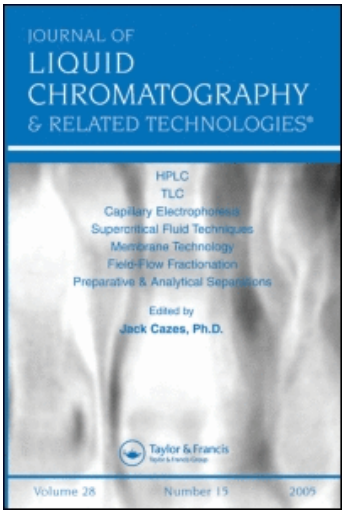

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LIQUID CHROMATOGRAPHIC ANALYSIS OF PENTAZOCINE AND TRIPELENNAMINE IN COMBINATION

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ABSTRACT

A reverse phase and normal phase liquid chromatographic procedure is described for the separation of pentazocine and tripelennamine. The isocratic methods use dual wavelength detection at 254, 280, and 313 nm.

INTRODUCTION

Pentazocine and tripelennamine is a combination of drugs which has been encountered in many parts of the United States as an intravenous substitute for heroin. Pentazocine, the active drug ingredient in Talwin tablets, is a potent analgesic with comparisons of analgesic properties made to codeine (1). Tripelennamine is an ethylenediamine class of antihistamine producing somnolence in a fair proportion of patients (2). Recently, tripelennamine has been encountered in counterfeit Quaalude preparations.

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The combined intravenous abuse of pentazocine and tripelennamine is referred to on the streets as "T's and Blue's" or "T's and B's." The "T's" is derived from the trade name of the 50 mg Talwin tablet and "Blue's" from the blue color of the PBZ or tripelennamine tablet. As early as 1973 in the state of Alabama Talwin tablets were documented as an intravenous drug of abuse when several deaths were documented as the result of pulmonary complications due to binder in the tablets. The combined abuse of pentazocine and tripelennamine apparently began about 1977, mainly in Chicago and St. Louis (3,4), and then spread throughout the Midwest (5).

Numerous items of drug paraphernalia suspected of containing pentazocine and tripelennamine have been submitted to this agency for analysis. High pressure liquid chromatography (HPLC) has proven to be an effective screening method for the presence of the two drugs. Reverse phase HPLC is a useful technique for screening paraphernalia when the suspected drugs are dissolved in an aqueous medium for injection. Thin layer chromatography (6) and gas chromatography/mass spectrometry (7) have been used for the simultaneous detection of pentazocine and tripelennamine. In a recent publication Monforte et al. (8) described a

procedure for the combined detection of pentazocine and tripelennamine by thin layer chromatography (TLC), gas chromatography (GC), ultraviolet spectrophotometry (UV), and spectrofluorometry in biological specimens. However, HPLC data was not included. This paper identifies the HPLC methods which have proven effective in identifying pentazocine and tripelennamine in combination.

EXPERIMENTAL

Reagents and Chemicals

Pentazocine hydrochloride and tripelennamine hydrochloride were obtained from their respective manufacturers and used without further purification. All solvents were HPLC grade except diethylamine which was reagent grade and were purchased from Fisher Scientific Co., Fair Lawn, NJ.

Instrumentation

The liquid chromatograph consisted of a Waters Associates (Milford, MA) Model 6000 A pump, Model U6K injector, Model 440 UV detector with dual wavelength accessory capable of operation at 254, 280, and 313 nm, and a Houston Instrument (Austin, TX) OmniScribe dual pen recorder.

Chromatographic Procedures

Reverse phase separations were carried out on a 30 cm x 3.9 mm id μBondapak C_{18} column (Waters Associates) at ambient temperature. The analytical column was preceded by a 7 cm x 2.1 mm id guard column dry packed with CO:Pell ODS (Whatman Inc., Clifton, NJ). Powdered samples (10 mg each) of the two drugs were dissolved in HPLC grade methanol and chromatographed using a mobile phase (solvent system A) of pH 3.0 phosphate buffer - HPLC grade methanol - HPLC grade acetonitrile (10+3+1). The pH 3.0 phosphate buffer was prepared by mixing 9.2 g monobasic sodium phosphate (NaH_2PO_4) in 1 L double distilled water and adjusting the pH to 3.0 with 2N H_3PO_4. The mobile phase flow rate was 2.0 mL/min and the detector was operated at 0.2 AUFS. Absorbance ratios were calculated from the average peak height measurements of a minimum of 3 injections for each drug tested.

Normal phase separations were carried out using a 30 cm x 3.9 mm id μPorasil column (Waters Associates) at ambient temperature. The analytical column was preceded by a 7 cm x 2.1 mm id guard column dry packed with HC Pellosil (Whatman Inc., Clifton, NJ). The mobile phase (solvent system B) was a mixture of cyclohexane-methylene chloride-methanol-diethylamine

(450+40+10+0.5). The mobile phase flow rate was 1.5 mL/min and the detector was operated at 0.2 AUFS. Absorbance ratios were calculated in the same manner as described above.

RESULTS AND DISCUSSION

The UV absorption spectra of pentazocine and tripelennamine have proven useful in screening various samples suspected of containing this combination of drugs. Pentazocine exhibits absorption maxima at approximately 280 and 222 nm in aqueous acidic medium while tripelennamine absorbs at approximately 314 and 240 nm. Monforte et al. (8) presented a detailed description of the ultraviolet spectra of these drugs when present in combination in an earlier publication. The absorption properties of these two drugs make them likely candidates for separation by HPLC with ultraviolet detection.

The chromatographic properties of pentazocine and tripelennamine were examined in reverse phase and normal phase systems. The reverse phase separation was maximized using an isocratic solvent system of pH 3.0 phosphate buffer-methanol-acetonitrile (10+3+1). The separation from a representative sample which was removed from a bottle cap is shown in Figure 1. The

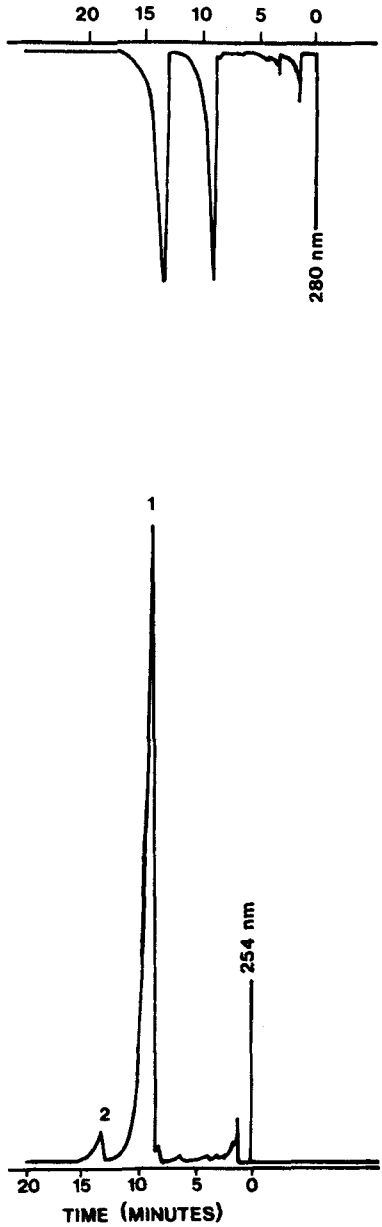

FIGURE 1

Reverse phase liquid chromatogram of (1) tripelennamine and (2) pentazocine at 254 and 280 nm. Mobile Phase, Solvent System A.

highly acidic mobile phase produces good resolution and sufficient solute retention. Under these conditions pentazocine (pKa 8.76) and tripelennamine (pKa 4.2, 8.71) should exist exclusively as the protonated amine.

The addition of acetonitrile to the mobile phase rather than using pH 3.0 phosphate buffer-methanol (5+2) decreased the elution time of tripelennamine and pentazocine approximately 3 min and 5 min respectively as well as improved the symmetry of the peaks. Figure 2 illustrates the improved peak symmetry and retention times using a combination of methanol and acetonitrile in the mobile phase.

The normal phase separation of pentazocine and tripelennamine was accomplished using an isocratic solvent system of cyclohexane-methylene chloride-methanol-diethylamine. Figure 3 illustrates the separation of a representative sample removed from a bottle cap.

Further proof of the identity of pentazocine and tripelennamine may be obtained from a ratio of absorbances at various wavelengths. Baker et al. (9) have used the A_{254}/A_{280} ratio to determine the identity of drugs having similar elution characteristics in an HPLC system. However, the interlaboratory use of

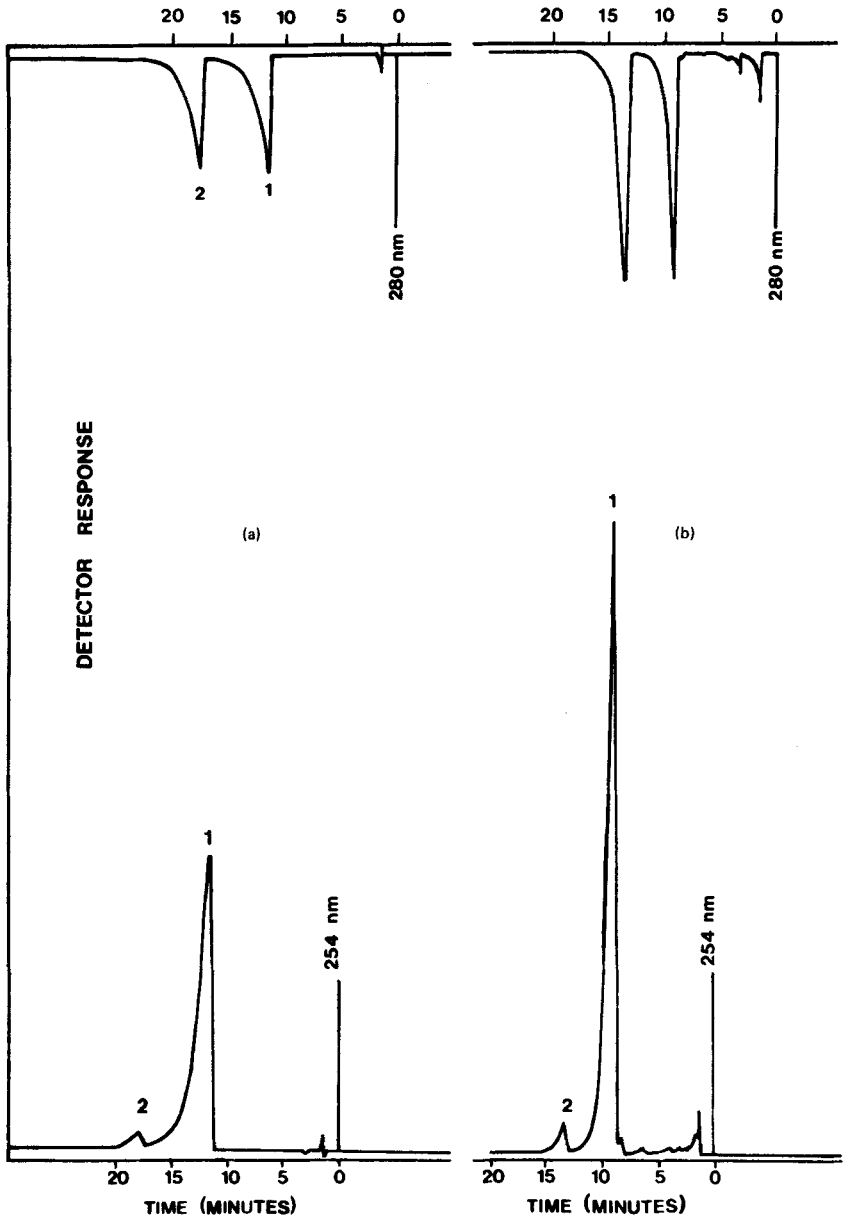

FIGURE 2

Reverse phase liquid chromatograms of (1) tripelennamine and (2) pentazocine at 254 and 280 nm. A, pH 3.0 phosphate buffer - methanol (5+2). B, pH 3.0 phosphate buffer - methanol - acetonitrile (10+3+1).

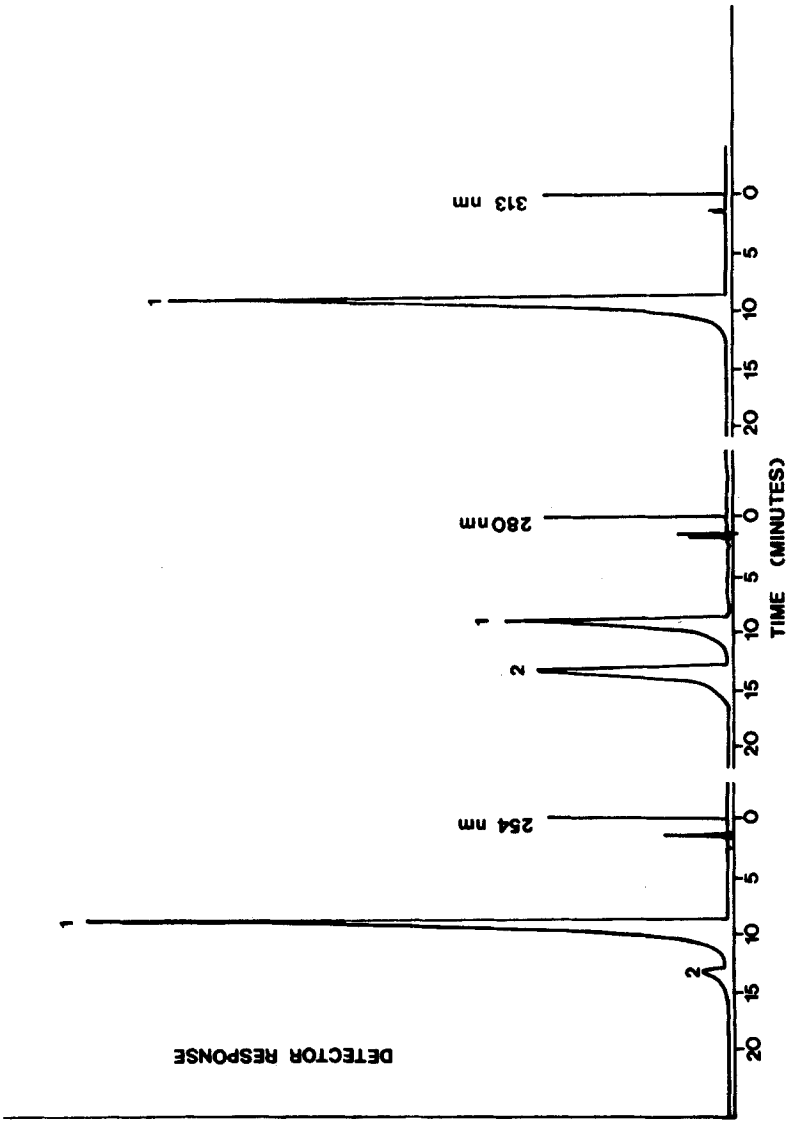

FIGURE 3

Reverse phase liquid chromatograms of (1) tripelennamine and (2) pentazocine illustrating the differences in absorbance at 313, 280, and 254 nm. Mobile Phase System A.

TABLE 1

Retention and Absorbance Data
for Pentazocine and Tripelennamine

Solvent System	Drug	Retention Time (min.)	Absorbance Ratios A_{254}/A_{280}	A_{254}/A_{313}	A_{280}/A_{313}
A	Tripelennamine	9.0	2.78	1.15	0.40
A	Pentazocine	13.6	0.15	----	----
B	Tripelennamine	3.7	14.20	4.91	0.35
B	Pentazocine	9.0	0.11	----	----

these ratios should be approached with caution. Variations may occur in the absorbance ratios because molar absorptivity and the wavelength of maximum absorbance vary with solvent composition, pH, and other factors (10). Absorbance ratios should be determined in an individual chromatographic system using reference standards. Table 1 gives retention data and absorbance ratios of pentazocine and tripelennamine which were determined in this study.

Figure 4 illustrates the variation in absorbance of the two drugs in the reverse phase system at 254, 280, and 313 nm. Since pentazocine exhibits no appreciable absorbance at 313 nm in the solvent systems investigated, this wavelength is of no value in the identification of pentazocine. A compromise wavelength which affords sufficient sensitivity for simultaneous detec-

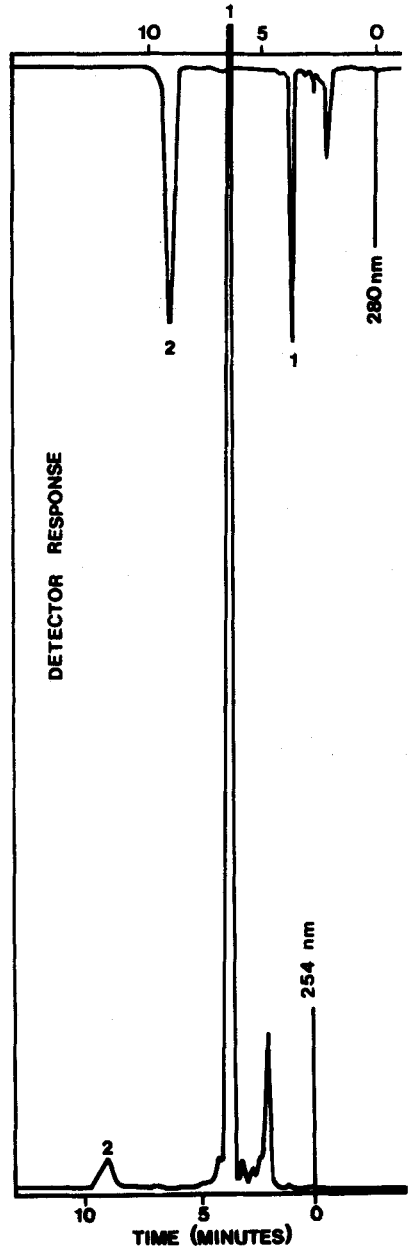

FIGURE 4

Normal phase liquid chromatogram of (1) tripelennamine and (2) pentazocine at 254 and 280 nm. Mobile Phase, Solvent System B.

tion of the two drugs utilizing a fixed wavelength detector is 280 nm. In most items of paraphernalia examined to date, the ratio of pentazocine and tripelennamine has varied. However, the majority of the items contain a greater quantity of pentazocine than tripelennamine confirming the findings of Monforte et al. (8).

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