

Furan derivatives. XXXVI. Chromatographic determination of some phenylfuran derivatives

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Chromatographic separation of substituted 5-(X-phenyl)-2-furaldehydes and 5-(X-phenyl)-2-furfuryl alcohols as intermediates of the 5-(X-phenyl)-2-furfuryl thiocyanate synthesis ($X = 4\text{-NO}_2$, 3-NO_2 , 2-NO_2 , 4-Cl , 3-Cl , 4-Br , H) on thin layers of silica gel is described.

Within the scope of the study of phenylfuran derivatives, we synthesized 2-, 3-, and 4-substituted 5-phenyl-2-furfuryl thiocyanates [1]. We investigated the problem of identity of the synthesis intermediates of substituted 5-(phenyl)-2-furfuryl thiocyanates and their isomerizations to the appropriate isothiocyanates [2].

There is no description of furan derivatives as to their chromatographic separation in the literature. Works on separation of furan derivatives by paper partition chromatography are known [3, 4]. Some nitrofur derivatives were separated by means of thin-layer chromatography [5, 6]. Chromatographic differentiation of isothiocyanates as possible products of rearrangement of thiocyanates is presented in works [7, 8].

In our work, we present the mode and the results of good differentiation of substituted 5-(X-phenyl)-2-furaldehydes, 5-(X-phenyl)-2-furfuryl alcohols, 5-(X-phenyl)-2-furfuryl thiocyanates, and 5-(X-phenyl)-2-furfuryl isothiocyanates by adsorption chromatography on thin layers of silica gel.

All developing systems used, but mainly the system *A* (see Table 1) were shown to be suitable for good differentiation of isothiocyanate from the appropriate thiocyanate. Thus the rearrangement of thiocyanates could be qualitatively followed.

The position and the nature of the substituent on the benzene ring affect the R_F value. The 4-nitrosubstituted phenylfuran derivatives had the highest R_F values, while those of 4-chlorosubstituted had the lowest R_F values in all four groups of compounds.

Experimental

The substituted 5-(X-phenyl)-2-furaldehydes were prepared according to [9–12], 5-(X-phenyl)-2-furfuryl alcohols according to [13–15], 5-(X-phenyl)-2-furfuryl thiocyanates and the appropriate isothiocyanates according to [1, 2].

The separation of these compounds was carried out on thin layers of silica gel L 5/40 (grain size 5–40 μm , Lachema, Brno) containing 10% of plaster as binding agent. The standard thickness of the layers was 0.275 mm. Simultaneously, the separation on thin layers of Silufol UV 254 (Kavalier, Sázava) was carried out. Compounds dissolved in dioxan were spotted and the plates developed with the systems: *A*. *n*-hexane–ethyl acetate (2 : 1), *B*. benzene–ethyl acetate–*n*-hexane (2 : 1 : 2), and *C*. chloroform–ethyl

Table 1
R_F values of the studied compounds

X	<i>R_F</i>			
	furylaldehydes	furfuryl alcohols	furfuryl thiocyanates	furfuryl isothiocyanates
4-NO ₂	0.75	0.64	0.84	0.93
3-NO ₂	0.62	0.47	0.80	0.92
2-NO ₂	0.55	0.39	0.69	0.82
4-Cl	0.56	0.43	0.69	0.79
3-Cl	0.71	0.55	0.82	0.92
4-Br	0.68	0.47	0.81	0.93
H	0.73	0.57	0.85	0.95

acetate (19 : 1) for 30 minutes at 20°C. Compounds were detected by spraying with 5% aqueous silver nitrate in 10% ammonia and subsequent heating for 5 minutes in an oven at 140°C.

The results of chromatographic separation of the substituted phenylfuran derivatives in the system *A* are summarized in Table 1.

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