

Ground Water Sampling at ISCO Sites – Oxidant Residuals and Sample Preservation Guidelines

**National Association of
Remedial Project Managers
Kansas City, KS**

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EPA Region 7 (Kansas City, KS)



EPA



“Binary Mixtures”

- Ground water samples collected specifically to be analyzed for organic contaminants; may contain a mixture of both the contaminant and the oxidant.
- Issue raised many times in the past, including
 - Savage Well, Olympia (Wells G&H) (Region 1)
 - Vieques PR (Region 2)
 - Fike/Artel, Berks Sand Pit (Region 3)
 - Southern Solvents (Region 4)
 - Parkview (Region 7)
- Preliminary review
 - Definitive published information - Limited
 - ISCO, Analytical



Overview

- **Why / why not should binary mixtures be analyzed**
- **Binary mixtures**
 - How does this condition occur
 - Potential impact on ground water quality
 - Potential impact on analytical instrument
- **Preservation techniques**
 - Potential impact on ground water quality
 - Potential impact on analytical instrument
- **Field test kits and methods**
- **Preliminary guidelines for preservation**



Why collect and analyze a ground water sample that contains oxidant?

- Preliminary assessment of ISCO performance
- Assess whether re-distribution of the contaminant plume may have resulted from ISCO activities
- Interim ISCO pilot-studies are implemented to establish design parameters for full-scale ISCO deployment (accelerated schedules)
- Regulatory-driven goals and specified timelines
- Long term permanganate persistence (closure-driven)
- Some decisions can be made (subsequent mobilization)
- Other reasons, ...



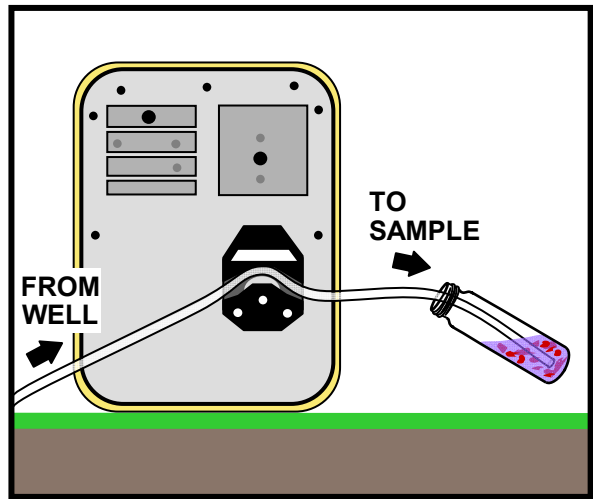
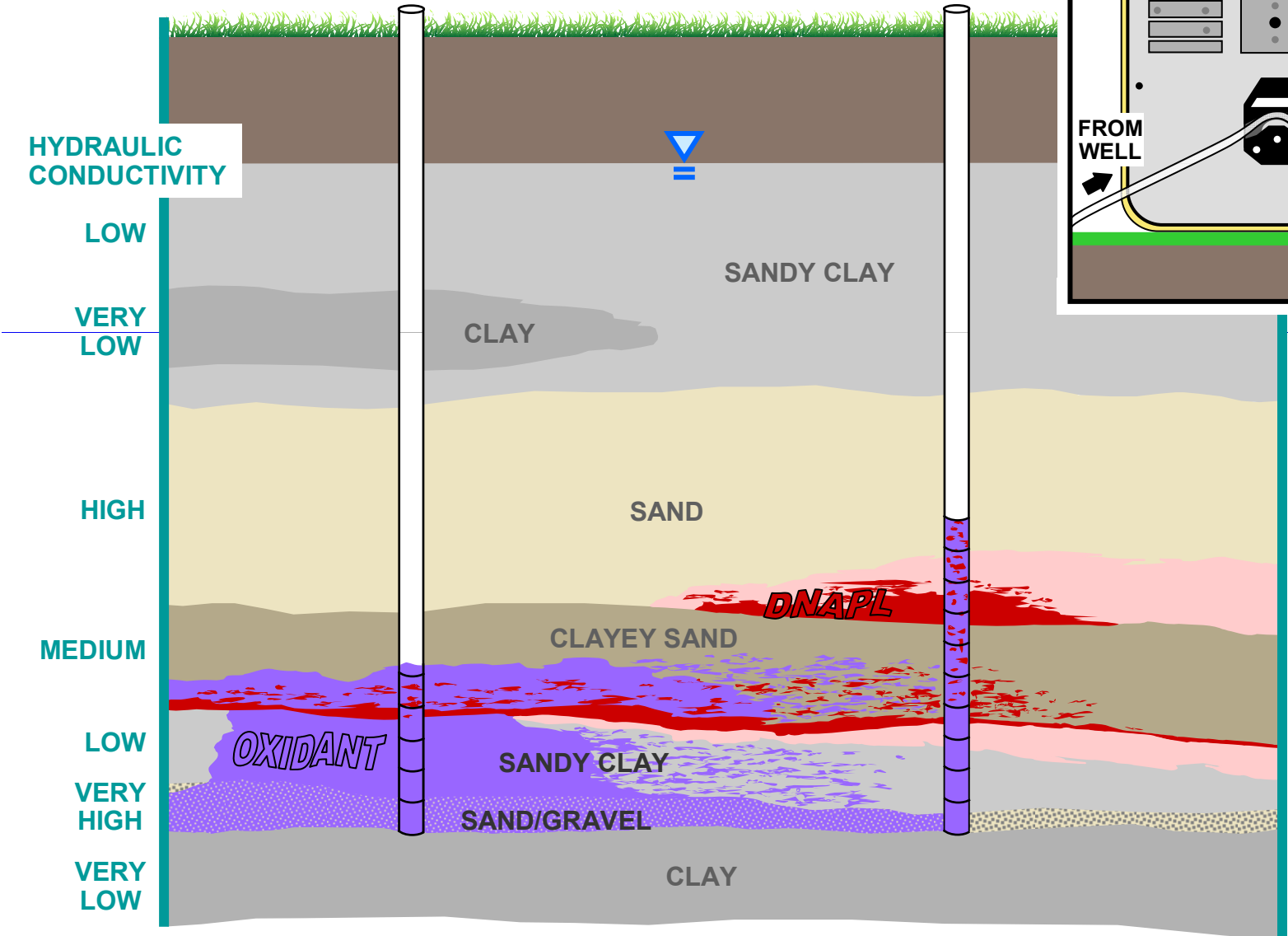
Why not collect and analyze binary mixture ground water samples?

- Oxidation of contaminants in sample
- Ground water sample not representative of subsurface conditions (non-equilibrium)
- Some preservatives used to neutralize oxidant
 - May impact quality of ground water
- Dilution requirements to lower oxidant concentration reduces detection limit to unacceptable range



**Oxidant Injection Well,
Well Point, or Other Oxidant
Injection Method**

**Monitoring
Well**



**Oxidant Injection Well,
Well Point, or Other Oxidant
Injection Method**

**Monitoring
Well**

**HYDRAULIC
CONDUCTIVITY**

LOW

**VERY
LOW**

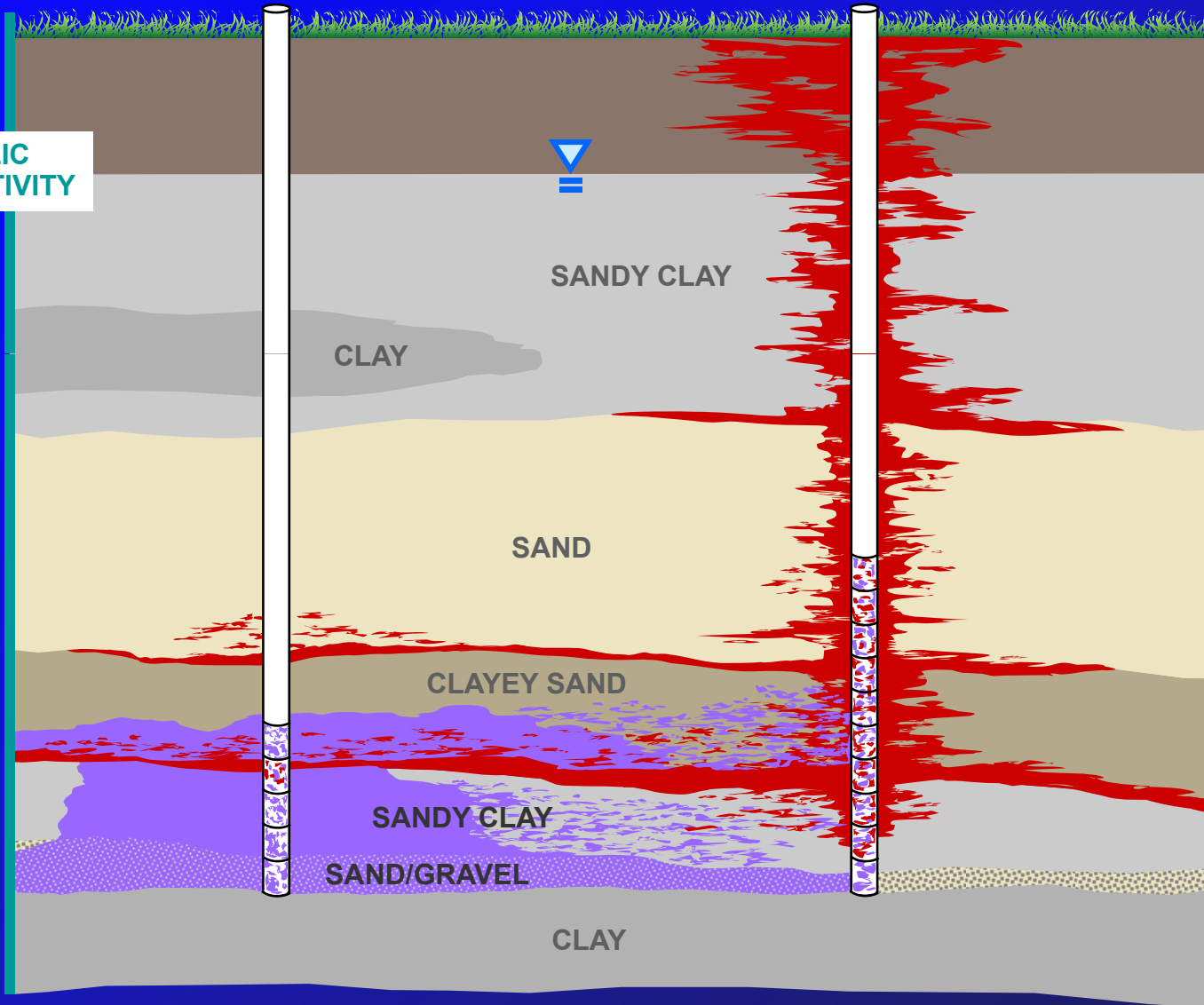
HIGH

MEDIUM

LOW

**VERY
HIGH**

**VERY
LOW**



SANDY CLAY

CLAY

SAND

CLAYEY SAND

SANDY CLAY

SAND/GRAVEL

CLAY



PCE Oxidation

KMnO₄ oxidation of PCE
under homogeneous
conditions (Yan and Schwartz,
1999)

50% removal in 4-9 hours
involving approximately 100
mg/L KMnO₄

Explains both PCE oxidation
and PCE persistence

sample is “transient”

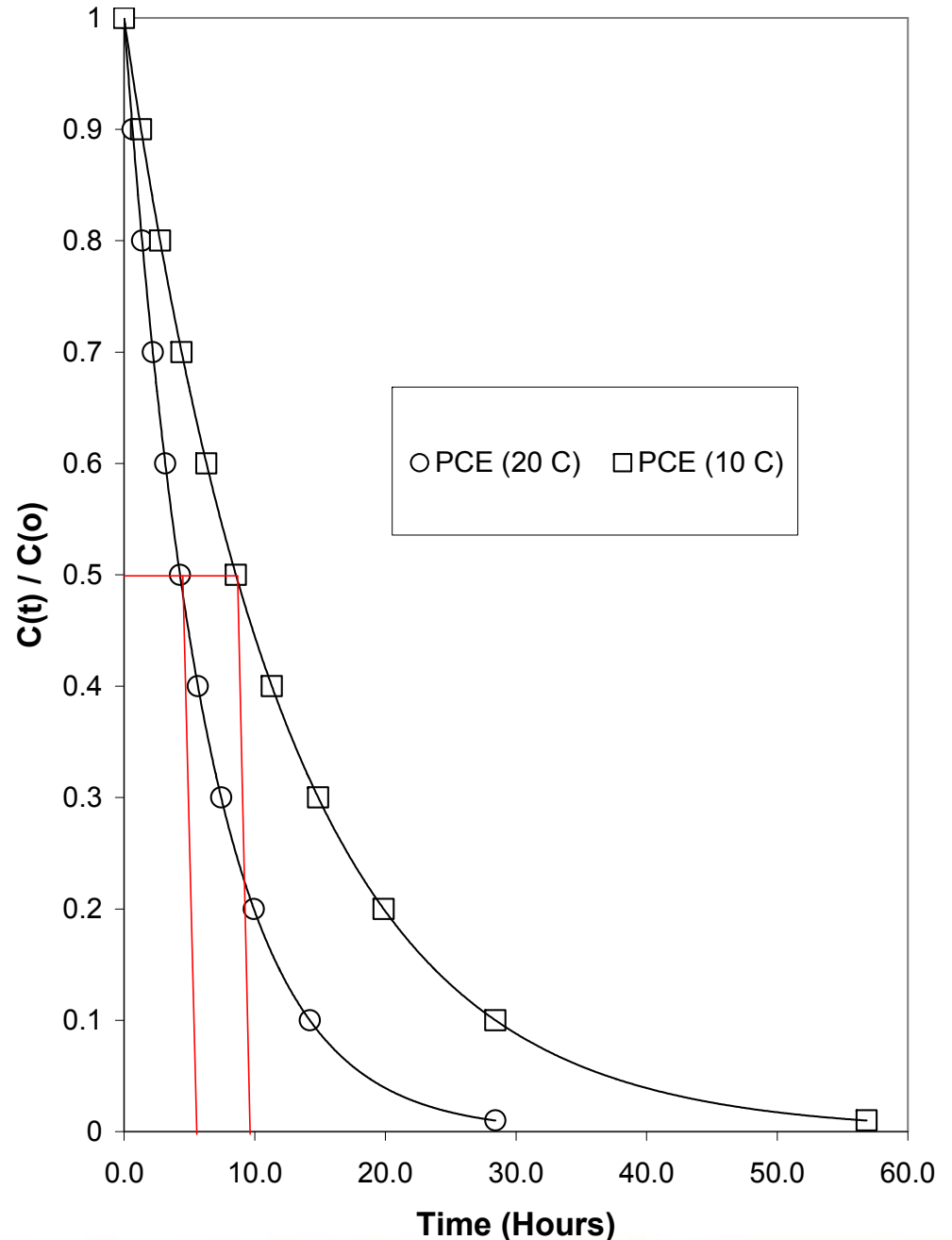


Table 1. First order transformation of chlorinate volatile organic compounds.

contaminants	¹ reaction rate constant, k (s ⁻¹) (20 °C)	² reaction rate constant, k (s ⁻¹) (10 °C)	Time required for 50% loss in contaminant in binary sample (hours)	
			(20 °C)	(10 °C)
PCE	4.5×10 ⁻⁵	2.25×10 ⁻⁵	4.3	8.6
TCE	6.5×10 ⁻⁴	3.25×10 ⁻⁴	0.3	0.6
c-DCE	9.2×10 ⁻⁴	4.6×10 ⁻⁴	0.2	0.4
t-DCE	3.0×10 ⁻²	1.5×10 ⁻²	< 0.1	< 0.1
1,1-DCE	2.38×10 ⁻³	1.19×10 ⁻³	4.9	9.7

¹ The first-order reaction rate constant involved MnO₄⁻ at 1 mM (Yan and Schwartz, 1999); rate constant adjusted for temperature ($k_{10} = k_{20} / 2$).

Observations:

1. t-DCE detection doubtful in samples that contain MnO₄⁻
2. 1,1-DCE is most resistant (5-10 hours)
3. Important to preserve sample with neutralizer *if it is to be analyzed*
4. Important not to analyze *if it is not preserved*



Oxidant Injection Well, Well Point, or Other Oxidant Injection Method

Monitoring Well

HYDRAULIC
CONDUCTIVITY

LOW
VERY LOW
HIGH
MEDIUM
LOW
VERY HIGH
VERY LOW

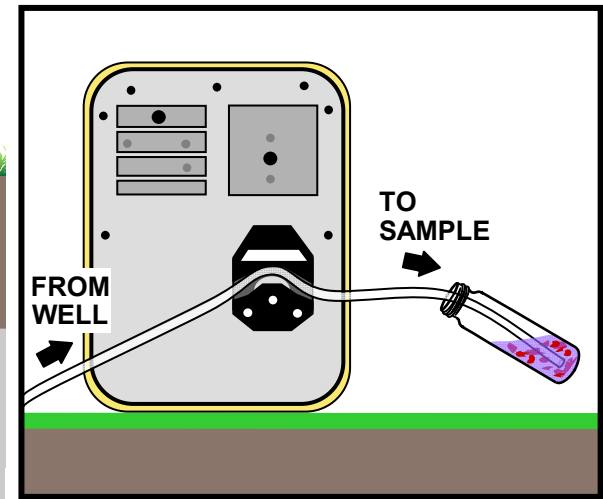
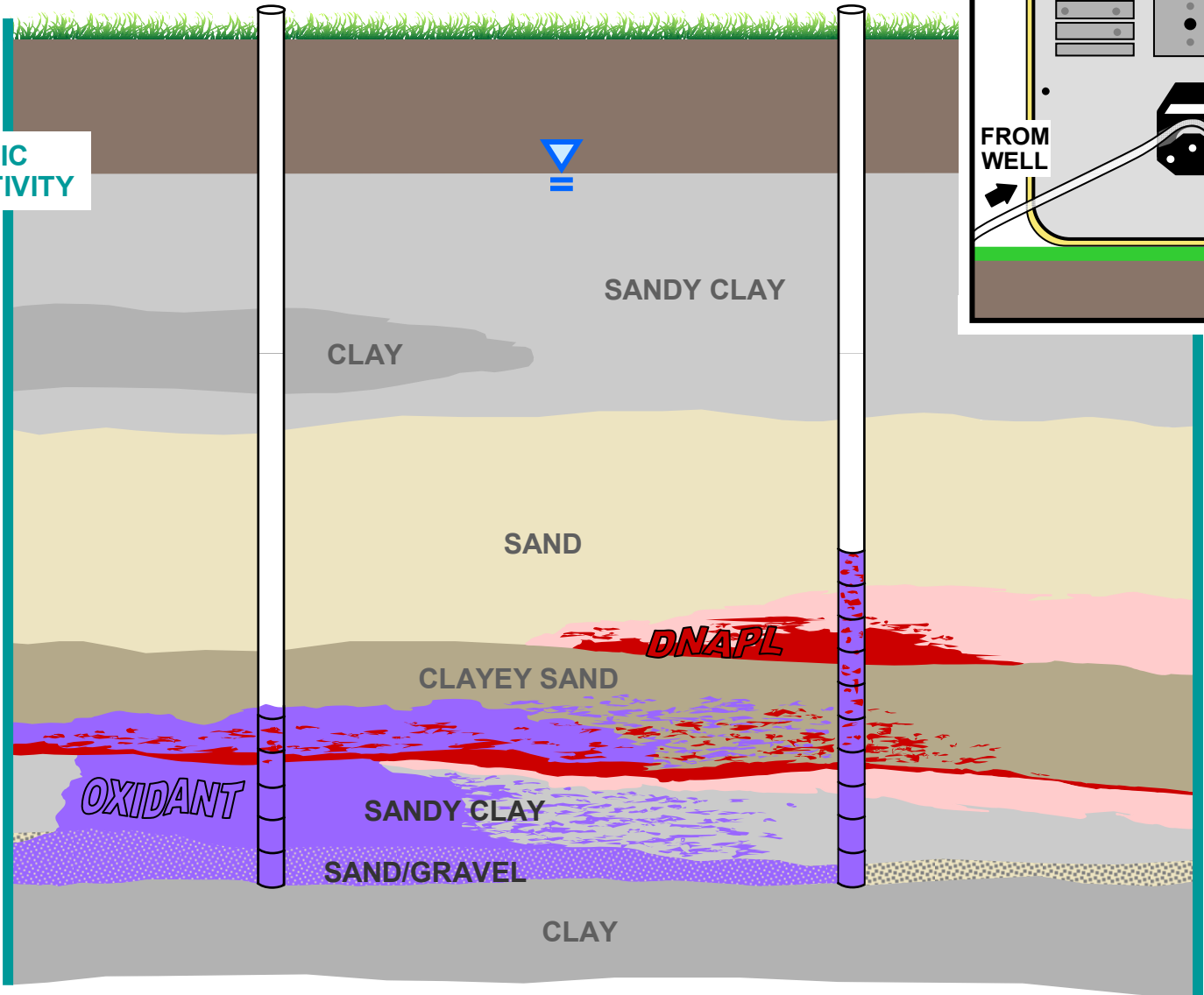
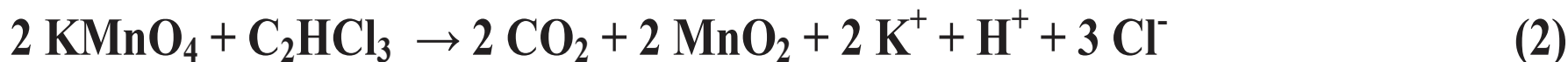


Table 2. Balanced chemical oxidation reactions involving chlorinated volatile organic compounds and potassium permanganate.



Stoichiometric requirements (mol KMnO₄/mol CVOC)

PCE 1.33

TCE 2.0

DCE 2.67

VC 3.33



Impact on ground water quality



Light pink
(1 mg/L)

- Dark pink
- (100 mg/L)

Contaminant	CVOC oxidized (µg/L)	CVOC oxidized (µg/L)
PCE	79,000	790
TCE	42,000	420
DCE	23,000	230
VC	13,000	130



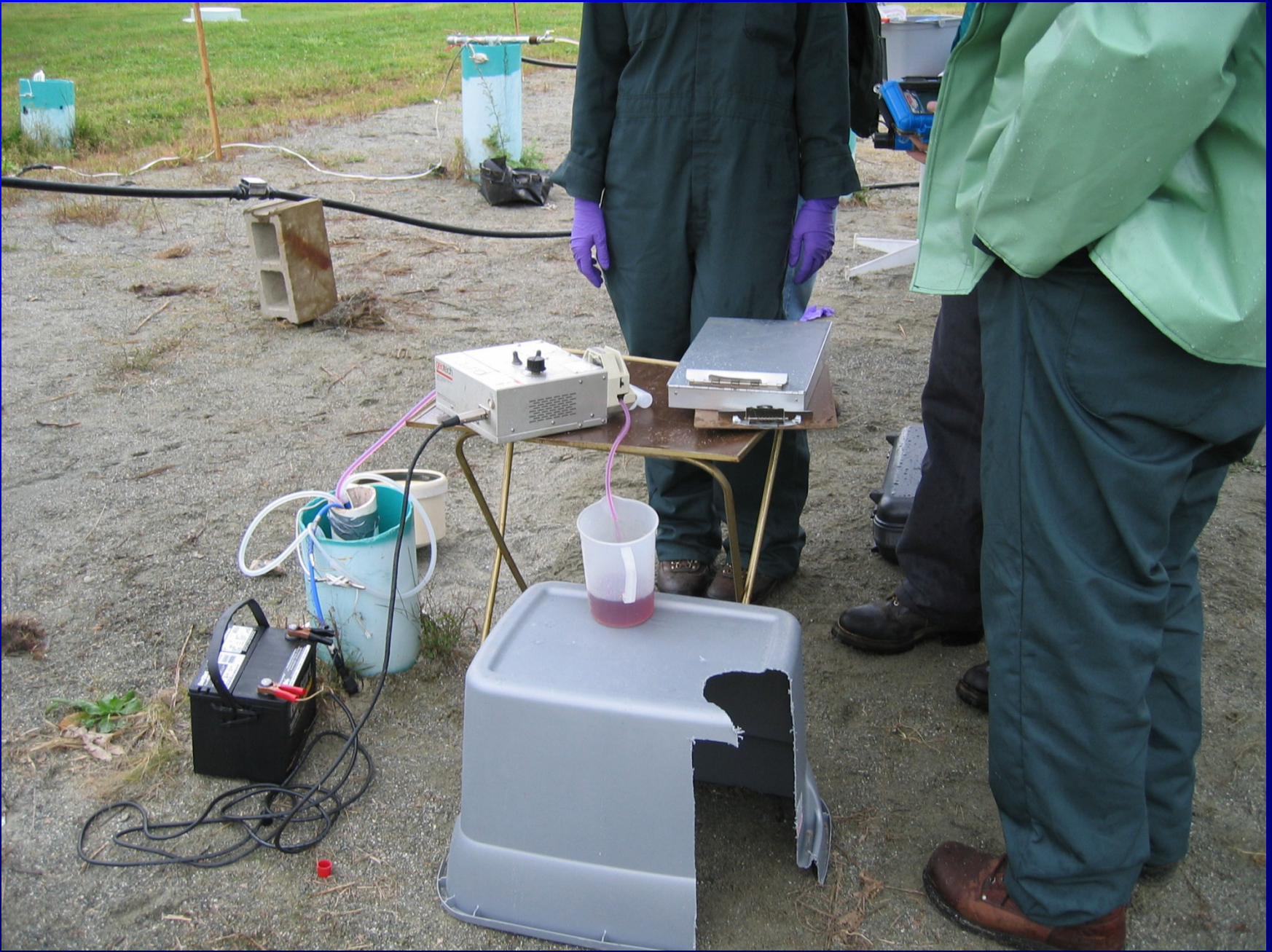
RESEARCH & DEVELOPMENT

Building a scientific foundation for sound environmental decisions



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Potential impact of $S_2O_8^{2-}$ and H_2O_2 on ground water quality

- **Persulfate**
 - Direct oxidation
 - Activation – base; thermal; iron chelate (Fe^{+2}); UV light
- **H_2O_2**
 - Direct oxidation - limited
 - Activation - Fenton mechanism (Fe); UV light
- **Conclusions**
 - oxidation of organics after ground water sample is collected
 - An aqueous sample needs to be analyzed to determine whether the oxidant is present; take appropriate action



Field Methods – Measure [Oxidant]

- **Field analytics - useful to provide quick, real-time measurements**
 - **field staff can make a decision and take appropriate steps**
 - Sample, detect, preserve (submit for analysis)
 - Sample, detect, don't analyze
- **Colorless – field measurement is needed**
 - **Persulfate**
 - Field test kit – CHEMetrics, FMC
 - Colorimetric - ferrous ammonium sulfate ($\lambda = 450 \text{ nm}$)
 - **H₂O₂**
 - Field test kits - CHEMetrics, LaMotte, Hach
 - Colorimetric – titanium sulfate ($\lambda = 407 \text{ nm}$)



Impact of oxidant on analytical instruments

(Karen T. Johnson and Margie Wickham-St Germain
US EPA Region 7)



RESEARCH & DEVELOPMENT

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Preservation techniques

- **Dilution (Margie St. Germain, US EPA Region 7)**
 - “Raises the reporting (detection) limit above mandated action levels making the data unusable”
 - Similar observation at Savage Well site (Region 1)
- **Oxidant neutralizers (reductants)**
 - ascorbic acid, sodium thiosulfate, sodium bisulfite, sodium metabisulfite, manganese sulfate, hydrazine hydrate, hydrazine sulfate, sugar, H₂O₂, hydrochloric acid, others, ...
 - **Add sufficient quantity to neutralize the oxidant**
 - Small volume to minimize dilution
 - Minimize other effects (CO₂ and O₂ sparging, etc.)



Preservation techniques

- Comparison study between ascorbic acid, acetic acid, sodium thiosulfate, and HCl (Karen T. Johnson and Margie St. Germain, US EPA Region 7)
- “Ascorbic acid was found to be an effective preservative which met the ideal conditions, as well as being safe, inexpensive, and easy to use in the field.”
- A field study was conducted where ascorbic acid was added in the field versus in the lab
 - CVOC concentrations: Field preserved > lab preserved
 - CVOC oxidation occurred after sample collected
- How much ascorbic acid should be added given the concentration of MnO_4^- ?



Table 1. Permanganate concentration, spectrophotometric absorbance at 525 nm, and required amount of ascorbic acid required to neutralize the oxidant (1.8 mol ascorbic acid / mol MnO_4^-)*.

[MnO₄⁻] (mg/L) (millimolar in parentheses)													
0 (0)	0.75 (0.01)	3.8 (0.03)	7.5 (0.06)	11.3 (0.09)	18.8 (0.16)	30.1 (0.25)	37.6 (0.32)	56.4 (0.47)	75.3 (0.63)	113 (0.95)	151 (1.27)	188 (1.58)	376 (3.16)
Absorbance⁽¹⁾, wavelength (λ) = 525 nm													
0	0.011	0.059	0.134	0.197	0.329	0.516	0.627	NL	NL	NL	NL	NL	NL
Ascorbic Acid Stock Solution (M)⁽²⁾													
-	0.015	0.015	0.15	0.15	0.15	0.15	0.15	1.5	1.5	1.5	1.5	1.5	1.5
Volume of Ascorbic Acid solution (μL)													
0	30	150	30	46	76	121	152	23	30	46	61	76	152
Mass of Ascorbic Acid (mg)													
0	0.08	0.4	0.79	1.21	2.1	3.32	4.17	6.1	7.9	12.2	16.1	20.1	40.2
(1) $[\text{MnO}_4^-]$ (mg/L) = $58.8 \times A_{525}$; A_{525} is the absorbance at 525 nm; non-linear above 38 mg/L MnO_4^- .													
(2) To minimize sample dilution, the ascorbic acid stock solution used was 0.015, 0.15, and 1.5 M.													

* EPA GROUND WATER ISSUE: Ground Water Sample Preservation at ISCO Sites – Recommended Guidelines



Binary System - Persulfate + VOC's

- GWMR “Ground Water Sampling at ISCO Sites – Binary Mixtures of Volatile Organic Compounds and Persulfate”

GWMR, (31) 2, Spring 2011, pages 72-79

Groundwater Sampling at ISCO Sites: Binary Mixtures of Volatile Organic Compounds and Persulfate

by Scott G. Huling, Saebom Ko, and Bruce Pivetz

Abstract

In situ chemical oxidation involves the introduction of a chemical oxidant into the subsurface for the purpose of transforming groundwater contaminants into harmless by-products. Owing to oxidant persistence, groundwater samples collected at hazardous waste sites may contain both the contaminant(s) and the oxidant in a “binary mixture.” Binary mixtures composed of sodium persulfate (2.5 g/L; 10.5 mM) and volatile organic compounds (VOCs) (benzene, toluene, *m*-xylene, perchloroethylene, trichloroethylene) were analyzed to assess the impact on the quality of the sample. A significant decline (49 to 100%) in VOC concentrations was measured in binary mixtures using gas chromatography (GC) purge and trap, and GC mass spectroscopy headspace methods. Preservation of the binary mixture samples was achieved through the addition of ascorbic acid (99 to 100% VOC average recovery). High concentrations of ascorbic acid (42 to 420 mM) did not interfere in the measurement of the VOCs and did not negatively impact the analytical instruments. High concentrations of ascorbic acid favored the reaction between persulfate and ascorbic acid while limiting the reaction between persulfate and VOCs. If an oxidant is detected and the binary sample is not appropriately preserved, the quality of the sample is likely to be compromised.

Introduction

Binary Mixtures of Oxidant and Organic Contaminants in Groundwater Samples

In situ chemical oxidation (ISCO) involves the introduction of a chemical oxidant into the subsurface for the purpose of transforming groundwater or soil contaminants into less harmful chemical by-products (Rivas 2006; Ferrarese et al. 2008; Kao et al. 2008). An integral component of ISCO is the collection and analysis of groundwater samples to assess ISCO treatment performance. Often, groundwater samples collected specifically to analyze organic contaminants may contain the oxidant and the organic contaminants in a “binary mixture.” This commingling of organic contaminants and oxidants in the groundwater sample represents a condition in which there is significant potential for oxidative transformation of the contaminants after sample collection. Consequently, the quality of the groundwater sample is compromised and a false negative may result. The oxidant may also potentially affect the analytical instruments used to quantify the concentration of groundwater analytes. To mitigate this potential problem, detection of the oxidant in

binary mixtures and neutralization of the oxidant are needed immediately after sample collection, and prior to analysis.

A site-specific critical analysis of contaminant and oxidant fate and transport conditions is needed to help understand the cause of binary mixtures. Several subsurface fate and transport conditions exist that result in commingling of organic contaminants and oxidant residuals in groundwater samples. The main cause of commingling is attributed to heterogeneities in aquifer materials and heterogeneous distribution of oxidants and contaminants in the subsurface. Groundwater solutes can enter a monitoring well screen from different lithologic zones, containing different levels of oxidant and contaminant. These groundwater solutes may be transported disproportionately in preferential pathways as separate solutes from different lithologic zones or as a binary mixture from the same lithologic zone (Figure 1). Insufficient contact time between the oxidant and contaminant prior to, or after, entering the well leads to binary mixtures. High levels of contamination, including dense nonaqueous phase liquids (DNAPLs) or light nonaqueous liquids (LNAPLs) immobilized in porous media near the monitoring well (i.e., in the source zone), will impart high levels of contaminant concentrations to the groundwater. During groundwater sampling, mobilization of LNAPL or DNAPL droplets from inside the well or from contaminated porous media near the well may result in high contaminant concentrations in the groundwater samples. Cold groundwater temperature or poor activation chemistry, and the resulting limited reaction

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Binary System - Persulfate + VOC's

- Benzene, Toluene, m-Xylene, PCE, TCE
 - 500 – 900 µg/L
- Persulfate
 - 0.1 - 2.5 g/L
- Stored at 4 °C until analyzed
- Sample sets were removed from the refrigerator (sequentially) and analyzed
 - GC/MS - Headspace (8260 C, 5021 A)
 - GC – purge and trap (EPA 501, 502.2, 503.1, 524.2, 601, 602, 624, 8010, 8020, 8021, 8240, and 8260)
- Prior to analyses - no reaction of persulfate or loss of VOC's (in controls) was measured



Table 2. Transformation of VOCs in binary mixtures containing persulfate (2.5-2.6 g L⁻¹; 10.5-10.9 mM) using the GC/MS headspace and GC purge and trap methods¹.

<i>GC/MS Headspace</i>	Initial Concentration (μM)	Final Concentration (μM)	Percent Loss (%)
Benzene	7.7 (7.3-8.1)	3.9 (3.9-4.0)	49
Toluene	7.9 (7.6-8.2)	2.3 (2.21-2.35)	71
<i>m</i> -Xylene	5.7 (5.58-5.90)	0.54 (0.29-0.79)	91
PCE	3.0 (2.98-3.11)	ND	100
TCE	4.8 (4.72-4.87)	ND	100
<i>GC Purge and Trap</i>			
Benzene	10.2 (10.1-10.4)	2.4 (2.1-2.7)	76
Toluene	8.7 (8.3-9.0)	0.52 (0.18-0.86)	94
<i>m</i> -Xylene	6.2 (5.9-6.4)	ND	100

¹ All aqueous samples prepared in triplicate; average value reported (n=3); 95% confidence interval in parentheses.

- Significant loss in all VOC's, especially CVOC's (initial concentrations 500-800 μg/L)
- Sparging step in headspace analysis involves heating; thermal activation
 - Sample is heated from room temperature to 80 °C, 30 minutes
- PS activation during GC analysis may be due to x-port of PS aerosols to the trap.
- No impact on analytical instrument



Analytical Method Analyte	² Ascorbic Acid / Persulfate (mmol/mmol)	Initial Concentration (µM)	Final Concentration (µM)	Percent Recovery (%)
GC/MS Headspace Benzene	0	7.29 (7.09 - 7.49)	7.32 (7.29 - 7.34) 7.29 (7.15 - 7.43) 7.23 (7.09 - 7.37) 7.47 (7.38 - 7.57) 6.88 (6.87 - 6.90)	100 100 99 102 94
	4			
	7			
	10			
	13			
	40			
Toluene	0	6.39 (6.16 - 6.62)	6.40 (6.19 - 6.61) 6.43 (6.29 - 6.56) 6.29 (6.08 - 6.51) 6.42 (6.36 - 6.48) 6.02 (5.98 - 6.07)	100 101 98 101 94
	4			
	7			
	10			
	13			
	40			
<i>m</i> -Xylene	0	3.35 (3.17 - 3.52)	3.19 (2.93 - 3.44) 3.28 (3.20 - 3.36) 3.42 (3.38 - 3.46) 3.48 (3.43 - 3.53) 3.27 (3.25 - 3.29)	95 98 102 104 98
	4			
	7			
	10			
	13			
	40			
PCE	0	1.63 (1.60 - 1.66)	1.54 (1.52 - 1.55) 1.53 (1.48 - 1.58) 1.55 (1.48 - 1.62) 1.60 (1.56 - 1.64) 1.61 (1.56 - 1.66)	95 94 95 98 99
	4			
	7			
	10			
	13			
	40			
TCE	0	4.29 (4.21 - 4.38)	4.41 (4.26 - 4.56) 4.28 (4.03 - 4.53) 4.31 (4.21 - 4.40) 4.34 (4.26 - 4.42) 4.12 (4.00 - 4.24)	103 100 101 101 96
	4			
	7			
	10			
	13			
	40			

**Average
Recovery
99%**



Analytical Method Analyte	² Ascorbic Acid / Persulfate (mmol/mmol)	Initial Concentration (μM)	Final Concentration (μM)	Percent Recovery (%)
<i>GC Purge and Trap</i> Benzene	0	11.16 (11.06-11.26)	10.85 (10.75 - 10.95) *	97
	4			
	7			
	10			
	13			
	40			
Toluene	0	10.20 (9.80-10.61)	9.85 (9.79 - 9.91) *	99
	4			
	7			
	10			
	13			
	40			
<i>m</i> -Xylene	0	10.20 (10.05-10.35)	10.11 (9.91 - 10.31) 10.36 (10.23 - 10.49) 10.30 (10.10 - 10.49) 10.58 (10.50 - 10.65) *	99 102 101 104
	4			
	7			
	10			
	13			
	40			
¹ All aqueous samples prepared in triplicate; average value reported (n=3); 95% confidence interval in parentheses. ² The 0 value represents the ascorbic acid- and persulfate-free samples used to establish baseline. * Data invalidated due to inverted septa.				

**Average
Recovery
100%**



Why does ascorbic acid work in this manner?

Relative reaction rate (R_R)

$$R_R = \frac{K_7 [\cdot\text{SO}_4^-] [\text{AH}_2]}{k_9 [\cdot\text{SO}_4^-] [\text{Benzene}]} \approx 125-1250$$

$$[\text{S}_2\text{O}_8^{2-}] = 10.5 \text{ mmol L}^{-1}$$

$$[\text{AH}_2] = 40-420 \text{ mmol L}^{-1}$$


$$[\text{Benzene}] = 11.2 \text{ } \mu\text{mol L}^{-1}$$

$$k_7 = 1 \times 10^9 \text{ M}^{-1} \text{ s}^{-1}$$

$$k_9 = 3 \times 10^9 \text{ M}^{-1} \text{ s}^{-1}$$



Table 2. Persulfate concentrations; absorbance at a wavelength of 450 nm; ferrous ammonium sulfate method; required amount of ascorbic acid required to neutralize the oxidant*.

													
[S₂O₈²⁻] (mg/L) (millimolar in parentheses)													
0	80	200	400	610	810	1210	1610	2020	2420	2820	3230	3630	4030
0	(0.42)	(1.1)	(2.1)	(3.2)	(4.2)	(6.3)	(8.4)	(10.5)	(12.6)	(14.7)	(16.8)	(18.9)	(21.0)
Absorbance⁽¹⁾, wavelength (λ) = 470 nm													
0	0.011	0.019	0.04	0.062	0.076	0.121	0.164	0.204	0.245	0.275	0.313	0.349	0.397
Volume of Ascorbic Acid solution (mL)													
0	0.04	0.11	0.22	0.34	0.45	0.67	0.89	1.12	1.34	1.57	1.79	2.02	2.24
Mass of Ascorbic Acid (176.12 g/mol) (g)													
0	0.01	0.03	0.06	0.09	0.12	0.18	0.24	0.3	0.35	0.41	0.47	0.53	0.59
(1) Solubility of ascorbic acid in water = 330 g/L (1.87 mol/L); 80% solubility (1.5 mol/L) used as stock solution; [S ₂ O ₈ ²⁻] (mg/L) = 10,000 × A ₄₅₀ ; where A ₄₅₀ is the absorbance at 450 nm.													

* EPA GROUND WATER ISSUE: Ground Water Sample Preservation at ISCO Sites – Recommended Guidelines



Former Naval Ammunition Support

Detachment, Vieques PR (Diana Cutt, EPA Region 2)

- **Small-scale persulfate injection (TCE, PCE)**
- **Persulfate residual in ground water (1-125 mg/L)**
 - Letter from FMC (< 0.5 g/L)
 - Previous work – 2.5 g/L
 - Would lower PS concentrations impact ground water quality (“probably, but no data to support”)
 - **Test 0.1 – 2.0 g/L persulfate with benzene**
 - **Analyze with GC (purge and trap) and GC/MS (headspace)**





Environmental Industry Team
Chemical Products Group
1735 Market St.
Philadelphia, PA 19103

September 10, 2010

RE: Reactivity of Dilute Concentrations of Klozur[®] Persulfate

It is the experience of FMC over the past ten years that the minimum reactive concentration of sodium persulfate in groundwater is 0.5 g / L (500 ppm). Oxidative reaction rate is proportional to the concentration of the contaminant, the concentration of the oxidant and the concentration of the persulfate activator. At concentrations below this level, the effective reaction rate with contaminants of concern is essentially zero, and for all intents and purposes the oxidative reaction is complete. This is further impacted by the co-incident reduction in persulfate activator concentration.

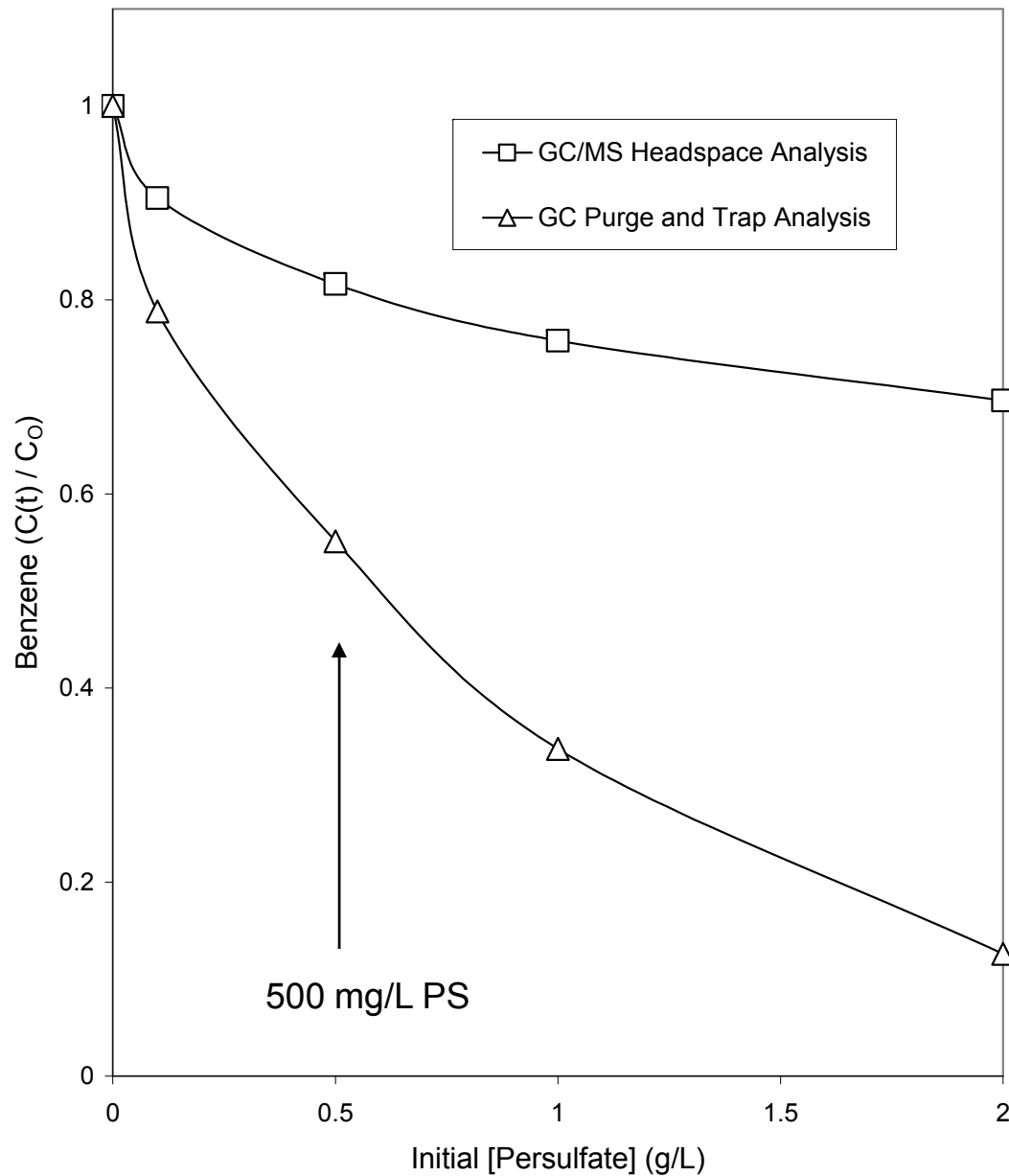
Transportation of groundwater samples containing less than 500 ppm of persulfate should not occur further significant contaminant reduction in route to the laboratory, assuming the transportation time is not significant (less than a couple of days) and the sample is not exposed to a significant heat source. This can be further mitigated by shipment of the sample on ice.

Philip Block
Technology Manager – Remediation
FMC Corporation



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Oxidation Results

0.1 – 2.0 g/L PS

**[Benzene]_{INITIAL} ≈ 760 µg/L GC/MS
≈ 720 µg/L GC**

0.1 and 0.5 g/L PS

**10% and 19% loss (GC/MS)
21% and 42% loss (GC)**

**Note: chemical oxidation sensitivity
Benz < Tol, Xyl < TCE, PCE**

Recommendation:

- 1. Allow PS to react longer, or**
- 2. Preserve the sample**



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Ongoing Work

- **Impact of persulfate on VOCs involving GC and GC/MS analysis (BTX, PCE, TCE)**
 - GC/MS - Headspace (8260 C, 5021 A)
 - GC – purge and trap (EPA 501, 502.2, 503.1, 524.2, 601, 602, 624, 8010, 8020, 8021, 8240, and 8260)
- **Investigating HPLC analysis of semi-volatiles**
 - Naphthalene, Pentachlorophenol, 1,4-Dichlorobenzene
 - Thermal step
 - Base extraction step
- **Base activated (pH > 10.5)**
- **Lower limit [S₂O₈²⁻] effects**
 - CHEMetrics (7-70 mg/L)
 - FMC (< 2.5 g/L)
- **UV light effects**



... Ongoing Work

- **Lab versus field preservation**
 - Advantages and disadvantages
- **Simplification of ascorbic acid amendment procedures to $S_2O_8^{2-}$ binary mixtures**
 - Uniform volume of ascorbic acid amendment - 2.5 mL
 - **Collaboration with FMC to develop a kit**
 - Free pipetter and ascorbic acid
- **Compile analytical methods and analytes**
 - eg. Fike/Artel Superfund Site
 - $MeCl_2$ extraction, GC/MS analysis
- **Provide guidelines for ground water sampling at ISCO sites – Ground Water Forum Issue Paper**



Binary Mixtures: Ground Water Sampling at ISCO Sites

- **Summary**

- Important issue that is relevant at a large number of ISCO sites (binary mixtures; questionable quality)
- May be responsible for some of the rebound effects reported at sites; applicable to bench-scale treatability studies where binary samples were collected and analyzed
- The effects of analyzing binary mixtures are real, significant, and have probably impacted decisions made at some sites
- The problem can be resolved by taking the appropriate steps



Binary Mixtures: Ground Water Sampling at ISCO Sites

- **Preliminary recommendations**
 - **Assess the benefit/risk of binary mixture analysis**
 - **Oxidant detection in ground water samples**
 - **Preserve the sample if it is to be analyzed**
 - **Oxidant neutralizer**
 - **Ascorbic acid (others available)**
- **Technology transfer**
 - **Technical presentations, journal articles, EPA research brief**



Ground Water Sampling at ISCO Sites – Effects of Binary Mixtures

Questions?

