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Chapter 14

FACT OR FICTION: THE SOURCE OF PERCHLOROETHYLENE CONTAMINATION IN GROUNDWATER IS A MANUFACTURING IMPURITY IN CHLORINATED SOLVENTS

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ABSTRACT

Manufacturing impurities in chlorinated solvents have been considered to be sources of contamination in groundwater. Chlorinated solvents are manufactured in a variety of grades; the technical grade is used at many industrial and manufacturing facilities. Compounds present as manufacturing impurities in technical grade chlorinated solvents vary, and their quantity is extremely small or not measurable, because chlorinated solvents historically have been manufactured to a high degree of purity. The purity of currently manufactured TCE ranges from 99.9% for reagent grade to 98.0% for the technical grade. Impurities in technical grade 1,2-dichloroethane, also known as ethylene dichloride (EDC), manufactured within the last 10 years with purities of 99.9991% and 99.9955% contained PCE between about 0.0001% and 0.0006%, respectively.

In a number of litigation cases where TCE released from a vapor degreaser is the major contaminant in groundwater, there is an accompanying minor concentration of PCE. The assumption made in these cases is that the PCE impurity in technical grades of TCE is the source of the PCE in groundwater. This assumption is based on the release of these two compounds together from a distillation bottom residue where PCE is concentrated, relative to the TCE, because of its much higher boiling point at atmospheric pressure.

There is no peer reviewed literature that can be cited to prove that PCE is a significant impurity in either the distillation bottoms produced from a vapor degreaser or technical grade TCE. PCE, if present as a manufacturing impurity, is present in such small amounts that significant concentrations would not be generated in groundwater. When PCE and TCE are present together in groundwater, the source of the PCE is likely not an impurity in the manufactured TCE.

Keywords: PCE, Manufacturing impurity, Vapor degreasing

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1. INTRODUCTION

When chlorinated solvents are manufactured, the resulting product often contains less than 100% of the chlorinated solvent itself. Very small amounts of other chlorinated solvents may also be present as “impurities.” Manufacturing impurities (MIs) in chlorinated solvents have been considered to be sources of contamination in groundwater. One chlorinated compound in particular, tetrachloroethylene (PCE), has been identified as a MI source of groundwater contamination.

PCE, as a manufacturing impurity (PCEMI), has been identified as the source of PCE at sites where trichloroethylene (TCE) releases from vapor degreaser operations occurred. In a number of litigation cases, in which TCE released from a vapor degreaser is the major contaminant in groundwater, there has been an accompanying minor concentration, relative to the TCE, of PCE in groundwater. The assumption made in these cases is that the PCEMI in technical grades of TCE is the source of the PCE in groundwater.

There is no peer-reviewed literature documenting studies that can be cited to show that PCE is a significant MI in either the distillation bottoms produced from a vapor degreaser or in technical grade TCE. Furthermore, there is a dearth of available data that quantifies the MIs in chlorinated solvents. There is some data available for the purity of manufactured TCE, but, to date, no quantifying data has been found in the literature for the PCE MI in TCE.

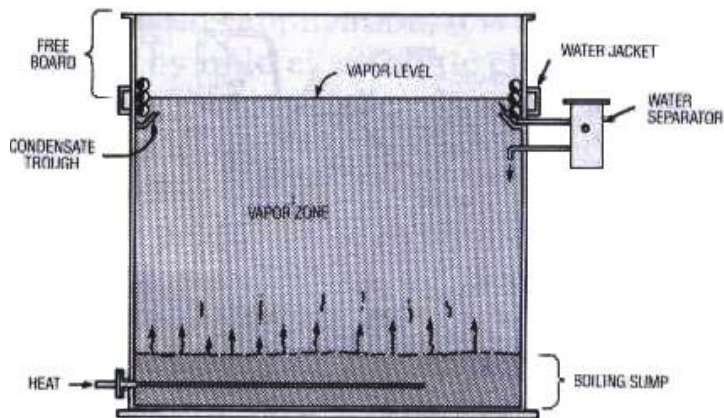
Although no published quantifying data for the PCEMI in TCE has been found to date, there is some quantifying data available for the PCEMI in TCE and in 1,2-dichloroethane (1,2-DCA). The data for the quantity of PCEMI in TCE is from a chlorinated solvent manufacturer. The data for the quantity of PCEMI in 1,2-DCA, also from a chlorinated solvent manufacturer, is from a site investigation performed at a single-component dense non-aqueous phase liquid (DNAPL) release site. These data provide further insight into the amount of PCE MI present in manufactured chlorinated solvents.

The purpose of this paper is to facilitate a more thorough understanding of the fate of MIs at sites where groundwater contamination has resulted from releases of chlorinated solvents. Two cases are examined: a conceptual model where TCE is used as the solvent in a vapor degreaser and an actual case study where 1,2-DCA, known to contain PCE MI, was released to the ground surface. Preliminary conclusions regarding the fate of MIs, such as PCE, can be made by examining these two cases.

2. CONCEPTUAL MODEL: VAPOR DEGREASER OPERATION USING TCE

The function of a vapor degreaser is to remove process oil, grease, other residues, and small particulate from non-porous materials such as metals. A solvent is heated to boiling to create a solvent vapor that condenses on a cooler metal object. Through this process, the oil, grease, other residues, and particulate, such as sand and metal grit, are stripped from the metal as the

solvent condensate drips from the metal object back into the boiling solvent. Over time, sludge forms in the bottom of the degreaser. This sludge, commonly referred to as distillation bottoms, contains the solvent plus the materials formerly coating the degreased objects. Figure 1 shows the design of a basic vapor degreaser (ASTM 1989).



Several chlorinated solvents have historically been used in vapor degreasers. They include methylene chloride, PCE, TCE, 1,1,1-trichloroethane, and trichlorotrifluoroethane (ASTM 1989). By 1991, 90% of the TCE produced was used for metal cleaning and degreasing (Doherty 2000).

At some sites where TCE was used as the vapor degreasing solvent and where PCE was detected in conjunction with TCE in groundwater, the assumption has been that PCEMI in TCE was the source of the PCE groundwater contamination. This assumption is based on a common sense approach to the release of these two chemicals together from a distillation bottom residue where PCE is concentrated, relative to the TCE, because of its much higher boiling point at atmospheric pressure.

Groundwater concentrations of PCE relative to TCE at vapor degreaser sites have been reported in the 0.5 to 5 percent range (ODHHS 2003). Assume a maximum concentration of 100 ppm PCEMI in TCE. Also assume the distillation bottoms contain 20% TCE and 500 ppm PCE relative to the TCE. The equilibrium solubility of PCE and TCE in water are 200 mg/L and 1,100 mg/L, respectively (Schwille 1988). TCE is five and a half times more soluble in water than PCE at equilibrium, and it is 2,000 times higher in concentration than PCE. This is an effective multiplier of 11,000. In terms of a rough estimate for a concentration of 100 $\mu\text{g/L}$ TCE in groundwater, it would be expected that the maximum PCE concentration generated from a manufacturing impurity would be about 0.01 $\mu\text{g/L}$ or 0.01% of the TCE in the groundwater.

Another approach to evaluation of the potential for PCEMI contamination of groundwater from distillation bottom residue is as follows: If there is 1% PCE in the groundwater relative to TCE, then the amount of PCEMI in the TCE representing the distillation bottoms would be in the neighborhood of 20%. This represents approximately 4% PCEMI in the manufactured TCE. This is three to four orders of magnitude greater than the 0.0001% (Table 1) to 0.001% of measured PCEMI, discussed in the next section.

2.1 Purity and Composition of Manufactured TCE

Chlorinated solvents are manufactured in a variety of grades; the technical grade is used at many industrial and manufacturing facilities. The purity of manufactured TCE is usually greater

than 99% (CCOHS 1999), and TCE purity in the technical grade has been cited as high as 99.97% (Morrison 2000). Other chlorinated solvents, such as PCE, dichloroethylene isomers, and trichloroethane isomers, may be present in TCE as MIs (CCOHS 1999, IPCS 1985). The quantity of these MIs is extremely small or nonexistent. Manufactured TCE may also contain stabilizers and additives. Stabilizers may be present in amounts between 0.1 to 0.5% and as much as 2.0% (Doherty 2000). Neu-Tri*E is TCE that has been manufactured specifically for vapor degreasing. The purity of TCE in Neu-Tri*E is 99.4%; stabilizers and additives comprise 0.1% and 0.5%, respectively (The Dow Chemical Company 1991, 1994, 1999).

For the past 10 to 15 years, TCE produced by a major manufacturer has contained less than the PCE detection limit, which is one ppm by weight PCEMI (Table 1) (Terry 2006). The PCEMI represents less than 0.0001% of the manufactured TCE solvent. Prior to that time, the greatest quantity of PCMI in TCE was 10 ppm, or 0.001%, which rarely occurred (Terry 2006). Because the boiling points of PCE and TCE are 121.5° C and 87.2° C, respectively (CRC 1995), separation of PCE from TCE by atmospheric distillation is easily accomplished. This is strongly supported by manufacturer product quality test results.

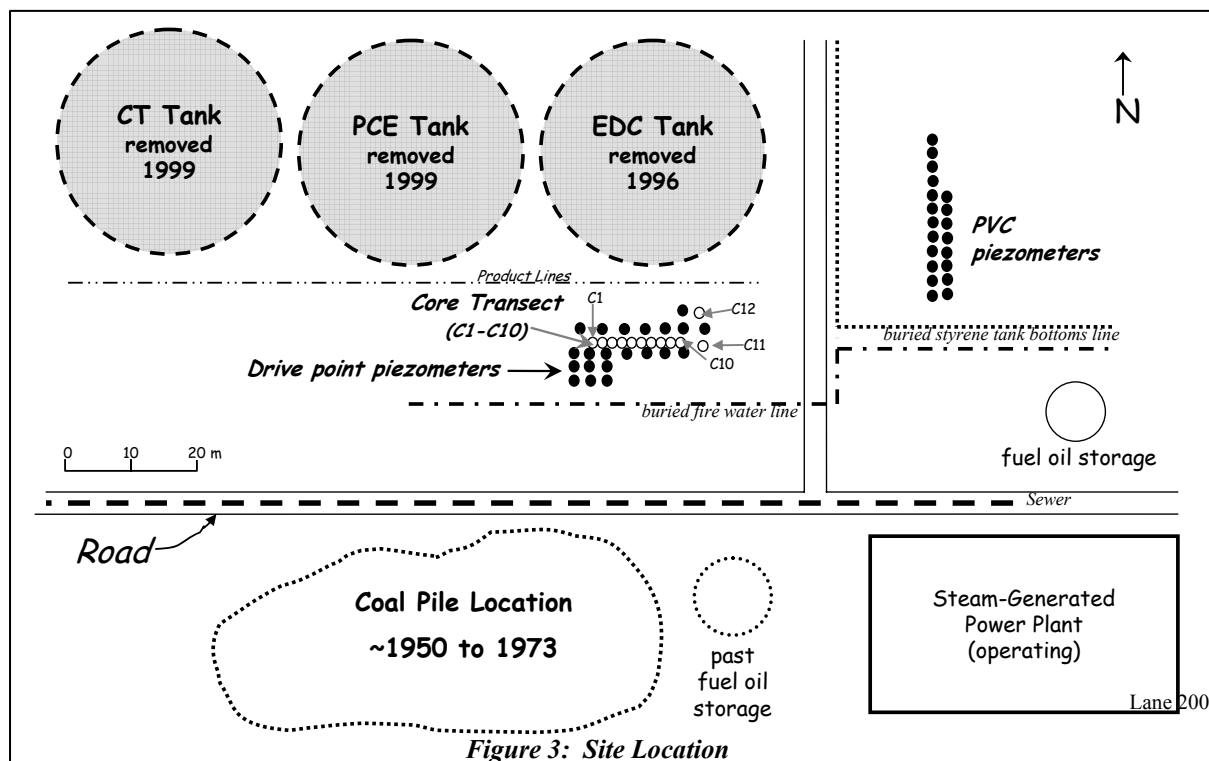
Table 1. Manufacturer's Quantification of PCEMI in TCE for the Past 10 to 15 Years

<i>Compound</i>	<i>MW</i> (g/mol)	<i>Quantity</i> (ppm w/w) ^a	<i>Fraction</i> <i>by Mass</i>	<i>Moles</i>	<i>Mole</i> <i>Fraction</i>	<i>Solubility</i> (mg/L)	<i>Solubility_{eff}</i> (mg/L)
TCE solvent	131.5	1,000,000		7.6046E+03	1.0000E+00	1100	1099.9991
PCE	165.8	1	1.00E-06	6.0314E-03	7.9312E-07	200	0.0002
Other MIs				unknown	unknown		
Stabilizers				unknown	unknown		
Additives				unknown	unknown		
Total				7.6046E+03			

^a Number of grams of impurity per 10⁶ grams of EDC

3. CASE STUDY: 1,2-DCA RELEASE AT A MAJOR CHEMICAL MANUFACTURING FACILITY

The field site for this case study (Lane 2001) is situated in an area of petrochemical production facilities in the city of Sarnia in southwestern Ontario (Figure 2). The study site is located at a major chemical manufacturing facility situated on an extensive area of clayey deposits between 130 and 230 feet thick that exist along the St. Clair River. It is a location where leaks from 1955 to 1972 from an above-ground product line connected to a tank of 1,2-dichloroethane (1,2-DCA), the monomer used to produce vinyl chloride, resulted in the cumulative input of a considerable 1,2-DCA volume to the subsurface (Figure 3).



The 1,2-DCA site was initially considered to be a site where only 1,2-DCA DNAPL had been released. However, analytical results from soil and groundwater samples showed that several compounds are present at this site, including PCE, TCE, cis-1,2-dichloroethylene (c-1,2-DCE), 1,1-dichloroethylene (1,1-DCE), and vinyl chloride (VC). Like 1,2-DCA, the PCE most likely originated as DNAPL from other product line or storage tank leaks in the area, and the TCE, cis-1,2-DCE, 1,1-DCE, and VC appear to be degradation or transformation products of PCE.

Concentrations of c-1,2-DCE and of 1,1-DCE were similar in most groundwater samples, with the concentration of 1,1-DCE exceeding that of c-1,2-DCE in several samples. Because c-1,2-DCE is typically the more predominant isomer, the source of the 1,1-DCE was evaluated. This evaluation included investigation of whether the detected 1,1-DCE was from a MI in 1,2-DCA solvent or a product of degradation.

3.1 Purity and Composition of Manufactured 1,2-DCA

In order to identify the MIs present in 1,2-DCA, a sample was retrieved in 1998 from a tank car containing recently manufactured 1,2-DCA. Table 2 shows the results from the manufacturer's product quality analysis (Lane 2001). Nine other chlorinated compounds were detected in this sample as manufacturing impurities, but 1,1-DCE was not present.

3.2 Use of Manufacturing Impurities to Identify Degradation Mechanisms

The data identifying MIs in the 1,2-DCA sample were used to conclude that the presence of TCE (degradation product and MI), significant amounts of 1,1-DCE, and the absence of 1,1-DCE in the 1,2-DCA solvent indicated that abiotic degradation of TCE may have occurred at this site. 1,1-DCE was not manufactured or used by the site owner; therefore, it was not disposed at this site, and it is not known to occur as an impurity in any of the DNAPLs that were disposed at this site (Prine 1999, Creber 2000). Studies (Klecka et al. 1990, Kastner 1991) indicated that 1,1-DCE in groundwater can occur as a product from the abiotic degradation of TCE.

Kriegman-King and Reinhard (1992) observed that abiotic degradation of another chlorinated solvent (carbon tetrachloride) was enhanced by the presence of iron sulfide (pyrite) and iron vermiculite. The clays at this site contain both pyrite and iron vermiculite (Abbott 1987, Quigley and Ogunbadejo 1976). TCE occurs in nearly all core samples where 1,1-DCE was found, and the mole fraction of 1,1-DCE was observed to increase as the mole fraction of TCE decreases, which is consistent with 1,1-DCE production from TCE. Therefore, it is likely that abiotic degradation of TCE produced the 1,1-DCE.

Table 2. Manufacturer's Analysis of 1,2-DCA Solvent: 1998 Sample

<i>Compound</i>	<i>MW</i> (g/mol)	<i>Quantity</i> (ppm w/w) ^a	<i>Fraction</i> <i>by Mass</i>	<i>Moles</i>	<i>Mole</i> <i>Fraction</i>	<i>Solubility</i> (mg/L)	<i>Solubility_{eff}</i> (mg/L)
EDC Solvent	99	1,000,000		1.0101E+04	9.9955E-01	8,500	8,496
1,1,2-TCA	133.4	210.9	2.11E- 04	1.5810E+00	1.5645E-04	4,400	0.6884
cis-1,2-DCE	97	180.1	1.80E- 04	1.8567E+00	1.8373E-04	3,500	0.6431
TCE	131.5	19.8	1.98E- 05	1.5057E-01	1.4900E-05	1,100	0.0164
PCE	165.8	9.9	9.90E- 06	5.9710E-02	5.9087E-06	200	0.0012
1,1-DCA	99	9.7	9.70E- 06	9.7980E-02	9.6957E-06	8,100	0.0785
H ₂ O	18	5.3	5.30E- 06	2.9444E-01	2.9137E-05		
NVM ^b	--	5	5.00E- 06	unknown	unknown		
HCL	13	1.73	1.73E- 06	1.3308E-01	1.3169E-05	miscible	miscible
CA	64.51	1	1.00E- 06	1.5501E-02	1.5340E-06	6,040	0.0093
trans-1,2- DCE	97	0.8	8.00E- 07	8.2474E-03	8.1613E-07	6,300	0.0051
VC	62.5	0.8	8.00E- 07	1.2800E-02	1.2666E-06	3,000	0.0038
1,1,2,2- TECA	167.9	0.8	8.00E- 07	4.7647E-03	4.7150E-07	2,900	0.0014
Iron	55.847	0.064	6.40E- 08	1.1460E-03	1.1340E-07		
Total				1.0106E+04	1.0000E+00		

^a Number of grams of impurity per 10⁶ grams of EDC

^b Non-volatile materials

3.3 PCEMI in Manufactured 1,2-DCA

The original use of the 1,2-DCA MI data was to evaluate degradation mechanisms at the site. This data also may be used to evaluate the potential impact to groundwater by PCEMI. PCEMI comprises, by mass, about 0.001% of this particular sample of 1,2-DCA.

3.1.1 Solubility of PCEMI

The solubility of PCE in water has been reported at 150 mg/L (CRC 73rd Ed) and 200 mg/L (Pankow and Cherry 1996). This solubility is valid for PCE present as a single component. When PCE is present in a mixture, its solubility must be calculated with respect to the other components of the mixture, based on Raoult's Law (Feenstra et al. 1991). The solubility of a component in a mixture is calculated by the following formula, where $C_{sat,m}$ is the aqueous solubility of a component in a mixture, X_m is the mole fraction of the component, and C_{sat} is the aqueous solubility of the compound as a single component:

$$C_{sat,m} = X_m C_{sat}^o$$

$C_{sat,m}$ is known as the "effective solubility" of a component in a mixture (Pankow and Cherry 1996).

Table 1 shows the calculated effective solubility of PCEMI in manufactured TCE, when the higher solubility is considered. For the past 10 to 15 years, the maximum effective solubility of PCEMI in manufactured TCE has been 0.0002 mg/L (0.2 µg/L), based on PCEMI present in an amount equal to the detection limit of 1 ppm w/w. Prior to that time, the effective solubility of PCEMI would have been, sporadically, as high as 0.002 mg/L (2.0 µg/L). Table 2 shows that the calculated effective solubility for PCEMI in the 1998 sample of 1,2-DCA solvent is 0.0012 mg/L (1.2 µg/L). These effective solubilities represent the calculated maximum possible groundwater concentrations of PCEMI from manufactured TCE and 1,2-DCA, because attenuating mechanisms, such as sorption, dilution, and degradation, will reduce the concentration in groundwater.

An aliquot of a different sample of 1,2-DCA, produced by the same manufacturer at about the same time as the sample analyzed in 1998, was dissolved in deionized water to determine, under laboratory conditions, aqueous concentrations of the chlorinated components present in the manufactured 1,2-DCA. The results from this analysis are tabulated in Table 3. These results show the presence of four MIs — cis-1,2-dichloroethylene (c-1,2-DCE), PCE, TCE, and 1,1,2-trichloroethane (1,1,2-TCA).

<i>Compound</i>	<i>Concentration</i> (mg/L)	<i>Fraction</i> <i>of EDC</i>
EDC	8,562	
cis-1,2-DCE	0.0641	7.4866E-06
PCE	0.0065	7.5917E-07
TCE	0.0061	7.1245E-07
1,1,2-TCA	Not Quantified	

The aqueous concentration of PCEMI generated under laboratory conditions was 0.0065 mg/L (6.5 ppb), which is about 5.5 times the calculated effective solubility. This high concentration is likely inaccurate due to the formation of an emulsion of NAPL in the water

during mixing of the sample (Feenstra et al. 1991, Billington et al. 1988). It is unlikely that this high concentration is a result of differences in 1,2-DCA batch production. PCEMI would have to be present in the manufactured 1,2-DCA at 50 ppm to generate a concentration of 6.5 ppb in water, which far exceeds the amount of PCEMI typically generated during solvent manufacturing.

4. CONCLUSIONS

The calculated maximum groundwater concentrations of PCEMI that might be generated from its presence in manufactured TCE or 1,2-DCA, are likely no more than 0.2 to 2.0 $\mu\text{g/L}$ for PCEMI in TCE and 1.2 $\mu\text{g/L}$ for PCEMI in 1,2-DCA. These concentrations, which are overestimated, are below or near the method detection limit (MDL) for some analytical methods (Table 4).

<i>Table 4. Detection Limits for PCE</i>		
Method	Method Detection Limit $\mu\text{g/L}$	
5032	1.4	Vacuum Distillation – Internal Standard
5032	1.8	Vacuum Distillation – External Standard
8021B	0.05	PID (photoionization detector)
8021B	0.04	HECD (electrolytic conductivity detector)
8260B	0.14	Wide-Bore Capillary Column
8260B	0.05	Narrow-Bore Capillary Column

Estimating groundwater concentrations generated by MIs from effective solubility calculations results in overestimation of the groundwater concentration of the MI, because processes such as sorption, dilution, and degradation reduce groundwater concentrations. Another important process in the generation of groundwater concentrations of PCEMI is the rate of dissolution for PCE and the chlorinated solvent. When these processes are considered, it is likely that the groundwater concentrations generated by PCEMI are much less than its effective solubility and are below the detection limit of commonly used analytical methods. This is supported by the evaluation of the vapor degreaser conceptual model based on reported field concentrations. The results of this evaluation indicate that the groundwater concentration that could realistically result is 0.01 $\mu\text{g/L}$, which is below all of the analytical methods listed in Table 4. PCEMI likely does not significantly contribute to groundwater contamination, as PCEMI is either not detectable or groundwater concentrations generated are likely below the maximum contaminant level of 5 $\mu\text{g/L}$.

This work represents an initial step toward greater understanding of the potential impact to groundwater from MIs and, as a result, better source identification. Greater participation from chemical manufacturing partners in research related to the issues presented here is necessary to better quantify the MIs in manufactured solvents, particularly over the history of their manufacture. Laboratory and field studies designed to improve our understanding of solubility issues related to manufactured solvents and site specific effective solubilities are also necessary

to understand the environmental impact from chlorinated solvents and MIs. This is important, because even minor concentrations of PCE may indicate sources of PCE contamination other than MIs. At sites where chlorinated solvent releases have occurred, better source identification and quantification of the resulting environmental impact are required for thorough site characterization, selection of effective remediation technologies, reliable forensic investigations, fair cost allocation, and presentation of sound, justifiable technical opinions for litigation support.

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