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ANALYSIS OF SOIL AND WATER FOR TATB CONTENT

Clifford L. Schaffer

NOVEMBER 1992

Process Development
Manufacturing Development
(Waste Minimization)

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ANALYSIS OF SOIL AND WATER FOR TATB CONTENT

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ABSTRACT

A reverse-phase liquid chromatography (HPLC) method was developed for the analysis of TATB in soil samples. The soil samples were extracted with dimethylformamide (DMF). The extract was analyzed to determine the TATB content in the soil. The detection limit using this procedure was 2 parts/million (ppm) for TATB in the soil.

An organic free sample of water was saturated with TATB. The water was filtered through a 0.2- μ filter, then injected into both a reverse-phase and normal-phase liquid chromatograph system. No peaks were detected. Therefore, the solubility of TATB in water is less than the detection limits of the chromatograph methods.

INTRODUCTION

Recent concerns over the contamination of soil and water with explosives associated with Department of Energy and Department of Defense sites motivated the development of analytical procedures for explosives in these matrices. A normal-phase HPLC method for the assay of TATB has been reported.(1) Most chemical laboratories, however, use reverse-phase HPLC methods and lack the columns and solvents necessary for the normal-phase TATB assay required by Reference 1. We report herein a reverse-phase HPLC method for determining the TATB content in soil and water samples.

EXPERIMENTAL

SAMPLE PREPARATION

Foreign particles such as rocks and grass were removed from the soil samples before extraction. The soil samples were subsequently pulverized with a mortar and pestle. The soil samples were weighed and dried in an oven to remove volatile components. This drying serves two purposes: (1) to determine the parts/million (ppm) content of TATB in the sample on a dry-weight basis and (2) to allow the extraction solvent, dimethylformamide (DMF), to dissolve the TATB from the soil. Our experience indicated that TATB from water-wet soil samples with DMF cannot be accomplished because of the presence of water. Though TATB is sparingly soluble in most solvents (including water), TATB is somewhat more soluble in pure DMF at a concentration of 5 mg in 200 ml. After drying, 25-g soil samples were weighed into 250-ml bottles. The samples were subsequently suspended in 100 ml of DMF. The sample bottles were placed in an 80°C bath, and the suspended solids were sonicated for 2 hours. After removal from the bath, the samples cooled to ambient temperature. During cooling, the suspended solids settled to the bottom of the bottle. The DMF supernatant was analyzed for TATB content by HPLC.

CHROMATOGRAPHY

Two liquid chromatographs were used for this experiment. The instruments are Laboratory Data Control (LDC) liquid chromatographs consisting of two Constametric pumps, a control module with integrator and printer/plotter, a variable-wavelength ultraviolet (UV) detector, a column temperature controller, and an autosampler fitted with a 20- μ l injection loop.

1. C. L. Schaffer and W. T. Quinlin, HPLC Assay Method for TATB, MHSMP-85-62, Mason & Hanger - Silas Mason Co., Inc., Pantex Plant, Amarillo, Texas (December 1985).

One instrument was modified for the normal-phase HPLC system previously reported.(1) A typical chromatogram is shown in Figure 1. The second instrument was fitted with a reverse-phase HPLC system. Reverse-phase separations were performed with a 4.6-mm x 25-cm Alltech Hypersil C-18 column (5- μ m mean particle size). The eluent was 75% water buffered with 50 μ M phosphoric acid (pH adjusted to 3.0 using triethylamine) and a 25% mixture of 57.5% DMF/42.5% acetonitrile. Eluent flow rate was 2 ml/minute. A chromatogram under these conditions is shown in Figure 2. The adjustment of the UV detector to 350 nm on each chromatograph provided optimum sensitivity. A column temperature on the normal-phase system was 40°C, and the reverse-phase column temperature was 23°C.

While developing the reverse-phase system, we searched for an eluent that separated TATB from its impurities without having to control the eluent's pH. Though we found such a system, we were plagued with peak tailing. Subsequently we added the phosphoric acid/triethylamine buffer to the water in order to control the pH. After experimenting with several pH levels, we found that buffering the eluent to a pH of 3.0 optimized peak symmetry.

Reference samples were prepared by dissolving 2.5 mg of TATB in 100 ml of DMF. Reference samples and soil samples were alternately placed into the autosampler to minimize experimental error with sample injection.

To establish the accuracy of the experimental technique, spiked samples of 50 g of soil containing TATB were created by adding 1 mg of TATB to virgin soil. Five other soil samples of 50-g mass, known to contain TATB, were selected for analysis.

Column:	25 cm x 4.6 mm 10- μ Alltech NH ₂
Carrier:	55% heptane/43.5% toluene/1.5% DMF
Flow:	2 ml/minute
UV Detector:	350 nm
Sample:	TATB
Injection Solvent:	DMF

Figure 1. Normal-Phase TATB Chromatogram

Column: 25 cm x 4.6 mm 10- μ Alltech C-18
Carrier: 75% water buffered with 50 μ M phosphoric acid (pH adjusted to 3.0 using triethylamine) and a 25% mixture of 57.5% DMF/42.5% acetonitrile
Flow: 2 ml/minute
UV Detector: 350 nm
Sample: TATB
Injection Solvent: DMF

Figure 2. Reverse-Phase TATB Chromatogram

RESULTS AND DISCUSSION

The solubility of TATB in any solvent, especially water, is very small. TATB, however, is soluble in DMF at concentrations of approximately 25 ppm (ambient temperature). To judge TATB's solubility in water, we suspended TATB in a 25-ml portion of clean water. To remove undissolved TATB crystals, the suspension sample was filtered through a 0.2- μ filter. Evaporating the filtrate reduced the sample to a dry residue. The sample residue, dissolved in DMF, was submitted for TATB analysis through both the normal- and reverse-phase systems. No TATB peak was detected with either system; therefore, the concentration of TATB in TATB-saturated water is so low that we cannot detect TATB directly from water with either chromatographic system.

The supernatant from a TATB-spiked soil sample was injected into the two chromatograph systems. Based on this sample, the TATB recovery exhibited by the normal-phase system was 98.24%, as compared to a 99.21% recovery on the reverse-phase system. The precision of the two techniques were comparable (a 95% confidence level of 0.014% for the normal-phase system and a 95% confidence level of 0.017% for the reverse-phase system). We feel the accuracy and precision of either technique justifies their use.

Five TATB-contaminated soil samples were analyzed using both chromatographic systems. The analyses are shown in Table I with 95% confidence level of each.

Table I. Soil Samples Analyses Data				
Sample No.	TATB (ppm)		95% Confidence Level (ppm)	
	Normal Phase	Reverse Phase	Normal Phase	Reverse Phase
1	54.05	56.41	0.51	4.20
2	291.65	292.77	1.40	2.23
3	11.12	10.21	0.15	1.39
4	185.75	189.85	3.35	2.77
5	39.65	39.85	1.10	0.50

To determine the method detection limits of the two chromatographic systems, a reference sample containing 2.4582 mg of TATB was dissolved in 100 ml of DMF. This sample was injected into both chromatographic systems 15 times. Data from the normal-phase system showed a standard deviation of 0.0179 mg, and the reverse-phase system data showed a standard deviation of 0.0488 mg. Using these standard deviations and assuming a 25-g soil sample mass, method detection limits (99% confidence that the value is not zero)(2) of 2 ppm for the normal-phase system and 5.8 ppm for the reverse-phase system were calculated.

2. The Analysis of Polychlorinated Biphenyls In Transformer Fluid and Waste Oils (Draft), Environmental Monitoring and Support Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268 (June 24, 1980).

If a sample shows to be saturated in TATB, then the sample must be reextracted with DMF and analyzed a second time. This would be repeated until the extracted sample is below the saturation point of TATB in DMF. The data from the extracted samples would be added to find the ppm of the soil sample.

CONCLUSIONS

A reverse-phase HPLC analysis was developed to assay TATB in soil and water samples. Comparison of the new method with the previously developed technique(1) showed that the two are comparable for soil analysis. Analysis of spiked samples revealed the normal-phase method showed somewhat better precision, but the reverse-phase method showed better accuracy. The solubility of TATB in water is, however, too low for these procedures to be useful in the analysis of TATB-contaminated water samples.

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