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# High-performance liquid chromatography–diode array detection of trichloroethene and aromatic and aliphatic anionic surfactants used for surfactant-enhanced aquifer remediation

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## Abstract

A method utilizing direct aqueous injection with high-performance liquid chromatography and diode array detection (HPLC–DAD) is presented for the quantitation determination of trichloroethene (TCE) in the presence of anionic surfactants that are used to enhance the recovery of dense non-aqueous phase liquids from contaminated groundwater aquifers. The anionic surfactants investigated in this study including alkyl diphenyloxide disulfonate (Dowfax 8390) and dihexylsulfosuccinate (Aerosol MA 80-I) are used to enhance the solubility, and hence recovery, of TCE. In this type of environmental engineering application, the levels of surfactants and TCE encountered are very high (part per million to part per thousand). The anionic surfactants and TCE are quantitatively determined by direct aqueous injection onto a reversed-phase HPLC column with diode array detection. The quantitation limits of the method obtained using 100  $\mu$ l injections are 0.1 mg/l for alkyl diphenyloxide disulfonates, 20 mg/l for dihexylsulfosuccinate, and 0.05 mg/l for TCE. This approach is advantageous over using gas chromatography for TCE and HPLC for the surfactants because the use of a single analytical instrument reduces sample preparation and analysis times, which increases sample throughput. © 2000 Elsevier Science B.V. All rights reserved.

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## 1. Introduction

Attention is now focused on chemical remediation technologies that enhance the removal of dense nonaqueous phase liquids (DNAPLs), which are common contaminants in subsurface environments. Sites containing residual DNAPL (liquid trapped in the pore space by capillary forces) are particularly

difficult to remediate by traditional pump-and-treat methods because of the slow rate of dissolution and typically small aqueous-phase solubility of DNAPL components.

Surfactant-containing products such as Dowfax 8390 and Aerosol MA 80-I, which contain alkyl diphenyloxide disulfonates and dihexylsulfosuccinates, respectively, have been identified as promising surfactant products for the remediation of groundwater contaminated by DNAPLs such as trichloroethene (TCE) and perchlorethene [1–5]. Increased DNAPL solubility results from the partitioning of

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DNAPL molecules into the hydrophobic interior of surfactant micelles, which form above the surfactant's critical micelle concentration (CMC).

Disulfonated surfactants are of particular interest in surfactant-enhanced aquifer remediation because they are less susceptible to losses due to precipitation and sorption onto aquifer sediments as compared to other anionic surfactants such as linear alkylbenzene sulfonates (LAS) [1,3,6]. In addition, their phase behavior can be controlled by the addition of salts [7,8] and the density of the surfactant solutions is modified with the addition of alcohols [9].

The conventional approach to selecting surfactants for surfactant-enhanced remediation is to select surfactants that enhance the solubility of oils (e.g., DNAPLs) by measuring increases or decreases in the *volume* of an aqueous surfactant phase relative to an oil (or DNAPL) phase [8]. However, measuring changes in parameters such as volume or surface tension is neither sufficiently quantitative nor selective for determining the concentrations of TCE and surfactants during field demonstrations of surfactant-enhanced DNAPL remediation. In surfactant-enhanced aquifer remediation, the concentrations of surfactant and DNAPL are very high and typically are expressed in units of mM or g/l [1,7,10,11].

The occurrence of volatile analytes, such as TCE, together with nonvolatile anionic surfactants in water samples obtained from laboratory and field experiments poses an analytical challenge due to the large difference in properties between the two classes of analytes. For example, although TCE is analyzed readily by gas chromatography (GC), sulfonated anionic surfactants such as alkyl diphenyloxide disulfonates and dihexylsulfosuccinates are not volatile and cannot be analyzed by GC without derivatization. Although sulfonated surfactants such as linear alkylbenzene sulfonate can be derivatized by injection-port derivatization GC at high temperatures [12,13], no information is available on the derivatization of alkyl diphenyloxide disulfonates and dihexylsulfosuccinates. Aromatic sulfonated surfactants typically are measured by means of high-performance liquid chromatography (HPLC) with UV detection [1,14–16] or with mass spectrometric detection [17]. Aliphatic sulfonated surfactants, on the other hand, lack a chromophore and are determined by ion chromatography with conductivity

detection [18–20], capillary electrophoresis with conductivity detection [21], or HPLC with indirect photometric detection [22] or off-line ion-pair extraction with fluorescence detection [23].

With existing analytical approaches for TCE and sulfonated surfactants, two analytical instruments are required. As an alternative, a simple and rapid analytical method was needed for the determination of high concentrations (part per million to part per thousand) of TCE and anionic surfactants. In addition, due to the large number of samples anticipated for field and laboratory experiments, a method was desired that required only minimal sample handling and preparation and the use of a single analytical instrument. As a result, a direct aqueous injection HPLC method was developed for the quantitative determination of TCE and its degradation products, such as dichloroethene which commonly co-occur with TCE, and anionic surfactants (e.g., alkyl diphenyloxide disulfonates and dihexylsulfosuccinate) used in surfactant-enhanced remediation.

## 2. Experimental methods

### 2.1. Standards

Acetonitrile and methanol (HPLC grade) were purchased from Fisher Scientific (Fair Lawn, NJ, USA). Standards for TCE, 1,1-dichloroethene (1,1-DCE), *trans*-1,2-dichloroethene (*trans*-DCE), and *cis*-1,2-dichloroethene (*cis*-DCE) were obtained from Aldrich (Milwaukee, WI, USA) and were diluted with methanol to create standards. Standards in methanol were stable for months when tightly capped and stored at 4°C. Dowfax 8390, which is a 33% active mixture that consists of 90% disodium hexadecyl diphenyloxide disulfonate and 10% disodium dihexadecyl diphenyloxide disulfonate, was donated by Dow Chemical (Midland, MI, USA; Fig. 1). The acronym MADS is used hereafter to refer to disodium hexadecyl diphenyloxide disulfonate, which is the mono alkylated component in the Dowfax mixture and DADS is used to refer to the disodium dihexadecyl diphenyloxide disulfonate or the dialkylated component of the Dowfax mixture. Aerosol MA 80-I, which is an 80% active mixture of sodium dihexylsulfosuccinate and 5% isopropanol,

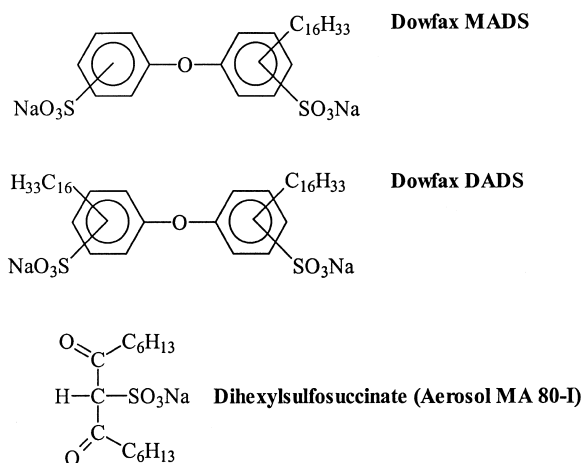


Fig. 1. Structures of disodium hexadecyl diphenyloxide disulfonate (MADS) and disodium dihexadecyl diphenyloxide disulfonate (DADS) in Dowfax 8390 and sodium dihexylsulfosuccinate in Aerosol MA 80-I.

was donated by Cytec (West Paterson, NJ, USA; Fig. 1). Surfactant standards were prepared as aqueous solutions. Groundwater contaminated with TCE was obtained from Lawrence Livermore National Laboratory (LLNL) Site 300, which is described in detail in Istok et al. [6] and Field et al. [11].

## 2.2. Samples

Samples containing either Dowfax 8390 and TCE were obtained during “push–pull” tests conducted in physical aquifer models that simulate the radial flow near an injection/extraction well during a push–pull test [6,11]. The physical aquifer models were constructed in a wedge shape and packed with sediment obtained from a field located at site 300 Building 834 operable unit at LLNL in Livermore, CA, USA. Liquid TCE was injected into the sediment pack to give a TCE saturation equivalent to 5% of the total pore volume. Solutions containing a conservative tracer (bromide) and either Dowfax 8390 or Aerosol MA 80-I were injected into the narrow end of the model. During the extraction phase, the flow was reversed and samples were collected from the narrow end and from ports in the top of the model. Samples obtained during experiments conducted with Dowfax 8390 in physical aquifer models containing TCE were diluted 100-fold without filtration.

Experiments were performed to determine the solubility of TCE in Aerosol MA 80-I solutions. Deionized water (3 ml) containing 47 500 mg/l Aerosol MA 80-I (38 000 mg/l active dihexylsulfosuccinate), 1052 mg/l KBr, and 10 000 mg/l isopropanol was combined with 1 ml of neat TCE and shaken for 30 s. The phases were allowed to separate overnight to allow for any unstable macroemulsions to break prior to analysis. Samples (3 ml) containing Aerosol MA 80-I and TCE also were obtained from physical aquifer model experiments [10]. From each individual sample, a sample was prepared for TCE analysis and consisted of removing 19  $\mu\text{l}$  and adding it to 1.88 ml of deionized water. The remaining sample was filtered through a 0.45  $\mu\text{m}$  nylon syringe filter before injecting it onto the HPLC for surfactant analysis. The portion immediately removed for TCE analysis that was not filtered minimized any TCE loss; the dilution effectively eliminated the need for pre-filtering the sample containing TCE.

Groundwater samples that contained TCE and its degradation products but no surfactant were obtained from the TCE-contaminated aquifer at the 300 Building 834 operable unit site at LLNL. Samples were collected in 40 ml bottles fitted with PTFE-lined lids, inverted for transport and storage, and stored at 4°C prior to analysis.

## 2.3. HPLC/DAD

All separations were performed on a Waters 2690 separations module equipped with a Waters 996 photodiode array detector (Waters, Milford, MA, USA) at 30°C on a 150 $\times$ 3.9 mm, 3  $\mu\text{m}$  particle size Waters C<sub>18</sub> NovaPak column and guard column. Three gradient separations are described for the simultaneous analysis of TCE and the alkyl diphenyloxide disulfonate surfactant in Dowfax 8390 (Method 1), dihexylsulfosuccinate in Aerosol MA 80-I in the presence of TCE (Method 2), and TCE and its degradation products in samples containing Aerosol MA 80-I as well as in TCE-contaminated groundwater that contains no surfactant (Method 3).

### 2.3.1. Alkyl diphenyloxide disulfonates (Dowfax 8390) and TCE (Method 1)

Samples containing <10 mg/l TCE and/or <30

mg/l Dowfax 8390 were injected by autosampler (2–100  $\mu$ l) directly onto the HPLC without filtration. The initial mobile phase consisted of 10% acetonitrile and 90% water at a flow-rate of 1 ml/min. The initial conditions were held for 1 min after which the acetonitrile was increased to 40% by 4 min using a sharp, non-linear, convex step. At 4 min, a similar gradient step was used to achieve 95% acetonitrile by 6 min. Initial conditions then were restored between 6 and 7 min to give a total analysis time of 10 min.

Alkyl diphenyloxide disulfonate detection was achieved at 238 nm while TCE detection was achieved at 210 nm. The concentration of the MADS and DADS were determined from linear calibration curves that ranged from 0.10 to 20.0 mg/l and from 0.01 to 2.5 mg/l, respectively. The concentration of the two components (MADS and DADS) was summed in order to report total alkyl diphenyloxide disulfonates. Concentrations of TCE and *trans*-DCE were determined from two linear calibration curves for each analyte ranging from 0.05 to 2.0 mg/l and from 0.5 to 15.0 mg/l. Concentrations of *cis*-DCE and 1,1-DCE were determined from two calibration curves for each analyte ranging from 0.05 to 1.5 mg/l and from 0.5 to 28 mg/l.

### 2.3.2. Aerosol MA 80-I and <150 mg/l TCE (Method 2)

Samples containing 20–120 000 mg/l dihexylsulfosuccinate and <150 mg/l TCE were injected by autosampler (2–100  $\mu$ l) directly onto the HPLC. The initial separation gradient conditions consisted of 80% 0.003 M NaCl and 20% acetonitrile at a flow-rate of 1.5 ml/min followed by a steep concave gradient to give 45% acetonitrile and 55% 0.003 M NaCl by 1.5 min. The percent acetonitrile then was increased in a linear manner to 70% by 4 min. The initial conditions then were restored between 4 and 5 min to give a total analysis time of 7 min. Dihexylsulfosuccinate was detected at 210 nm and quantified from a single linear calibration curve ranging from 20 to 120 000 mg/l.

### 2.3.3. Aerosol MA 80-I and >150 mg/l TCE and TCE-contaminated groundwater (Method 3)

Because of the high solubility of TCE in Aerosol MA 80-I, samples containing >150 mg/l TCE were

diluted to get the TCE response on scale. These samples were analyzed by direct aqueous injection with 5–100  $\mu$ l injection volumes. Because no surfactant was present, a simple linear gradient was used to elute TCE. The initial HPLC conditions consisted of 70% water and 30% acetonitrile at a flow-rate of 1 ml/min were followed by a linear gradient to 95% acetonitrile by 4 min. These conditions were held for 2 min after which initial conditions are reestablished between 6 and 7 min to give a total run time of 10 min. Groundwater samples containing <150 mg/l TCE and its degradation products but no surfactant also were analyzed by this method. Concentrations of TCE, 1,1-DCE, *cis*-DCE, and 1,1-DCE were quantified as described above under Method 1. Because dihexylsulfosuccinate absorbs only weakly at 210 nm, it is below detection by this simple linear gradient.

## 3. Results and discussion

Standards containing Dowfax 8390 and TCE and its degradation products initially were analyzed as separate standards by Method 1 to determine retention times and to optimize the UV wavelength for detection. At 238 nm, the disodium hexadecyl diphenyloxide disulfonates (MADS) eluted as a single peak at 3.50 min and the disodium dihexadecyl diphenyloxide disulfonates (DADS) eluted as a single peak at 5.16 min (Fig. 2a). The standards of *cis*-DCE, *trans*-DCE, 1,1-DCE, and TCE, which were detected at 210 nm, eluted as sharp peaks at 5.58, 5.74, 5.89, and 6.14 min, respectively (Fig. 2a). Single analyses then were performed on samples obtained from the physical aquifer model experiment conducted with Dowfax 8390 in the presence of 5% residual TCE. Although the surfactant components responded at 210 nm and gave a slightly greater signal than at 238 nm, 238 nm was selected for surfactant detection and quantification because it yielded a smoother baseline for samples containing lower DADS concentrations. In addition, 210 nm was selected for the detection and quantification of TCE and its degradation products because it was approximately 17 times more sensitive than the corresponding signal obtained at 238 nm (Fig. 2b).

The precision of DOWFAX surfactant component

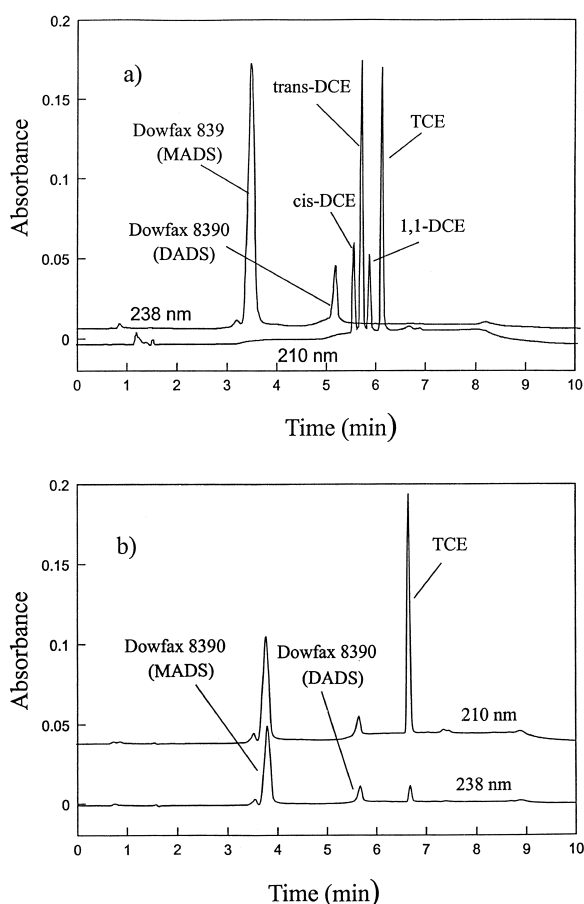


Fig. 2. Chromatograms obtained by Method 1 for 10  $\mu$ l injections of standards containing (a) 140 mg/l total Dowfax 8390 and 20 mg/l each of *cis*-DCE (5.58 min), *trans*-DCE (5.74 min), 1,1-DCE (5.89 min), and TCE (6.14 min) and (b) a sample (diluted 100-fold) that originally contained 3375 mg/l alkyl diphenyloxide disulfonates (Dowfax 8390) and 2000 mg/l TCE.

analyses, determined as the relative standard deviation (RSD), were determined from multiple ( $n=5$ ) injections of a single sample and analysis by Method 1 were  $\pm 1.2\%$  for the MADS and  $\pm 3.8\%$  for the DADS components of the Dowfax 8390 mixture. The quantitation limits of the method, defined as those concentrations that gave a signal-to-noise ratio of 10:1, were 0.1 mg/l for each of the alkyl diphenyloxide disulfonate components, 0.05 mg/l for TCE and *trans*-DCE, and 0.13 mg/l for *cis*-DCE and 1,1-DCE.

Attempts to analyze samples containing dihexylsulfosuccinate surfactant and TCE by HPLC Method

1 were unsuccessful as the surfactant eluted at the void volume of the column. However, under Method 2 conditions, in which NaCl was added to the mobile phase, dihexylsulfosuccinate eluted at 3.06 min followed by a large, off-scale peak at 4.8 min that corresponded to TCE (Fig. 3a). Replicate ( $n=5$ ) injections of samples containing Aerosol MA 80-I gave a RSD for dihexylsulfosuccinate of 0.3%. The quantitation limit of Method 2 using 100  $\mu$ l injections for dihexylsulfosuccinate was 20 mg/l. For Method 2, the mobile phase composition of 70% acetonitrile is more than is required to elute the dihexylsulfosuccinate; however, the high proportion

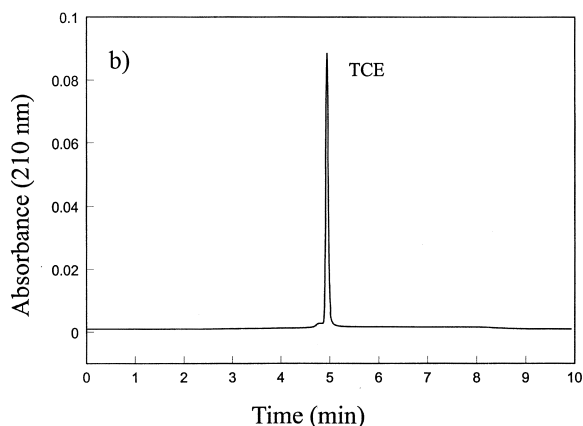
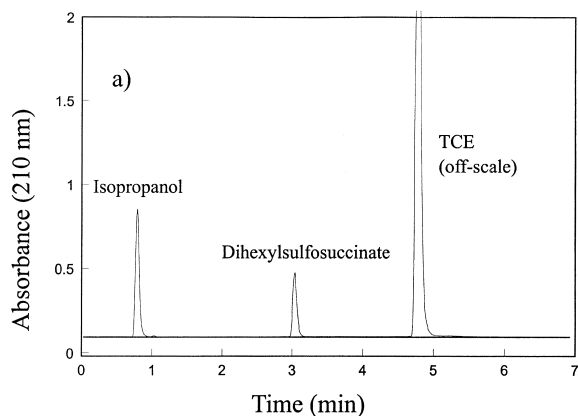


Fig. 3. Chromatograms of (a) 38.0 g/l dihexylsulfosuccinate and 27.0 g/l TCE (off-scale) obtained by Method 2 conditions (210 nm) and (b) the same sample diluted by a factor of 1000 and analyzed by Method 3 at 210 nm with the dihexylsulfosuccinate below detection and TCE on-scale.

of acetonitrile is required to elute large amounts of solubilized TCE. For this reason, when TCE is not present and only quantification of dihexylsulfosuccinate is required, the gradient between 45 and 70% acetonitrile can be eliminated.

Because Aerosol MA 80-I solubilizes high concentrations of TCE, dilution was necessary to get the TCE response on scale. For example, when diluted and analyzed by Method 3, the simple linear gradient method, the TCE appeared on-scale with the dihexylsulfosuccinate below detection (Fig. 3b). Under these conditions, two separate HPLC analyses must be performed to quantify the dihexylsulfosuccinate surfactant and TCE.

Prior to injecting solutions (e.g., no TCE present) containing the sodium salt of dihexylsulfosuccinate surfactant into the physical aquifer, chromatograms obtained by HPLC Method 2 indicated an isopropanol peak that eluted at the void volume of the column and a peak at 3.06 min that corresponded to dihexylsulfosuccinate (Fig. 4a). Chromatograms for samples obtained from within the physical aquifer model that contained TCE and that had been treated with Aerosol MA 80-I gave an isopropanol peak at the void volume of the column, and a split peak with the first peak at 3.06 min and the second peak centered at 3.22 min, and an off-scale TCE peak at 4.80 min (Fig. 4b). Although split, the summed peak areas of the two peaks were equivalent to that of the original mass of dihexylsulfosuccinate in the sample. The earlier eluting component (3.06 min) of the split peak had a retention time that matched that of the sodium dihexylsulfosuccinate originally present in Aerosol MA 80-I injectate. To test this hypothesis that the calcium salt of dihexylsulfosuccinate was the second peak at 3.22 min, calcium chloride was added to a sample of the injectate to give a final concentration of 800 mg/l. The reanalyzed sample contained a split peak (Fig. 4c) with retention times and UV spectra matching that for samples collected from the physical aquifer model (Fig. 4b), which indicated that the second of the two split peaks corresponded to the calcium salt of dihexylsulfosuccinate.

Detection of the calcium salt of dihexylsulfosuccinate indicates that Method 2 conditions are capable of separating the sodium and calcium salt of dihexylsulfosuccinate. Moreover, the presence of the

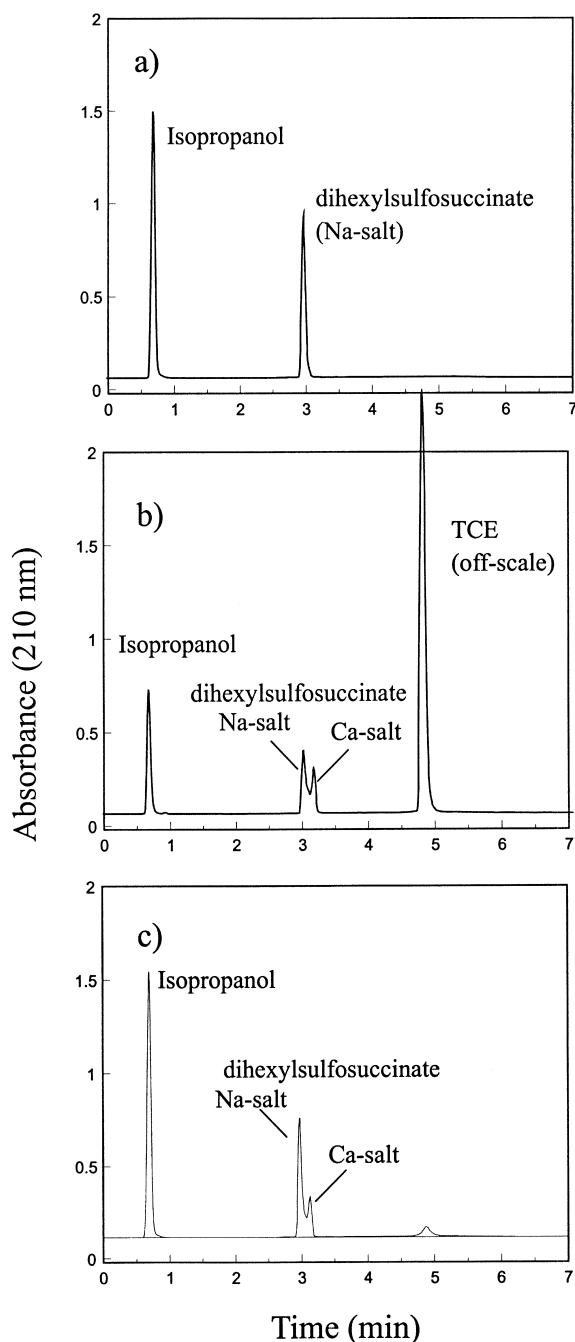


Fig. 4. Chromatograms obtained by Method 2 at 210 nm for (a) the Aerosol MA 80-I injectate that contains 38.0 g/l sodium dihexylsulfosuccinate, (b) a water sample (diluted 10-fold) that originally containing 71.7 g/l sodium dihexylsulfosuccinate and 14.1 mg/l TCE, and (c) the Aerosol MA 80-I injectate with 800 mg/l of added calcium chloride.

calcium salt in samples obtained from the physical aquifer model experiment indicated that cation exchange had occurred during the transport of the sodium dihexylsulfosuccinate through sediments contained in the physical aquifer model [10]. The confirmation of the calcium salt form of dihexylsulfosuccinate was critical in explaining the changes in surfactant phase behavior and TCE solubilization observed during physical aquifer model tests conducted with Aerosol MA 80-I and TCE [10].

Method 3 was used to analyze samples containing Aerosol MA 80-I and <150 mg/l that had been diluted as well as for groundwater samples obtained from a TCE-contaminated site that contained no surfactant. Under Method 3 conditions, the surfactants in Dowfax 8390 and Aerosol Ma 80-I elute at the void volume of the column; thus, no information is obtained by Method 3 for the surfactants tested in this study. However, Method 3 conditions offer greater resolution between TCE and its degradation products compared to those of Method 1 and 2. Chromatograms obtained by Method 3 for standards of *cis*-DCE, *trans*-DCE, 1,1-DCE, and TCE indicated that they eluted as single sharp peaks at 4.00, 4.27, 4.50, and 4.90 min, respectively (Fig. 5a). Chromatograms for groundwater samples obtained from LLNL, which did not contain surfactant, indicated the presence of TCE, *cis*-DCE and a third unidentified peak at 4.25 min, which did not match the retention time or the UV spectrum of the *trans*-DCE standard (Fig. 5b). By comparison, Method 1 conditions did not resolve the TCE and DCE isomers as well, such that the unidentified peak had the same retention time as that of *trans*-DCE. The positive identification of TCE and *cis*-DCE and the absence of *trans*-DCE in this groundwater sample was confirmed by gas chromatography/mass spectrometry (unpublished data).

The accuracy and precision of TCE determinations in water were determined by the analysis of TCE-saturated aqueous solutions at 25°C. Six replicate samples were prepared by injecting 19  $\mu$ l of TCE-saturated water into six sealed autosampler vials containing 1.88 ml of distilled water at 25°C. Single samples were not repeatedly analyzed due to the volatility of TCE. Concentrations of TCE were  $1094 \pm 23$  mg/l with an RSD of 2.1%, which was consistent with the published TCE aqueous solubility

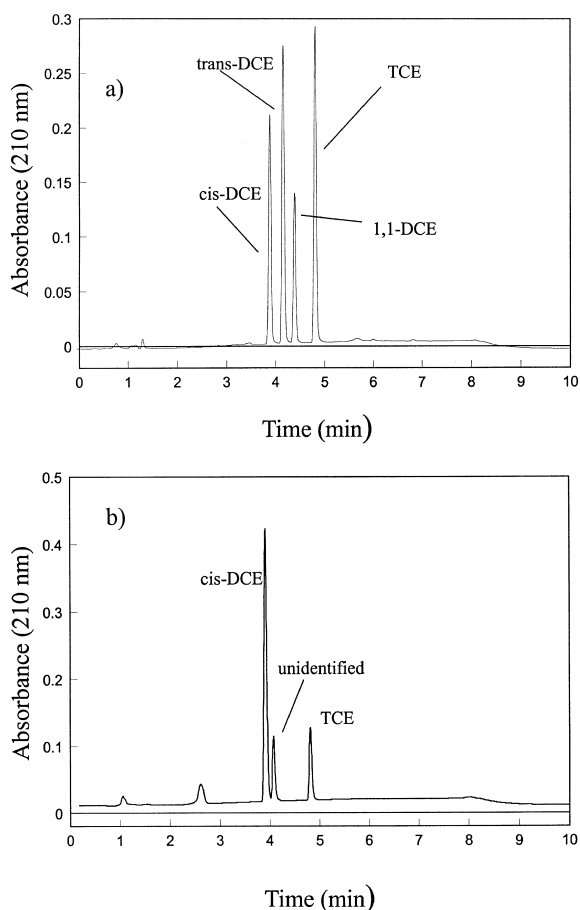


Fig. 5. Chromatograms obtained at 210 nm by Method 3 of (a) 20  $\mu$ l injection of a standard containing 21.7 mg/l *cis*-DCE, 10.5 mg/l *trans*-DCE, 18.8 mg/l 1,1-DCE, and 10.0 mg/l TCE and (b) a 10  $\mu$ l injection of a TCE-contaminated ground water sample containing 128 mg/l *cis*-DCE and 11 mg/l TCE.

at 25°C of 1100 mg/l [24]. The detection limits of Method 3 for TCE and the DCE isomers were identical to that of Method 1 with 0.05 mg/l for TCE and *trans*-DCE and 0.13 mg/l *cis*-DCE and 1,1-DCE.

#### 4. Conclusions

Surfactant-enhanced aquifer remediation requires the analysis of groundwater samples that contain both non-volatile (aromatic or aliphatic) surfactant and volatile analytes such as TCE. Dowfax (alkyl

diphenyloxide disulfonate) surfactants and TCE were simultaneously determined in a single reversed-phase HPLC analysis (Method 1). The addition of NaCl to the mobile phase (Method 2) enabled the detection and quantification of dihexylsulfosuccinate surfactants in Aerosol MA 80-I. Resolution between the sodium and calcium forms of dihexylsulfosuccinate was detected and indicated that injected surfactant solutions had undergone cation exchange within the physical aquifer models packed with sediment. Because Aerosol MA 80-I solubilizes very large quantities of TCE and because the aliphatic dihexylsulfosuccinate surfactants absorb relatively weakly at 210 nm, samples containing Aerosol MA 80-I and >150 mg/l were diluted and analyzed by a simple linear gradient (Method 3) to determine TCE. Groundwater samples containing only TCE and its reductive-dechlorination degradation products also were quantified by Method 3.

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