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Note

Purity testing of N-ethylaminoacetylmesidine using thin-layer chromatography

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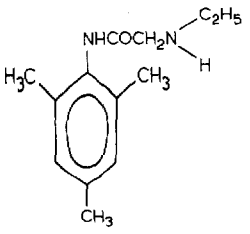
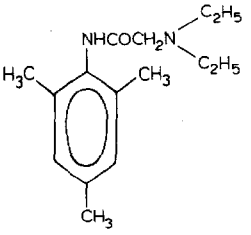
N-Ethylaminoacetylmesidine (I)[1] is a pharmacologically active metabolite of N-diethylaminoacetylmesidine (Trimecain) (II), and was recently pharmacologically tested[2]. In this paper we describe the thin-layer chromatographic (TLC) behaviour of synthetic I.



(I)



(II)

EXPERIMENTAL

Chemicals

N-Ethylaminoacetylmesidine (I) was prepared synthetically from chloroacetylmesidine and ethylamine, and had the following properties: m.p., 313–314°C (in a sealed tube); IR spectrum (KBr), 3270 cm^{-1} (NH) and 1670 cm^{-1} (CO).

Ethylammonium chloride was prepared from ethylamine (Fluka) and hydrochloric acid, and had m.p. 106–107°C.

Ninhydrin, chloroform, methanol, ethanol, acetone, *n*-butanol, and acetic acid were of analytical-reagent grade.

TLC was carried out on Kieselgel F_{254} and Kieselgel 60 thin-layer plates (Merck), using the solvent systems S_1 = chloroform–ethanol–acetone (15:10:5) and S_2 = *n*-butanol–acetic acid–water (40:10:50). The colour reagent was ninhydrin (2 g) dissolved in acetone (100 ml).

Standard solutions were prepared in methanol at concentrations of 20 mg per 10 ml for I and 10 mg per 100 ml for ethylammonium chloride.

Procedure

Volumes of 20 μl of standard solutions were applied to the plates using a Hamilton syringe, followed by drying and development with solvent system S_1 or S_2.

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TABLE I

R_F VALUES OF STANDARD SOLUTIONS OF THE TWO COMPOUNDS TESTED

Compound	R_F *values*	
	S_1	S_2
N-Ethylaminoacetylmesidine	0.50	0.57
Ethylammonium chloride	0.0	0.30

The developed chromatograms were examined under a UV lamp at 254 nm and then sprayed with ninhydrin reagent and placed briefly in an oven at 80°C. The R_F values are given in Table I.

RESULTS AND DISCUSSION

The method gives a good separation of I from ethylamine, which is the starting material in the synthesis of I. Using the TLC separation method, less than 0.25% of ethylamine in I can be detected. We have analysed several batches of I and only in one instance did we find 2.5% of ethylamine. No other spots were detectable. Our TLC findings were confirmed by non-aqueous titration[3].

Hence the described TLC method can be recommended for quality testing of synthetically prepared N-ethylaminoacetylmesidine.

ACKNOWLEDGEMENT

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