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Note

Separation of isomeric ureas and oxazoles by thin-layer chromatography

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During the synthesis of oxazoles^{1,2} from substituted ureas and α -haloketones, it was necessary to study the separation and identification of the final product by thin-layer chromatography (TLC). In recent years, this technique has also been applied successfully to the separation of isomeric compounds using suitable solvent systems³⁻⁶. From the present investigation, it was evident that it is not only possible to separate oxazoles from corresponding ureas, but also to separate isomeric ureas and oxazoles from one another.

EXPERIMENTAL

A slurry was prepared by mixing 25 g of silica gel H (according to Stahl, Type 60; E. Merck, Darmstadt, G.F.R.) in 50 ml of distilled water. Ten well cleaned glass plates (20 × 10 cm) were coated with the adsorbent to a thickness of 250 μ m using an adjustable Desaga Model S II (Stahl-type) applicator. The spotted plates were developed in air-tight glass chambers (25 × 12 × 25 cm) that had previously been saturated with the solvent vapour and the developing time was 24-82 min, depending on the solvent used. The operating temperature was 25-30 °C and the relative humidity of the atmosphere was 60-70%. The developed chromatograms, after being dried, were sprayed with a 1% solution of *p*-dimethylaminobenzaldehyde in 2% alcoholic hydrochloric acid followed by heating at 60 °C for 1-2 min, when the substituted ureas were detected as lemon-yellow spots. The oxazoles were detected as fluorescent spots by viewing under UV light (366 nm).

RESULTS

A large number of solvent systems were tried, and the solvent systems in which there was a reasonable difference in R_f values of the isomers have been recorded in Table I. The isomeric compounds tried for separation were: *o*-, *m*- and *p*-chlorophenylurea, *o*-, *m*- and *p*-methoxyphenylurea, *o*-, *m*- and *p*-tolylurea, 2-*o*-, *m*- and *p*-chlorophenylamino-4-phenyloxazole, 2-*o*-, *m*- and *p*-methoxyphenylamino-4-phenyloxazole and 2-*o*-, *m*- and *p*-tolylamino-4-phenyloxazole.

An equimolar mixture of the isomeric compounds was chromatographed by ascending unidimensional TLC. Effective separations of all of the above compounds were possible with the solvent systems light petroleum (b.p. 60-80 °C)-diethyl ether,

TABLE I

 R_f VALUES OF ISOMERS IN DIFFERENT SOLVENT SYSTEMS

Solvent systems: A = light petroleum (b.p. 60–80 °C)–diethyl ether (1:1); B = light petroleum (b.p. 60–80 °C)–benzene (1:1); C = light petroleum (b.p. 60–80 °C)–chloroform (1:1); D = light petroleum (b.p. 60–80 °C)–methanol (1:1); E = light petroleum (b.p. 60–80 °C)–isopropanol (1:1).

Compound	R_f value				
	A	B	C	D	E
<i>o</i> -Chlorophenylurea	0.00	0.01	0.12	0.12	0.28
<i>m</i> -Chlorophenylurea	0.18	0.15	0.33	0.35	0.40
<i>p</i> -Chlorophenylurea	0.38	0.28	0.60	0.60	0.65
<i>o</i> -Tolylurea	0.02	0.03	0.02	0.02	0.22
<i>m</i> -Tolylurea	0.17	0.22	0.32	0.32	0.50
<i>p</i> -Tolylurea	0.30	0.40	0.50	0.50	0.70
<i>o</i> -Methoxyphenylurea	0.00	0.06	0.08	0.08	0.18
<i>m</i> -Methoxyphenylurea	0.35	0.28	0.40	0.40	0.48
<i>p</i> -Methoxyphenylurea	0.15	0.42	0.18	0.18	0.73
2-(<i>o</i> -Chlorophenyl)amino-4-phenyloxazole	0.50	0.12	0.28	0.58	0.22
2-(<i>m</i> -Chlorophenyl)amino-4-phenyloxazole	0.72	0.43	0.00	0.70	0.32
2-(<i>p</i> -Chlorophenyl)amino-4-phenyloxazole	0.92	0.65	0.48	0.93	0.49
2-(<i>o</i> -Tolyl)amino-4-phenyloxazole	0.52	0.12	0.00	0.32	0.24
2-(<i>m</i> -Tolyl)amino-4-phenyloxazole	0.77	0.50	0.01	0.42	0.36
2-(<i>p</i> -Tolyl)amino-4-phenyloxazole	0.89	0.28	0.18	0.68	0.52
2-(<i>o</i> -Methoxyphenyl)amino-4-phenyloxazole	0.43	0.00	0.28	0.28	0.29
2-(<i>m</i> -Methoxyphenyl)amino-4-phenyloxazole	0.60	0.28	0.42	0.58	0.39
2-(<i>p</i> -Methoxyphenyl)amino-4-phenyloxazole	0.88	0.45	0.00	0.70	0.56
Time of development (min)	40	47	37	51	68

light petroleum (b.p. 60–80 °C)–benzene, light petroleum (b.p. 60–80 °C)–methanol, light petroleum (b.p. 60–80 °C)–isopropanol and light petroleum (b.p. 60–80 °C)–chloroform, marked differences in the R_f values of the *o*-, *m*- and *p*-isomers being obtained.

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