220	75.0	75.8	76.0	0.8	1.0	48.41
228	---	77.1	---	1.8	---	70.53
230	75.3	---	78.4	---	3.1	79.63
240	76.3	---	---	---	---	---
247 (Dry Point)	79.1	---	---	---	---	---

*A and B denote the flux from which the rosin was obtained.

Table B-7. Boiling Point Measurements of As-Received and Rosin-Contaminated TCA With the Thermometer in the Solution (250-mL Solution)

Amount Distilled (mL)	Temp. in As-Received Solvent ([C)	Temp. in Contaminated Solvent ([C)		Temp. Change Caused by Contamination ([C)		Contamination Level (Percent by Weight)
		A*	B*	A*	B*	
First Drop	74.6	74.6	74.6	0.0	0.0	5.0
5	74.6	74.8	74.9	0.2	0.3	5.1
10	74.8	75.0	75.0	0.2	0.2	5.2
15	74.8	75.1	75.2	0.3	0.4	5.3
20	74.8	75.2	75.3	0.4	0.5	5.4
25	75.0	75.1	75.4	0.1	0.4	5.6
30	74.9	75.3	75.3	0.4	0.4	5.7
35	74.9	75.3	75.4	0.4	0.5	5.8
40	74.9	75.3	75.5	0.4	0.6	6.0
45	74.9	75.4	75.5	0.5	0.6	6.1
50	74.9	75.4	75.5	0.5	0.6	6.3
55	75.1	75.5	75.5	0.4	0.4	6.4
60	75.1	75.5	75.6	0.4	0.5	6.6
65	75.1	75.5	75.7	0.4	0.6	6.8
70	75.0	75.4	75.7	0.4	0.7	7.0
75	75.1	75.6	75.8	0.5	0.7	7.2
80	75.1	75.6	75.8	0.5	0.7	7.4
85	75.2	75.7	75.9	0.5	0.7	7.6
90	75.1	75.7	76.0	0.6	0.9	7.9
95	75.2	75.8	76.0	0.6	0.8	8.1
100	75.2	75.8	76.1	0.6	0.9	8.4
105	75.2	75.9	76.1	0.7	0.9	8.7
110	75.2	76.0	76.1	0.8	0.9	9.0
115	75.3	76.1	76.2	0.8	0.9	9.4
120	75.4	76.1	76.2	0.7	0.8	9.7
125	75.3	76.2	76.4	0.9	1.1	10.1
130	75.3	76.3	76.4	1.0	1.1	10.6
135	75.4	76.3	76.5	0.9	1.1	11.0
140	75.4	76.3	76.5	0.9	1.1	11.5

*A and B denote the flux from which the rosin was obtained.

Table B-8. Boiling Point Measurements of Solvents With Unknown Contamination From Various Manufacturing Departments

Amount Distilled (mL)	Solution Boiling Point* (°C) Related to Solvent Manufacturing Department Source					
	Timer Assembly		Multiwire Cables		Electronic Assembly	
	S	V	S	V	S	V
First Drop	76.9	74.8	87.7	86.5	76.4	69.3
10	77.9	76.5	87.8	87.0	78.8	75.1
20	78.0	76.7	87.7	87.1	80.5	77.2
30	78.0	76.7	87.8	87.1	81.7	80.1
40	78.1	76.7	87.9	87.2	82.3	80.9
50	78.1	76.7	87.9	87.2	82.8	81.0
60	78.2	76.9	87.8	87.2	83.0	81.8
70	78.3	77.0	87.8	87.2	83.4	82.2
80	78.5	76.9	87.9	87.2	83.7	82.4
90	78.6	76.8	87.9	87.2	83.9	82.7
100	78.7	76.8	87.9	87.2	84.3	83.1
110	78.6	77.0	87.9	87.2	84.4	83.4
120	78.8	77.1	87.9	87.2	84.8	83.5
130	79.0	77.0	87.9	87.2	85.1	84.1
140	79.1	77.1	87.9	87.2	85.4	84.3
150	79.2	77.1	87.9	87.2	85.6	84.5
160	79.3	77.1	87.9	87.2	85.9	84.8
170	79.6	77.3	88.0	87.2	86.1	85.1
180	79.7	77.3	87.9	87.2	86.3	85.2
190	79.8	77.5	87.9	87.2	86.6	85.5
200	80.0	77.6	87.9	87.2	86.7	85.7
210	80.1	77.6	87.9	87.2	86.8	86.0
220	80.4	77.8	87.9	87.2	87.1	86.1
230	80.6	77.9	87.9	87.2	87.2	86.2
240	80.8	77.8	87.9	87.2	87.3	86.4
250	81.2	78.2	87.9	87.2	87.4	86.5
Nonvolatile** (% by Weight)	2.1 to 4.2		0.32 to 0.65		0.41 to 0.81	

Key:
S denotes temperature measured in the solution, and V denotes temperature measured in the vapor zone.
* 250 mL of solvent were distilled from 500 mL in a 1000-mL flask.
Measurements of a sample taken in December 1988.
** The two values are the initial and final concentrations of nonvolatile residue in the boiling flask.

Table B-8 Continued. Boiling Point Measurements of Solvents With Unknown Contamination From Various Manufacturing Departments

Amount Distilled (mL)	Solution Boiling Point* (°C) Related to Solvent Manufacturing Department Source					
	PWB Fabrication		Electrical Assembly		Plating and Chem Mill	
	S	V	S	V	S	V
First Drop	76.2	74.9	86.7	84.6	87.9	87.0
10	76.3	75.6	87.2	85.7	88.1	87.2
20	76.4	75.8	87.4	86.2	88.1	87.2
30	76.5	75.8	87.7	86.5	88.1	87.3
40	--	--	87.8	86.6	88.1	87.3
50	--	--	87.9	86.7	88.1	87.3
60	76.6	75.9	88.1	86.9	88.1	87.3
70	76.6	76.0	88.1	87.0	88.1	87.3
80	--	--	88.2	87.0	88.1	87.3
90	76.6	76.1	88.2	87.1	88.1	87.3
100	--	--	88.3	87.1	88.1	87.3
110	76.7	76.1	88.4	87.1	88.1	87.3
120	--	--	88.4	87.1	88.1	87.3
130	--	--	88.4	87.1	88.1	87.3
140	--	--	88.5	87.2	88.1	87.3
150	76.8	76.2	88.5	87.2	88.1	87.3
160	77.0	76.3	88.5	87.2	88.1	87.3
170	--	--	88.6	87.2	88.1	87.3
180	77.0	76.3	88.6	87.2	88.1	87.3
190	77.1	76.3	88.6	87.2	88.2	87.4
200	--	--	88.8	87.2	88.1	87.4
210	77.2	76.5	88.8	87.2	88.2	87.4
220	77.3	76.5	89.0	87.2	88.2	87.4
230	77.4	76.5	88.8	87.2	88.2	87.4
240	77.5	76.7	89.0	87.2	88.2	87.4
250	77.5	76.8	89.1	87.2	88.2	87.4
Nonvolatile* (% by Weight)	0.15 to 0.30		3.6 to 7.2		0.09 to 0.19	

Key:

S denotes temperature measured in the solution, and V denotes temperature measured in the vapor zone.

* 250 mL of solvent were distilled from 500 mL in a 1000-mL flask.

\# Measurements of a sample taken in October 1988.

* The two values are the initial and final concentrations of nonvolatile residue in the boiling flask.

Table B-8 Continued. Boiling Point Measurements of Solvents With Unknown Contamination From Various Manufacturing Departments

Amount Distilled (mL)	Solution Boiling Point* (°C) Related to Solvent Manufacturing Department Source			
	Heat Treating		Heat Treating	
	S	V	S	V
First Drop	88.0	86.8	88.5	86.9
10	88.1	87.0	88.7	87.1
20	88.2	87.1	88.8	87.2
30	88.3	87.1	88.8	87.2
40	88.3	87.2	88.7	87.2
50	88.3	87.2	88.8	87.3
60	88.4	87.2	88.9	87.3
70	88.4	87.2	88.9	87.3
80	88.4	87.2	88.9	87.3
90	88.5	87.2	89.0	87.3
100	88.5	87.2	89.0	87.3
110	88.5	87.2	88.9	87.3
120	88.6	87.2	89.1	87.3
130	88.6	87.2	89.1	87.3
140	88.6	87.2	89.1	87.3
150	88.6	87.2	89.3	87.3
160	88.6	87.2	89.3	87.3
170	88.6	87.2	89.4	87.3
180	88.7	87.2	89.4	87.3
190	88.8	87.2	89.4	87.3
200	88.8	87.2	89.6	87.3
210	88.8	87.3	89.5	87.3
220	88.9	87.3	89.6	87.3
230	88.9	87.3	89.7	87.3
240	89.0	87.3	89.9	87.3
250	89.0	87.3	89.9	87.3
Nonvolatile** (% by Weight)	0.5 to	1.0	1.4 to	2.9

Key:

S denotes temperature measured in the solution, and V denotes temperature measured in the vapor zone.

* 250 mL of solvent were distilled from 500 mL in a 1000-mL flask.

\# Measurements of a sample taken in October 1988.

\## Measurements of a sample taken in December 1988.

** The two values are the initial and final concentrations of nonvolatile residue in the boiling flask.

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