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Thin-layer chromatography of ferrocene compounds and their benzenoid analogues

The loose analogy between the phenyl group and the ferrocenyl group is commonly employed and comparisons between the two are frequently made^{1,2}. While it might be expected that the more polar ferrocenyl group should confer a smaller R_F value upon the ferrocene-containing molecule, we were unable to find any quantitative data making a direct comparison. To that end, we investigated the R_F values of nine pairs of compounds using thin-layer chromatography. The results are discussed below.

Experimental

The ferrocene compounds were prepared in a standard fashion and their properties were in good agreement with reported values. The benzenoid compounds were reagent grade quality and were used directly from the container. The compounds were spotted on the plates in a solution of methylene chloride. All the chromatographic equipment used was from the Mallinckrodt CHROMA-KIT using SilicAR TLC-7G—a silicic acid/gypsum-binder adsorbent. The layer thickness was 0.25 mm and activation was at 92° for one hour. The elutropic solvent was benzene-acetone (30:1)*. Detection of the ferrocene compounds was done visually and detection of the benzenoid compounds was by means of iodine vapor treatment.

Results and discussion

In all cases but two the ferrocenyl analogue has a lower R_F than the phenyl compound (Table I). In one of those cases, No. 8, ferrocenylphenylcarbinol, the apparent lower polarity can probably be ascribed to intramolecular hydrogen bonding to the iron atom⁴. To ensure that no reaction had taken place on the adsorbent, a methylene chloride solution of No. 8 was allowed to stand overnight in contact with SilicAR TLC-7G, then filtered and evaporated to dryness. The infrared spectrum proved to be identical to the original, indicating that no reaction had taken place.

* Previous TLC work on ferrocene derivatives used Merck Kieselgel G and benzene or benzene-ethanol mixtures³.

The explanation for the higher R_F of No. 17 might lie in a "shielding" of much of the ferrocenyl group, producing a lower effective polarity.

TABLE I

CHROMATOGRAPHIC DATA OF FERROCENE COMPOUNDS AND THEIR BENZENOID ANALOGUES

No.	Compound*	R_F value	No.	Compound*	R_F value
1	ϕ CHO	0.75	9	ϕ CHOHCH ₃	0.29
2	Φ CHO	0.30	10	Φ CHOHCH ₃	0.25
3	ϕ CO ϕ	0.69	11	ϕ CH=CH—COCH ₃	0.32
4	Φ CO ϕ	0.30	12	Φ CH=CH—COCH ₃	0.28
5	ϕ COCH ₃	0.45	13	ϕ CH=CHCO ϕ	0.61
6	Φ COCH ₃	0.25	14	Φ CH=CHCO ϕ	0.41
7	ϕ_2 CHOH	0.30	15	Φ CH=CHCO Φ	0.26
8	Φ CH ϕ OH	0.32	16	ϕ CH ϕ CH ₂ CO ϕ	0.73
			17	Φ CH ϕ CH ₂ CO ϕ	0.77

* The symbol Φ stands for C₁₀H₉Fe.

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