

THIN LAYER CHROMATOGRAPHIC SEPARATION OF ISOHYDRAFURAL AND METHYLURACYL IN MECHANICAL COMBINATION AND EAR DROPS

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INTRODUCTION

The combined ear drops represent an advantage over the monocomponent ones due to their polyvalent action. The thin layer chromatography (TLC) method is used to separate the active principles.

The purpose. Separation and identification of isohydrifural and methyluracil in mechanical combination and pharmaceutical form by TLC.

MATERIAL AND METHODS

Experimental series of isohydrifural (IHF), methyluracil (MU) (Sigma Aldrich, USA), chromatographic plates "Silufol", chromatography chamber, solvents, reagents according to the European Pharmacopoeia.

RESULTS

Mobile phases were used: chloroform-acetone (70:30), 1-butanol-diethyl ether-acetone (10:85:5), ethyl acetate-hexane (2:1), glacial acetic acid-water-butanol (1:1) 1:4), chloroform-methanol-glacial acetic acid (90:8:8 and 95:10:2). During the analyzes, 4 working techniques were used with the detection of spots in UV light at different wavelengths, spraying with pyridine and p-dimethylaminobenzaldehyde solutions. The mobile phase ethyl acetate-hexane (2:1) was selected for the qualitative analysis of the compounds in mechanical mixture (I) and the pharmaceutical form (II). The values of the retention factors are: I-IHF-R_f = 0,37; MU-R_f = 0,72 and II-IHF-R_f = 0,35; MU-R_f = 0,70.

Table 1. Mobile phase selection results

Mobile phase	Results	
	Izohidrafural	Metiluracil
<i>Chloroform-acetone (70:30)</i>	Yellow-orange spot with fluorescence in UV light at $\lambda = 254$ nm. R _f = 0,32	Indeterminate spots.
<i>1-Butanol-diethyl ether-acetone (10: 85: 5)</i>	Yellow-orange spot with fluorescence in UV light at $\lambda = 254$ nm. R _f = 0,32	Indeterminate spots.
<i>Glacial acetic acid - water - butanol (1: 1: 4)</i>	Indeterminate spots.	Indeterminate spots.
<i>Chloroform - methanol - glacial acetic acid (90: 8: 8)</i>	Indeterminate spots.	Red spot after spraying with soil. 1% p-dimethylaminobenzaldehyde in H ₂ SO ₄ and visualization at $\lambda = 254$ nm. R _f = 0,87
<i>Chloroform - methanol - glacial acetic acid (95: 10: 2)</i>	Pink spot when sprayed with soil. 1% p-dimethylaminobenzaldehyde in H ₂ SO ₄ . R _f = 0,78	Indeterminate spots.
<i>Ethilacetat-hexan (2:1)</i>	Red spot when sprayed with pyridine solution and UV visualization at $\lambda = 366$ nm. R _f = 0,37	Red spot when sprayed with pyridine solution and UV visualization at $\lambda = 254$ nm. R _f = 0,72

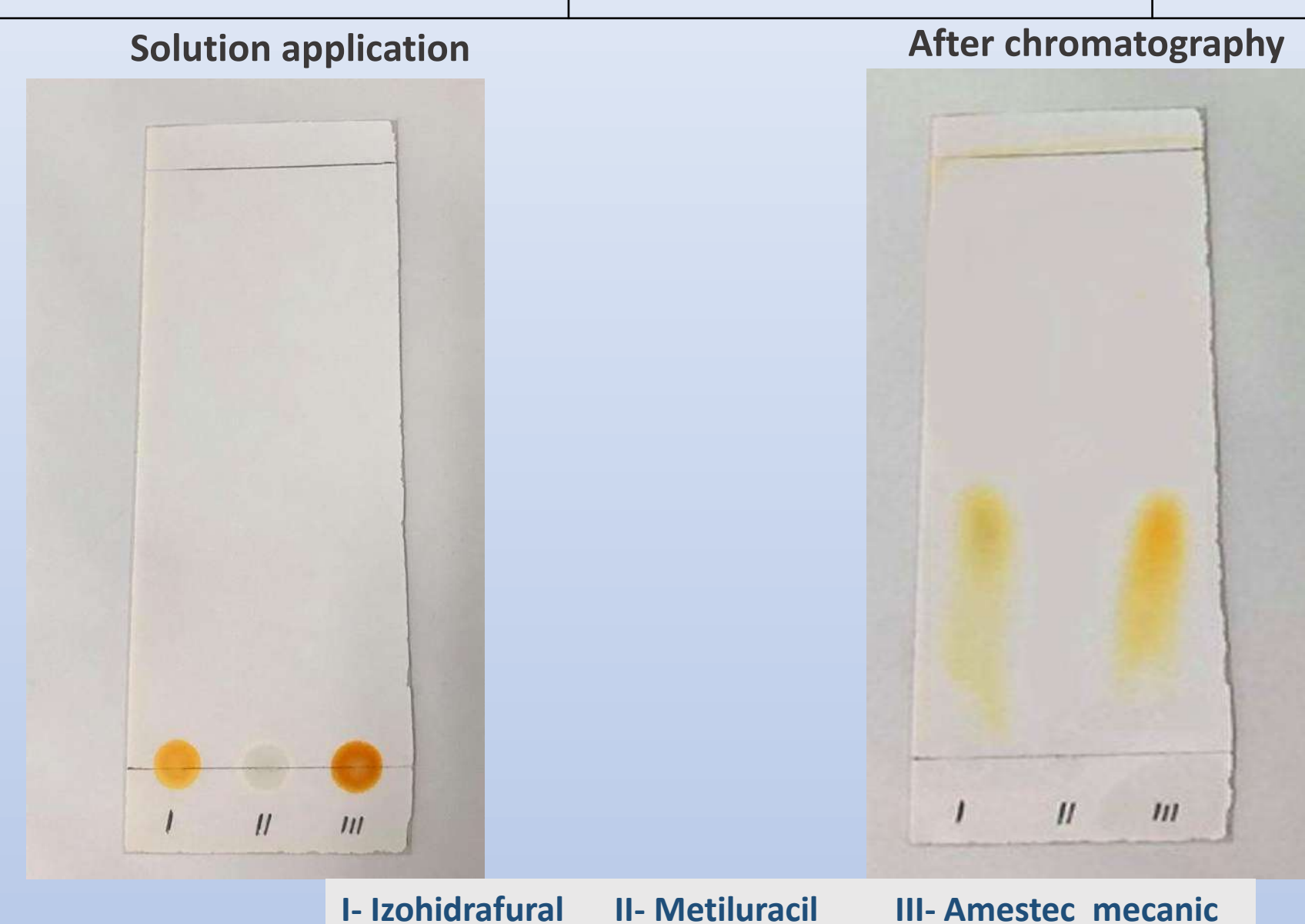
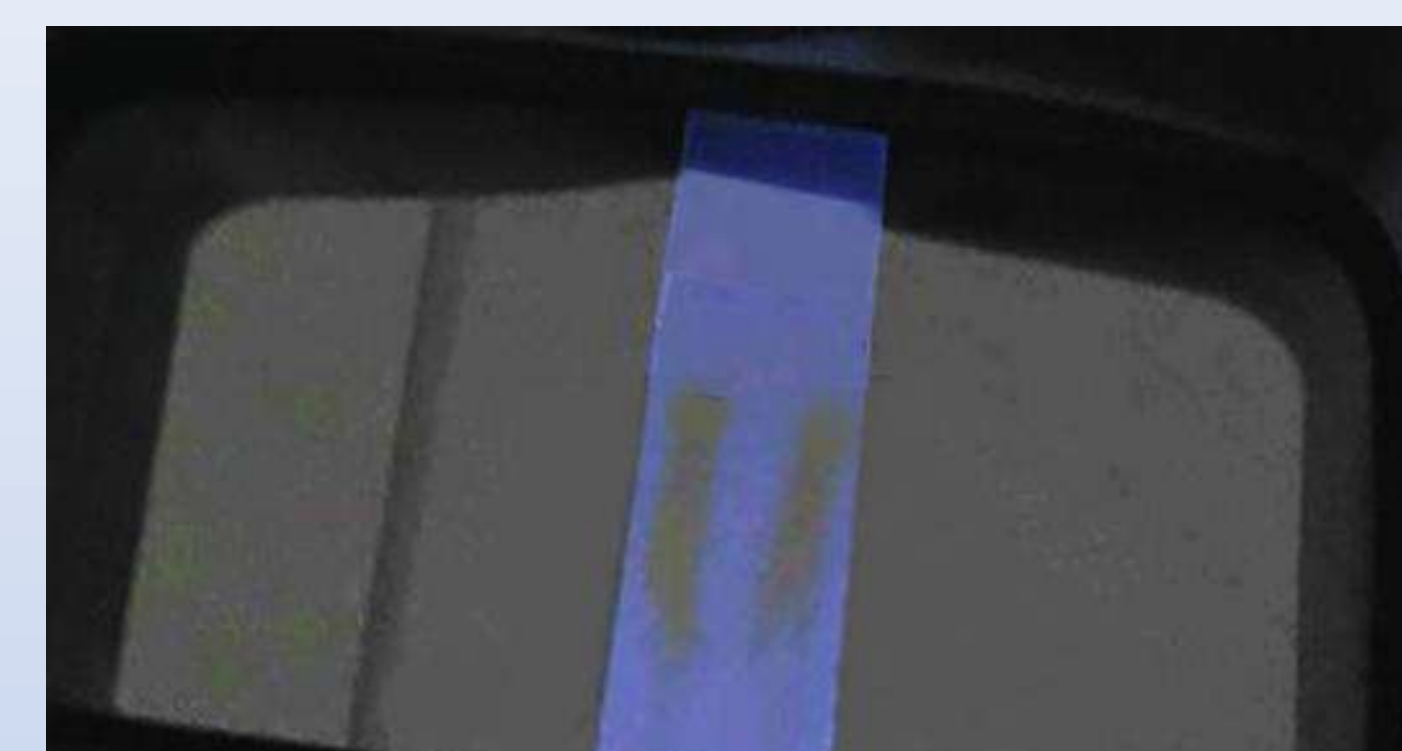


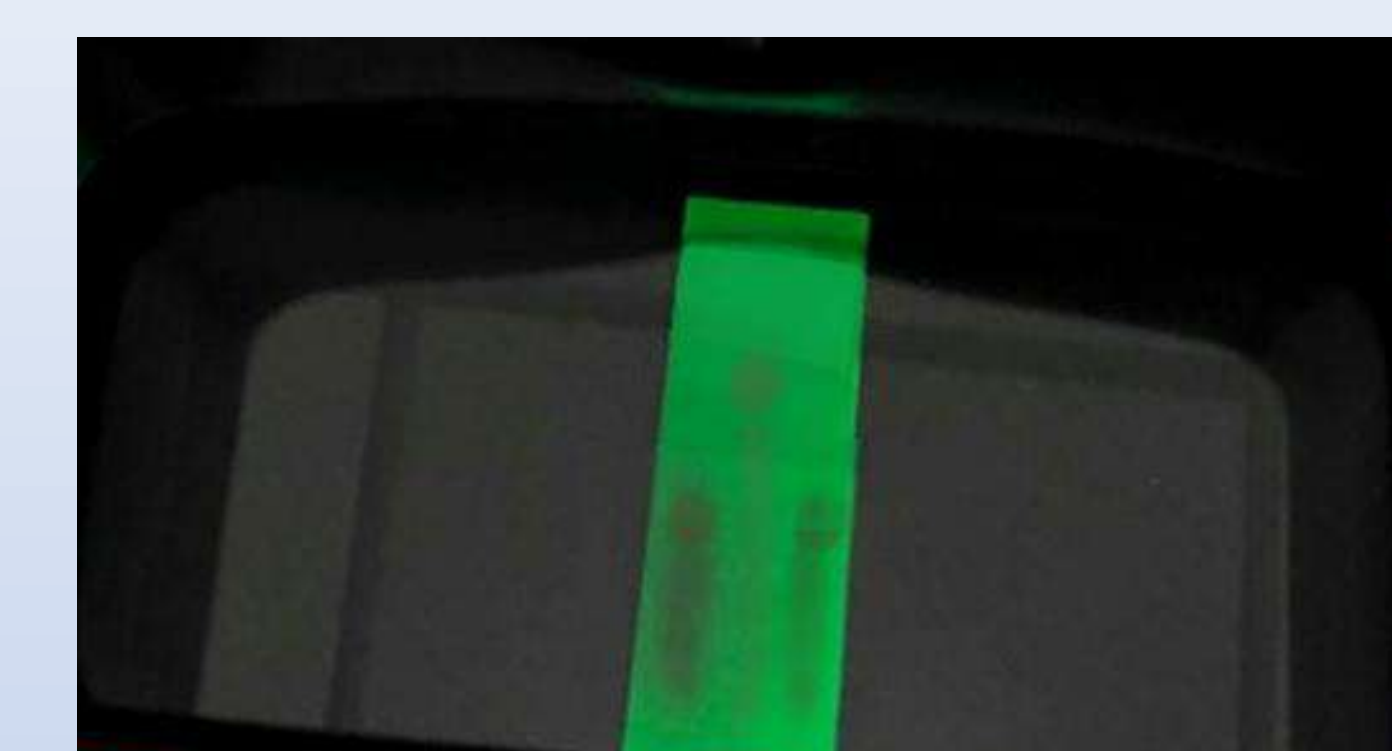
Table 2. Optimal separation on ethyl acetate-hexane (2: 1) mobile phase

IHF	Methyluracil	Mechanical mixture	Ear drops
Red spot when sprayed with pyridine solution and UV visualization at $\lambda = 366$ and 254 nm.	Pink spot when sprayed with pyridine solution and UV visualization at $\lambda = 254$ nm	IHF - red spot when sprayed with soil. pyridine and UV visualization at $\lambda = 366$ and 254 nm.	IHF - red spot when sprayed with soil. pyridine and UV visualization at $\lambda = 254$ nm
		-	R _f = 0.35
R _f = 0,37	R _f = 0,72	Methyluracil - pink spot when sprayed with soil. pyridine and UV visualization at $\lambda = 254$ nm.	Methyluracil - red spot when sprayed with soil. pyridine and UV visualization at $\lambda = 254$ nm.
		R _f = 0,72	R _f = 0,7

Viewing at $\lambda=254$ nm



Viewing at $\lambda=366$ nm



CONCLUSIONS

The elaborated working technique will be included in the quality specification of the combined ear drops for the identification of IHF and MU.

Keywords: Thin layer chromatography, isohydrifural, methyluracil.