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ASSAY OF TATB BY HPLC

Clifford L. Schaffer

DEVELOPMENT DIVISION

DECEMBER 1978

Process Development

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ABSTRACT

An assay method for 1,3,5-triamino-2,4,6-trinitrobenzene (TATB) by high pressure liquid chromatography is being developed using a carrier solvent of 1.5% DMF, 43% toluene and 55.5% heptane and a μBondapak NH_2 column. TATB was separated from all known impurities using this system.

DISCUSSION

TATB is an explosive that possesses excellent thermal stability and sensitivity characteristics. However, the properties of any explosive are dependent on the purity of the material. Due to the low solubility of TATB in most solvents no satisfactory assay technique for TATB has previously been reported. The molar absorbance of several lots of TATB in DMSO was observed to have a maximum absorbance at 355 nm. With this information a study to find an assay method for TATB by HPLC was initiated.

A mini-pump (Milton Roy Co.) and damper (Laboratory Data Control) with an 837 variable wavelength spectrophotometer (DuPont Instruments) were used. Many solvent systems were tried along with various column lengths and column packing. It was found that TATB would pass through a DuPont ETH column using dimethyl sulfoxide as an injection solvent and tetrahydrofuran as a carrier solvent. The chromatograms of impurities from TATB (Table I) showed no separation from TATB. However, separation of the TATB from its impurities was obtained using two each 1 m ETH columns and a carrier system of 60% heptane and 40% toluene. DMSO as the injection solvent was found to be immiscible with the carrier solution. When the carrier solution was changed so the DMSO was miscible, the polarity was increased to the point that again no separation was obtained. Heating the column did not aid the miscibility or separation.

The column was changed to μBondapak NH_2 (Waters Associates) and N,N-dimethylformamide (DMF) was utilized as the injection solvent. Initially the carrier solvent was the same as the previous system. The DMF was found to be miscible with the carrier solvent. Better separation was obtained with this system. The TATB was found to have a retention time different from the various impurities.

Table I. Impurities Found in TATB

(1)*	1,2,3,5-Tetrachloro-4,6-Dinitrobenzene
(2)	1,3,5-Trichloro-2,4,6-Trinitrobenzene
(3)	1,3,5-Trichloro-2,4-Dinitrobenzene
(4)	2,6-Dinitro-3,4,5-Trichloroaniline
(5)	2,4-Dinitro-3,5,6-Trichloroaniline
(6)	3,5-Dinitro-2,4,6-Trichloroaniline
(8)	1,3-Diamino-5,6-Dichloro-2,4-Dinitrobenzene
(10)	1-Chloro-3,5-Diamino-2,6-Dinitrobenzene
(11)	1-Chloro-3,5-Dinitro-2,4,6-Triaminobenzene
(12)	1,3-Dinitro-2,4,6-Triaminobenzene

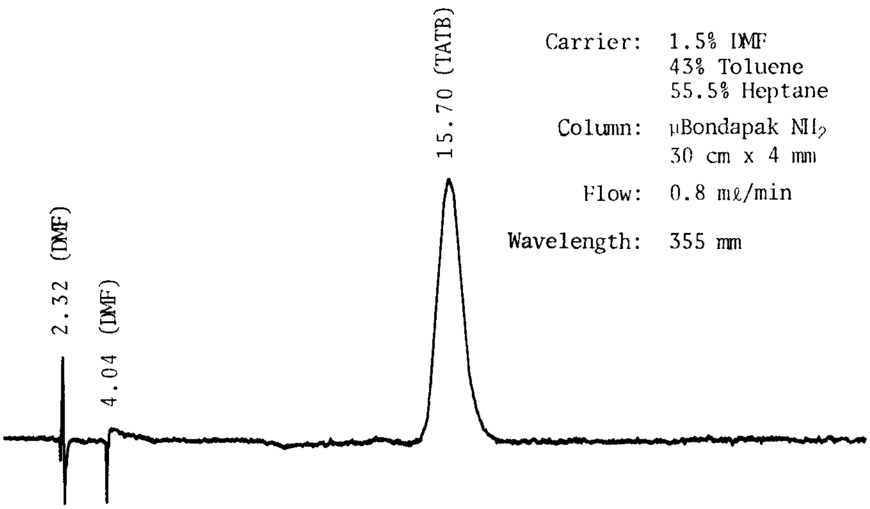
**Numbering system from Yasuda, S. K., J. Chromatogr., 71, 481 (1972).*

The carrier solvent was changed to 1.5% DMF, 43% toluene and 55.5% heptane to optimize the resolution. A flow of 0.8 mℓ per minute with 600 psi inlet pressure on the column was used. The concentration of 10 mg of TATB in 250 mℓ of DMF was used for sample preparation. Twenty μℓ were injected using an injection valve. The peaks were integrated on a 3380 A Hewlett Packard integrator. An internal standard was added to each sample for ratio comparison. The TATB used in this work had previously been determined to be the purest TATB synthesized at Pantex. Two grams of this TATB were recrystallized in boiling DMF. A chromatograph of this TATB is shown in Fig. 1. Fig. 2 shows a chromatograph of a composite sample made of known impurities. The concentration of each impurity peak is approximately 5 times that of the TATB peak.

CONCLUSION

The separation achieved using a carrier solvent of 1.5% DMF, 43% toluene and 55.5% heptane and a μBondapak NH_2 column should be sufficient for an assay method for TATB.

The precision and accuracy of this method are presently being determined.



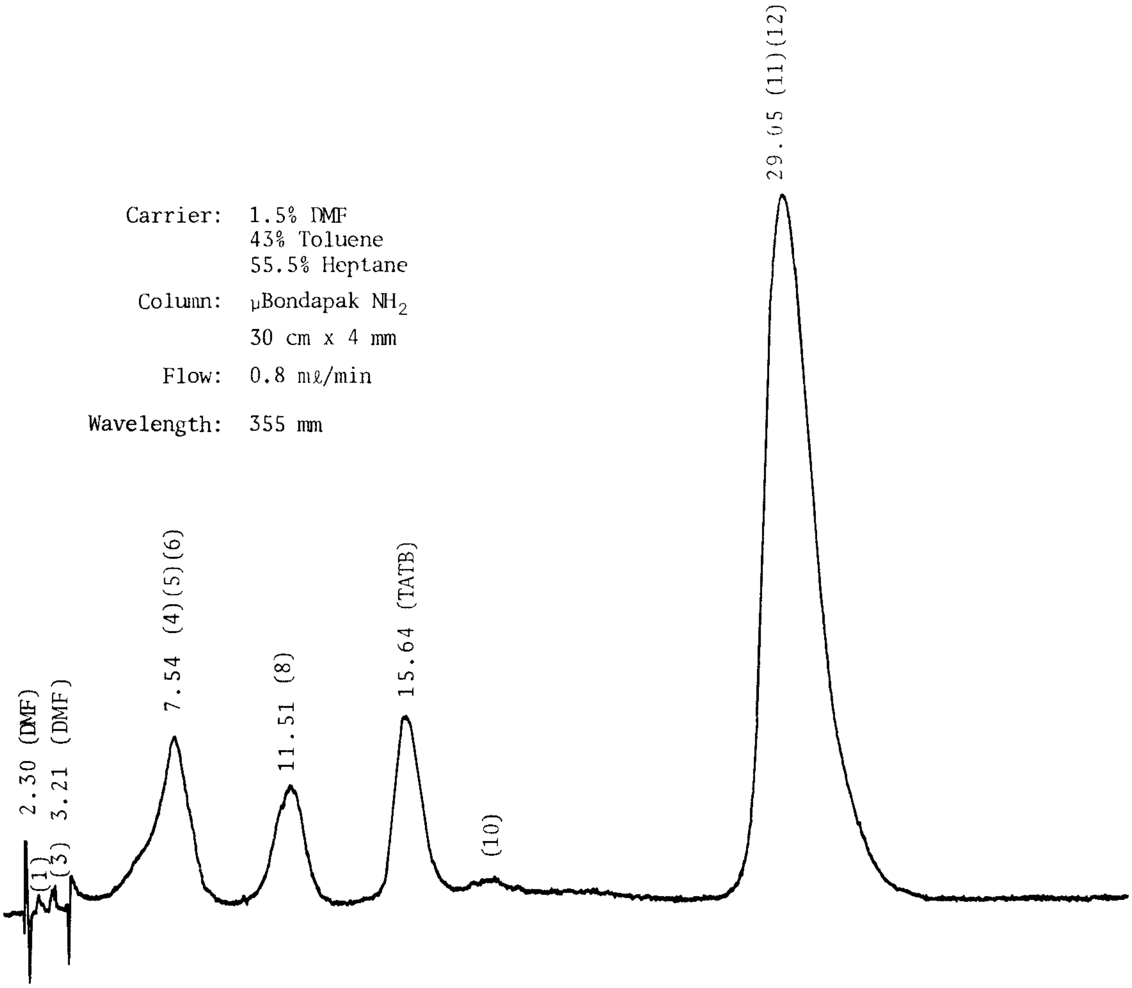
Carrier: 1.5% DMF
43% Toluene
55.5% Heptane

Column: μBondapak NH_2
30 cm x 4 mm

Flow: 0.8 mℓ/min

Wavelength: 355 mm

Fig. 1. Chromatograph of Recrystallized TATB



Carrier: 1.5% DMF
43% Toluene
55.5% Heptane

Column: μBondapak NH_2
30 cm x 4 mm

Flow: 0.8 mℓ/min

Wavelength: 355 mm

Fig. 2. Chromatograph of TATB and its Impurities

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