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DETERMINATION AND QUANTIFICATION OF NON-AQUEOUS PHASE LIQUID MIXTURES IN ENVIRONMENTAL MEDIA

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1.0 ABSTRACT:

It is important to recognize the presence of Non-Aqueous Phase Liquids (NAPLs) in soils at a waste site in order to design and construct a successful remediation system. NAPLs often manifest as a complex, multi-component mixture of organic compounds that can occur in environmental media, such as vadose zone soil, where the mixture will partition and equilibrate with soil particles, pore vapor, and pore water. Complex organic mixtures can greatly complicate the determination and quantification of NAPL in soil due to inter-media transfer. NAPL thresholds can also change because of mixture physical properties and can disguise the presence of NAPL. A unique analytical method and copyrighted software have been developed at the Department of Energy's Savannah River Site that facilitates solution of this problem. The analytical method uses a classic chemistry approach and applies the principals of solubility limit theory, Raoult's Law, and equilibrium chemistry to derive an accurate estimation of NAPL presence and quantity. The method is unique because it calculates an exact result that is mass balanced for each physical state, chemical mixture component, and mixture characteristics. The method is also unique because the solution can be calculated on both a wet weight and dry weight basis – a factor which is often overlooked. The software includes physical parameters for 300 chemicals in a database that self-loads into the model to save time. The method accommodates up to 20 different chemicals in a multi-component mixture analysis. A robust data display is generated including important parameters of the components and mixture including: NAPL thresholds for individual chemical components within the mixture, mass distribution in soil for each physical state, molar fractions, density, vapor pressure, solubility, mass balance, media concentrations, residual saturation, and modest graphing capabilities. This method and software are power tools to simplify otherwise tedious calculations and eliminate guesswork for site characterizations.

2.0 INTRODUCTION

Non-Aqueous Phase Liquid or “NAPL” is a term that most environmental professionals are familiar with because NAPL is well recognized as a significant source of soil and groundwater contamination. According to the US Environmental Protection Agency 2004, “Since NAPLs are only slightly soluble in water, NAPL source zones can persist for many decades and in some cases for the foreseeable future.”

NAPLs can manifest as either single component liquids or as complex, multi-component mixtures of organic compounds that are slowly released from environmental media, such as vadose zone soil, where the contaminants will partition into soil pore vapor, soil pore water, and soil particles and eventually migrate to groundwater (Figure 1). The identification of NAPL in the environment is complicated because of inter-media transfer within the soils and because of the altered physical and chemical properties of complex organic mixtures. Complex multi-component mixtures distributed in environmental medium can mask their appearance and complicate their identification.

Soil analytical results can be compared to a simply calculated NAPL threshold value to determine if a single component NAPL maybe present in a soil sample. Conversely, determination of a multi-component NAPL mixture in soil media from analytical soil data is a different matter and is complicated by the altered physical and chemical properties of the organic compounds that are present. One of the most important chemical properties used to determine the presence of a NAPL in environmental media is the solubility limit, although other major properties are also important including vapor pressure and density. For multi-component mixtures, it is important to comprehend that the chemical properties of the mixture will not be exactly the same as any of the pure phase components. This fact makes the identification of the NAPL more convoluted and also means the NAPL thresholds will be much lower than may be assumed.

The above statements suggest a possibility that NAPL may be more frequently encountered at contaminated waste sites but its presence may have be accidentally overlooked. This reason may partially explain why some conventional remedial technologies have failed to effectively clean up contaminated source zones and groundwater plumes. Too many groundwater clean ups have dragged on for decades and can be traced to previously unrecognized NAPL source zones. Remedial measures that have been implemented where the presence of NAPL has been unrecognized, have resulted in longer than expected clean-up times and rebounding effects after the remedies have been implemented because residual and persistent NAPL residuals remain trapped in the soil where it acts as a continuing source of contamination to the groundwater.

The dynamic alteration of physical properties of chemical mixtures may disguise the presence of NAPL in environmental media during site characterization and remediation, but this doesn't need to be the case because there is a solution. The method and software described in this manuscript are simple yet powerful tools to arrive at a solution for determination and quantification of NAPL in environmental media. The analytical method uses a classic chemistry approach and applies the principals of solubility limit theory, Raoult's Law, and equilibrium chemistry to derive an accurate estimation of NAPL quantity.



Figure 1: Multi-component, dense NAPL recovered from a ground water monitoring well at the Savannah River Site, Aiken, SC. This sample consists of PCE (94%), polychlorinated biphenol (3%), and TCE (3%) in order of molar fraction composition.

3.0 GOVERNING ASSUMPTIONS AND EQUATIONS

Because of the number of equations, logic functions, and numerical iterations included in the model, it is beyond the scope of this manuscript to present all the details and keep within the publication guidelines. Therefore, only a selected number of key governing assumptions and equations will be discussed to elucidate the conceptual framework of this analytical method.

Assumptions

1. The NAPL mixture will behave as an ideal solution. Ideal solutions will obey Raoult's Law.
2. All mass and volume is conserved. There is no loss of mass or volume due to volatilization, degradation, evaporation, photolysis, etc. or other physical-chemical processes. At the end of the calculation, the final mass balance and volume will equal the initial mass and volume.

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3. Contaminants are uniformly distributed throughout the contaminated media.
4. Equilibrium partitioning is instantaneous and linear for vapor, aqueous, and solid phases.
5. All NAPL components or NAPL mixtures are liquids at ambient temperatures.
6. Environmental media is composed of three phases: 1.) solid phase as soil particles, 2.) vapor phase as soil pore-air, and 3.) aqueous phase as soil pore-water.
7. Mass is composed of four states: 1.) solid, 2.) vapor, 3.) aqueous, and 4.) NAPL, if any.
8. Water saturated media has no air-filled porosity.
9. Soil temperature is assumed to be 20degrees C.

Solubility Limit Theory for Single Component NAPL and NAPL Mixtures

The simplest method to determine if a NAPL exists in a water sample would be to compare the concentration of an organic chemical mixed in water to its aqueous solubility limit. If the dissolved chemical concentration in water exceeds the solubility limit, then NAPL would be present in the sample. NAPL exists for a single component NAPL when:

$$(1a) \quad C_w > S, \text{ or}$$
$$(1b) \quad \text{for a multi-component NAPL mixture when } C_{wi} > S_i^E;$$

where:

-
- S is aqueous solubility limit of the organic component i;
- C_{wi} is the water concentration of component i; and
- S_i^E is the effective solubility limit of the organic component i,.

Determination and quantification of a multi-component NAPL mixture in soil is a much more challenging proposition than presented in the example above. The method to determine and quantify a NAPL mixture in soil requires the application of solubility limit theory, Raoult's Law, equilibrium partitioning theory, mass balancing of all the chemical components including the NAPL portion, and volumetric balancing of all the phases within the sample.

Raoult's Law

Raoult's Law must be applied to calculate the "effective solubility" for each organic compound within the mixture to establish the starting point for the equilibrium calculations. Raoult's Law states that the effective solubility of a compound is equal to its molar fraction in the solution times its pure-phase solubility (Moeller, 1980). From Feenstra, et al, 1991, the effective solubility (S_i^E) of each component of a mixture can be expressed mathematically as:

$$(2) \quad S_i^E = X_i \cdot S_i \text{ [mg/L]}$$

where:

- X_i = Molar fraction of component i in a mixture [fraction, unitless]
- S_i = Pure phase solubility limit of the component i [mg/L]

The molar fraction is the percentage of each compound within the mixture, calculated using the molecular weight of each of the components present in the mixture. The sum of all the component (i) solubility's ($\sum S_i^E$) in an equilibrated mixture is equal to the solubility of the mixture and the sum of all the molar fractions ($\sum X_i$) is equal to 1. Like the solubility, density and vapor pressure for a mixture may be similarly calculated using molar fractions.

Equilibrium Partitioning Theory

Equilibrium partitioning calculations allow for the phase distribution and approximation of the concentration for each of the chemical components within the sample. Equilibrium partitioning theory in the model assumes that an organic compound will partition into the environmental media according to a water-soil (soil-water partitioning coefficient) and a water-vapor (Henry's Law constant) distribution coefficient. Equilibrium calculations begin by determining the initial soil pore-water concentration. The initial pore-water concentration can be one of three possibilities: 1) the aqueous solubility limit (S) for a single component, 2) the effective solubility limit (S^E) for each component in a multi-component mixture, or 3) a potential pore-water concentration (ICw_i) from Feenstra, et al, 1991, if NAPL is not associated with that component in the mixture. ICw_i is illustrated below.

$$(3) \quad ICw_i = \left(\frac{Ct_i}{\rho_b} \right) \cdot (K_d \cdot \rho_b + \theta_w + H' \cdot \theta_a) \text{ [mg/L]}$$

where:

- Ct_i = Total soil concentration for component i from soil sample [mg/kg]
- ρ_b = Bulk density [kg/L]
- K_d = Soil-water partitioning coefficient [L/kg]
- θ_w = Water filled soil porosity [fraction]
- H' = Henry's Law Constant [dimensionless]
- θ_a = Air-filled porosity [fraction]

The correct initial pore-water concentration is selected by logic arguments built into the model. Once the initial pore-water concentration has been solved, the other remaining partitioning calculations can be performed to estimate vapor phase, sorbed phase, and NAPL concentration and mass in sequence.

The Soil Saturation Limit (C_{sat}) (USEPA 1996 and others) represents the upper boundary saturation limits in soil media based on the chemical characteristics of the compound(s) present. C_{sat} is defined as the contaminant concentration in soil at which pore-air and pore-water are saturated with the chemical(s) and the sorptive capacity of the soil particles have been reached (USEPA 1996). C_{sati} is calculated for each component of a multi-component mixture. Above the C_{sat} threshold a contaminant may be present as a NAPL in soil. The C_{sat} threshold is an important type of equilibrium and mass calculation that will be used later in the calculations.

$$(4) \quad C_{sat\ i} = \left(\frac{S_i^{E_i}}{r_b} \right) \cdot (K_d \cdot r_b + \Theta_w + H' \cdot \Theta_a) \text{ [mg/kg]}$$

A form of the Freundlich isotherm (USEPA 1996) is used as a linear isotherm to calculate C_{si} , the sorbed soil concentration, for each component in a mixture as in equation 5. The dimensionless form of Henry's Law Constant is used to describe the partitioning of a compound from the liquid phase to the vapor phase concentration for each component, C_{ai} , using equation 6. Use of the equilibrium calculations allows media concentrations to be partitioned accordingly to the different phases, which in turn allows calculation of the mass balance.

$$(5) \quad C_{si} = K_d \cdot C_w \text{ [mg/kg]}$$

$$(6) \quad C_{ai} = H' \cdot C_w \text{ [mg/L]}$$

where:

- H = Henry's Law Constant [dimensionless]

Mass Balance and Volume Conservation

Because of the assumption of conservation of mass, the partitioning of a compound into each of three media phases plus the NAPL, must balance with the total mass of each component M_{ti} and the total contaminant mass in the sample Ct . This is expressed by the following equation:

$$(7) \quad M_{ti} = M_{ai} + M_{wi} + M_{si} + M_{ni} \text{ [mg/kg]}$$

$$(8) \quad Ct = \sum M_{ti} \text{ [mg/kg]}$$

where:

- M_{ai} = Mass of component i in soil-air, [mg/kg]
- M_{wi} = Mass of component i dissolved in soil pore-water, [mg/kg]
- M_{si} = Mass of component i sorbed to soil particles, [mg/kg]
- M_{ni} = Mass of free product or NAPL, [mg/kg]

C_{sat} may also be expressed as:

$$(9) \quad C_{sat} = M_a + M_w + M_s \text{ [mg/kg]}$$

M_a is deleted from the mass balance equation for water saturated samples because of the assumption there is no air-filled porosity in the saturated zone. In this case, the software will solve for three phases (pore-water, sorbed, and NAPL) instead of four phases (pore-water, sorbed, vapor and NAPL).

To determine the contaminant volume distribution in a sample, the compounds are partitioned into the vapor, aqueous, solid and NAPL phases of the sample and the appropriate volumes are determined according to equation 10, from Mariner, et al, 1997. The volume balance V_t equation is:

$$(10) \quad V_t = V_a + V_w + V_s + V_n \text{ [L/L]}$$

where:

- V_a = Volume of soil-pore air or Θ_a , [L/L]
- V_w = Volume of soil-pore water or Θ_w , [L/L]
- V_s = Volume of soil particles or $1-(\Theta_a+\Theta_w)$, [L/L]
- V_n = NAPL volume [L/L]

The soil-pore air and pore-water volumes are determined from user input values or estimates. The total porosity n_t used in the volume calculations is composed of:

$$(11) \quad n_t = \Theta_a + \Theta_w \text{ [fraction]}$$

In the model the wet bulk density ρ_b is calculated according to the equation prescribed by Mariner, et al 1997.

$$(12) \quad \rho_b = (W_v * d_w) + (A_v * d_a) + (S_v * d_s) + (V_n * \rho_{napl}) \text{ [kg/L]}$$

where:

- W_v = Soil pore-water volume [fraction, L/L]
- d_w = Water density [kg/L]
- A_v = Soil pore-air volume [fraction, L/L]
- d_a = Air density [kg/L]
- S_v = Soil volume [fraction, L/L]
- d_s = Soil Density [fraction, L/L]
- V_n = NAPL volume [fraction, L/L]
- ρ_{napl} = NAPL density [fraction, L/L]

In situ NAPL volume and saturation in the model are calculated using the wet bulk density because the model is primarily concerned with estimating representative values of NAPL present in the subsurface, but the dry bulk density can also be used for comparison purposes. Wet or dry density inputs are uniquely calculated by the model and can be selected by the user to load either the dry or wet weight component concentrations as site specific conditions indicate.

For unsaturated zone samples, there will be little difference (+/-1%) in calculated NAPL mass if using either the adjusted wet or dry weight basis. However, for some matrices, the model will compensate for extremes caused by analytical concentrations based

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on dry weight only. It is important with some matrices to distinguish between the dry versus wet bulk density basis for several reasons. Analytical laboratories conventionally report soil analytical results on a dry weight basis. As most regulatory soil limits are based on the dry weight basis (including C_{sat}), it is essential when making direct comparisons (of the soil analytical results to the modeled parameters) to be equal and consistent. Additionally, site-specific matrices may impose a unique set of conditions that would favor the selection of a particular density over the other. For example, lake-bottom sediments maybe characterized by low solids content, high water content, and high porosity. However, using analytical soil data reported on a dry weight basis might significantly overstate the total mass of contaminants present in this type of system. In this case, use of the wet bulk density basis may be the best choice to accurately represent the mass of contaminants.

NAPL Saturation

Residual saturation is a calculated value essential to estimate the magnitude of NAPL contamination in subsurface soils and is applied to determine the volume of contaminated media at a waste site. The residual saturation can also be used as a contaminant baseline against which to measure remediation system clean up performance. Brost et al, 2000, has reported that residual saturation may also be used to evaluate NAPL mobility in subsurface soils. Because the volume of a sample is composed of solids (soil particles) and voids (porosity), the NAPL must physically occupy the void space within the sample. Therefore, residual saturation is defined as the fraction of pore space filled by NAPL.

The mathematical expressions used to determine the mass of NAPL for component i ($NAPL_i$) and the total mass of NAPL within the sample ($NAPL_t$) is:

$$(13) \quad NAPL_i = M_{ii} - C_{sati} \text{ [mg/kg]}$$

$$(14) \quad NAPL_t = \sum NAPL_i \text{ [mg/kg] where:}$$

- $NAPL_i$ = Mass of NAPL of component i [mg/kg]
- M_{ii} = Total mass of component i in the soil sample [mg/kg]
- C_{sati} = Soil Saturation Limit of component i [mg/kg]
- $NAPL_t$ = Total NAPL mass in the soil sample [mg/kg]

If the total mass (M_{ii}) exceeds the Soil Saturation Limit (C_{sati}), then NAPL will be present in the sample for component i . Because C_{sat} is the theoretical upper-bound saturation limit for partitioned contaminants, any contaminant mass greater than C_{sat} must, by definition, be residual (NAPL) saturation. The residual saturation is defined as:

$$(15) \quad S_r = \frac{V_n}{n_t} \text{ [% , fraction]}$$

where:

- S_r = Residual NAPL saturation in soil [% , L/L]
- n_t = Total porosity [% ,L/L]

The NAPL volumetric content N_v is:

$$(15) \quad V_n = \left(\frac{NAPL_t \cdot r_b}{r_{napl}} \right) [\%, L/L]$$

- $NAPL_t = \sum_{NAPLi} [mg/kg]$
- ρ_{napl} = Density of the NAPL mixture [mg/L]
- ρ_b = Wet bulk density [kg/L]

4.0 DISCUSSION OF HYPOTHETICAL MODELING

It is useful to demonstrate by means of an example the type of data and information the model will generate. The model automatically loads chemical specific parameters, Table 1, when the chemical of interest is selected from the menu. The model requires a minimum number of geotechnical data inputs which are provided in Table 2.

Table 1. Chemical Parameters Automatically Loaded by the Model

Component	Molecular Weight (g/mol)	Solubility (mg/L)	Vapor Pressure (mm/Hg)	Henry's Law Constant (unitless)	Organic Carbon Coefficient (L/kg)	Density (kg/L)
Trichloroethylene	131.0	1100.0	74.3	0.365	126.0	1.46
Tetrachloroethylene	166.0	200.0	18.5	0.943	364	1.63
PCB-1260	372.0	0.0144	1.1E-05	0.0103	6.7E+06	1.57
1,3,5-Trinitrobenzene	213.0	385.0	3.03E-06	9.06E-08	20.0	1.50
2,4,6-Trichlorophenol	197.0	434.0	0.012	1.96E-04	2000.0	1.50
Carbon Tetrachloride	154.0	758.0	110.0	0.943	439.0	1.59

Table 2. Geotechnical Parameters Loaded by the User.

Component	Analytical Dry Soil Concentration (mg/kg)	Total Porosity (fraction)	Soil Water Content (fraction)	Soil Air Content (fraction)	Fraction Organic Carbon (fraction)
Trichloroethylene	8,500.0	0.40	0.30	0.10	0.1
Tetrachloroethylene	8,000.0	0.40	0.30	0.10	0.1
PCB-1260	3000.0	0.40	0.30	0.10	0.1
1,3,5-Trinitrobenzene	11,000.0	0.40	0.30	0.10	0.1
2,4,6-Trichlorophenol	12,000.0	0.40	0.30	0.10	0.1
Carbon Tetrachloride	9,800.0	0.40	0.30	0.10	0.1

Table 5. Summary of NAPL Saturation and NAPL Characteristics

TOTAL NAPL SATURATION			NAPL Mixture Properties	
d=	1.53E+00	kg/L	Density of NAPL Mixture	5.90E+02
NAPL _t =	2.8482E+04	mg/kg	NAPL Mass	4.27E+01
F _{napl} =	1.53E+06	mg/L	NAPL Density	176.05
S _r =	9.03E-02	fraction	NAPL Saturation	
%S _r	9.0340%	%	Residual NAPL Saturation*	
			*(% Total porosity occupied by NAPL)	
VOLUME				
N _v =	3.6136E-02	L/L	NAPL Volume	
A _v =	6.3864E-02	L/L	Air Volume (V _a)	
W _v =	3.0000E-01	L/L	Water Volume (V _w)	
S _v =	6.0000E-01	L/L	Soil Volume	
V _t =	4.0000E-01	L/L	Total Porosity (n _t)	
P _b =	1.9455E+00	kg/L	Wet Bulk Density	
P _d =	1.5900E+00	kg/L	Dry Bulk Density	

GRAPHS	
Graph Mixture Composition	Graph Volumetric Distribution
Graph Effective Solubility	Graph Mass Distribution
Graph Effective VP	

NAPL in SAMPLE	YES
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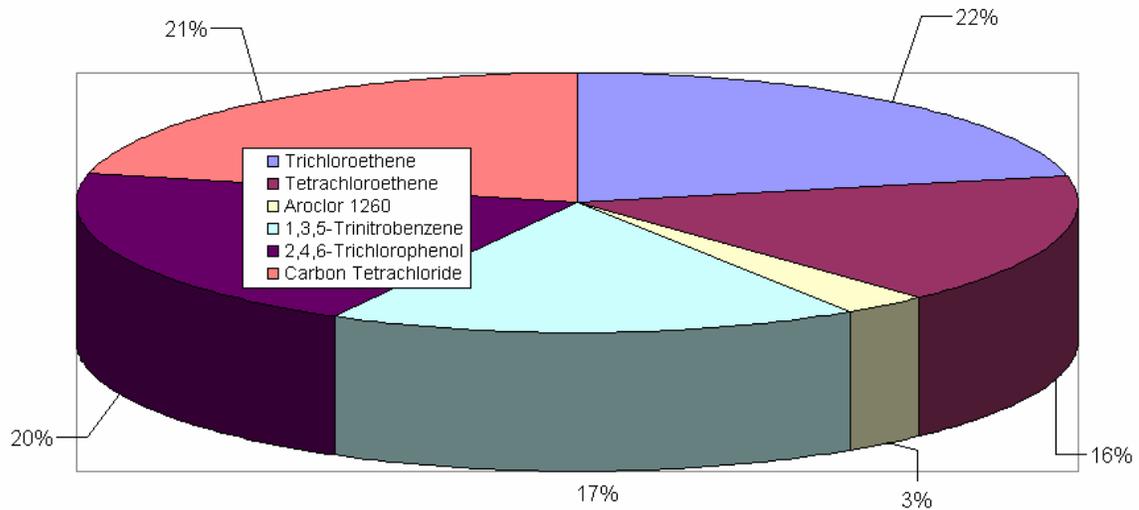


Figure 2. Molar Fractions in Mixture

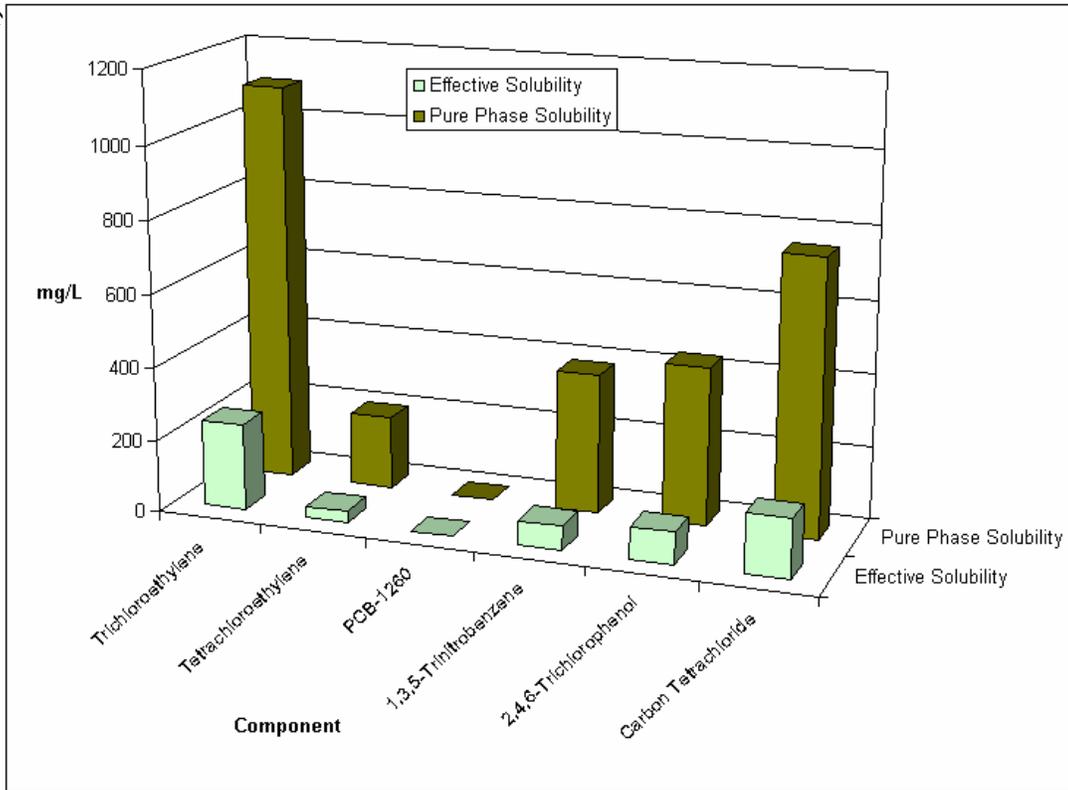


Figure 3. Comparison of Pure Phase Aqueous Solubility to Mixture Solubility

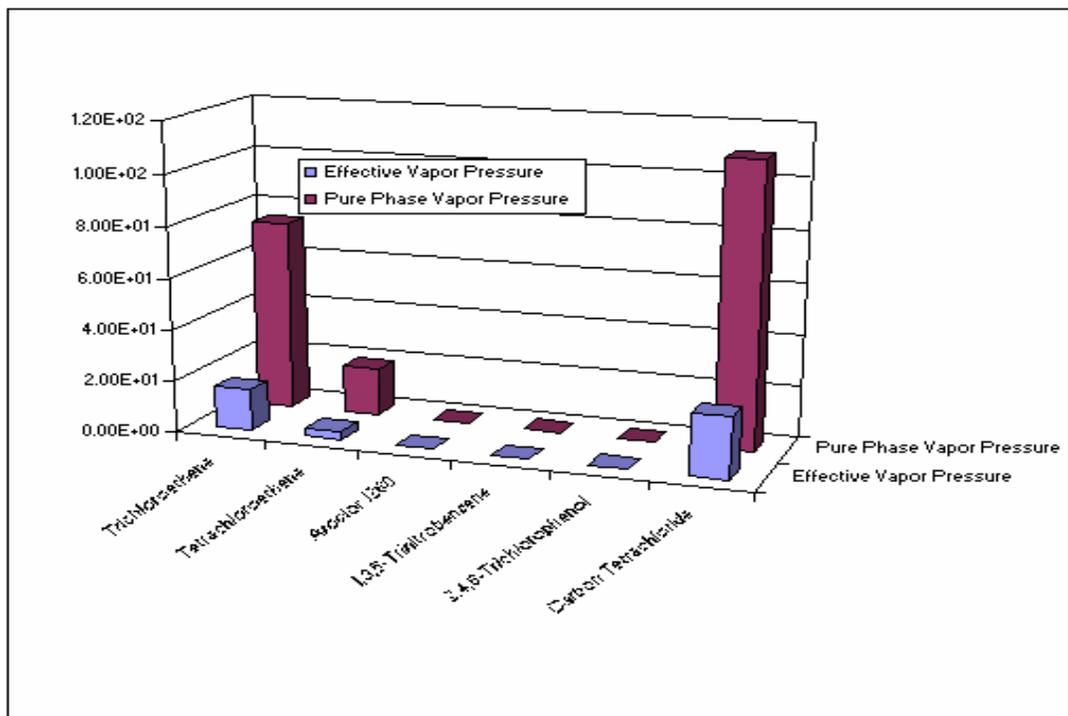


Figure 4. Comparison of Pure Phase Vapor Pressure vs. Vapor Pressure in Mixture.

Table 3 lists the soil saturation limits (C_{sat}) for each component in the sample on both a wet and dry weight basis. C_{sat} is an important threshold since any component concentration that exceeds C_{sat} may be considered a NAPL. Table 4 presents a summary of the mass balances for each component within the mixture. The distribution of mass residing in each phase is presented in Table 4 also, as determined by the equilibrium calculations. Interestingly, the component with the highest mass (2, 4, 6-trichlorophenol) in the soil sample does not contribute to the NAPL mass in the sample due to its relatively high K_{oc} value with over 99% of the mass sorbed to soil particles.

In Table 5, the NAPL volume is mathematically related to the 28,482 mg/kg NAPL mass and void space within the media matrix. Table 5 displays a residual saturation of 9.0% which is considered a relatively high level of residual NAPL. It can also be determined from Table 5 that 3.5% of the volume of each liter of representative soil contains NAPL.

Figure 3 illustrates the NAPL mixture molar fraction composition according to Raoult's Law. On a mass basis, 2, 4, 6-trichlorophenol would represent 23% of the total contaminant mass but on a molar fraction basis represents 20% of the mixture. All the components are relatively equally represented in the mixture except for Arochlor (PCB)-1260 which represents 3% of the mixture.

Figure 4 demonstrates the impact of Raoult's Law on a mixtures' solubility. In the graph, it will be noted that the solubility limit for every component within the mixture has been significantly depressed when compared to its respective pure phase aqueous solubility limit. This example also helps to explain the reason why a NAPL mixture can be persistent in environmental media for decades due to low solubility. The depressed solubility of the mixture means low dissolution rates in soil pore-water and media contaminated with the above mixture could serve as a continuing source of groundwater contamination for many decades. Low solubility mixtures in the source zone will not respond well using conventional groundwater or soil remediation technologies such as groundwater pump and treat, engineered cover systems, or soil vacuum extraction systems and it is best to recognize this fact as early in the characterization as possible.

Figure 5, illustrates the vapor pressure for each component within the mixture will be also be depressed. The implication of a low vapor pressure mixture means that volatilization rates will also be low. Particularly, some components within the mixture could have vapor pressures too low ($>0.5\text{mmHg}$) to be suitable for remediation with a soil vacuum extraction system. In the case of carbon tetrachloride, the mixture vapor pressure is 79% lower than its pure phase vapor pressure which is a significant reduction in volatility and needs to be realized before remedial engineering design by remedial engineers.

5.0 CONCLUSIONS

The model has demonstrated that it can handily determine and quantify NAPL mixtures in environmental media. The software includes physical parameters for 300 chemicals in a database that self-loads into the model to save time. The method accommodates up to 20 different chemicals in either a single component or multi-component mixture analysis. The software will evaluate either fully water saturated soils or unsaturated vadose zone soils. The model will also uniquely calculate chemical concentrations on either a wet or dry weight basis to reflect realistic site specific conditions.

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The method used by the software is extremely powerful to determine and quantify if NAPL is present in environmental media using commonly available analytical soil or groundwater results. It is extremely important to use the model early in the site characterization to interpret and assess the extent of impacted soils and groundwater at a waste site. The information provided by the model can guide additional phases of characterization and the output is of particular relevance for remedial design engineers.

The information generated by the model is plentiful and robust and provides essential information for site characterization personnel to assess the magnitude and volume of contaminated media as well as data for engineers to design effective remedial systems. The results of the model provide an early indication of the extent and magnitude of subsurface contamination which is necessary to develop an appropriate remedial response for NAPL contaminated environmental media such as electrical resistance heating, dynamic underground stripping, soil flushing, or excavation.

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7.0 BIOGRAPHICAL SKETCH

Gregory Rucker is employed as a Senior Advisory Engineer in Soil and Groundwater Projects by the Washington Savannah River Company in Aiken, South Carolina. He is the

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