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Note

Thin-layer chromatographic separation of some ferrocene alcohols

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Ferrocene derivatives are relatively stable organometallic compounds which can be analyzed by chromatographic techniques without undergoing changes in chemical structure. Early publications dealt with the separation of ferrocene derivatives from reaction mixtures by thin-layer chromatography (TLC) using silica gel^{1,2} and alumina³, and by column chromatography⁴⁻⁷. High-performance liquid chromatography was also used^{8,9} and separation of some diastereomers was achieved⁹. Systematic investigations of the application of gas-liquid chromatography in the analysis of ferrocene derivatives were carried out by Tanikawa and Arakawa¹⁰. Further work yielded the retention data for a number of ferrocene compounds on different stationary phases^{11,12}.

This paper describes the TLC separation of ferrocene alcohols and diols using silica gel and two mobile phases. Most of the investigated compounds had not previously been analyzed by chromatography.

EXPERIMENTAL

Preparation of ferrocene compounds

Compound I was prepared from ferrocenecarbaldehyde and 2-mercaptoethanol in the presence of *p*-toluenesulphonic acid as a catalyst¹³. Compounds II-XI and XIII-XVII (Table I) were obtained as described previously¹⁴⁻²⁵. Compound XII was prepared by multistep synthesis from 1,1'-ferrocenedicarboxylic acid via 1,1'-di-(hydroxymethyl)ferrocene, 1,1'-di(cyanomethyl)ferrocene and 1,1'-ferrocenediacetic acid²⁵.

Thin-layer chromatography

Separation of ferrocene compounds was performed using precoated TLC glass plates covered with silica gel 60-F₂₅₄ and having a concentrating zone (E. Merck, Darmstadt, G.F.R.). Plates were heated at 110°C for 30 min immediately before use. Two microlitres of the ferrocene compounds (10 mg/ml) in benzene were developed using the following mobile phases:

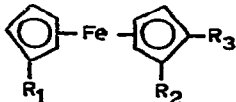
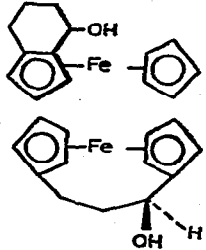
- (a) *n*-hexane-benzene-acetone (8:1:1)
- (b) benzene-chloroform-acetone (4:1:1)

Visualization of the separated substances was accomplished with 3.5% molybdato-phosphoric acid spray reagent or UV light at 254 nm.

RESULTS AND DISCUSSION

The R_F values of the examined substances in two mobile phases are given in Table I.

TABLE I
STRUCTURES AND R_F VALUES OF FERROCENE COMPOUNDS

Compound	Structural formula	Ref.	Mobile phase	
			a	b
<i>Alcohols</i>				
	$R_2 = R_3 = H,$	$R_1 =$		
		$CH(SCH_2CH_2OH)_2$	13	0.16
		$CH_2SCH_2CH_2OH$	14	0.12 0.48
		CH_2OH	15	0.14 0.52
		$CH(C_6H_5)SCH_2CH_2OH$	14	0.15 0.54
		$CH(CH_3)SCH_2CH_2OH$	14	0.19 0.59
		$CH(OH)CH_3$	16	0.21 0.61
		$CH(C_2H_5)SCH_2CH_2OH$	14	0.22 0.62
		$CH(OH)C_6H_5$	17	0.29 0.68
		$CH(OH)CF_3$	18	0.36 0.74
		$CH(OH)CH(CH_3)_2$	19	0.50 0.81
<i>Diols</i>				
	$R_2 = R_3 = CH_2OH,$	$R_1 = H$	20	0.11
	$R_3 = H,$	$R_1 = R_2 = CH_2CH_2OH$	26	0.13
		CH_2OH	21	0.16
		$CH(OH)CH_3$	22	0.28
		$CH(OH)C_6H_5$	23	0.34
<i>Other alcohols</i>				
		<i>exo</i>	24	0.32 0.70
		<i>endo</i>		0.40 0.75
			25	0.83

The R_M values in mobile phases a and b showed a linear correlation, $R_M^a = f(R_M^b)$, as presented in Fig. 1.

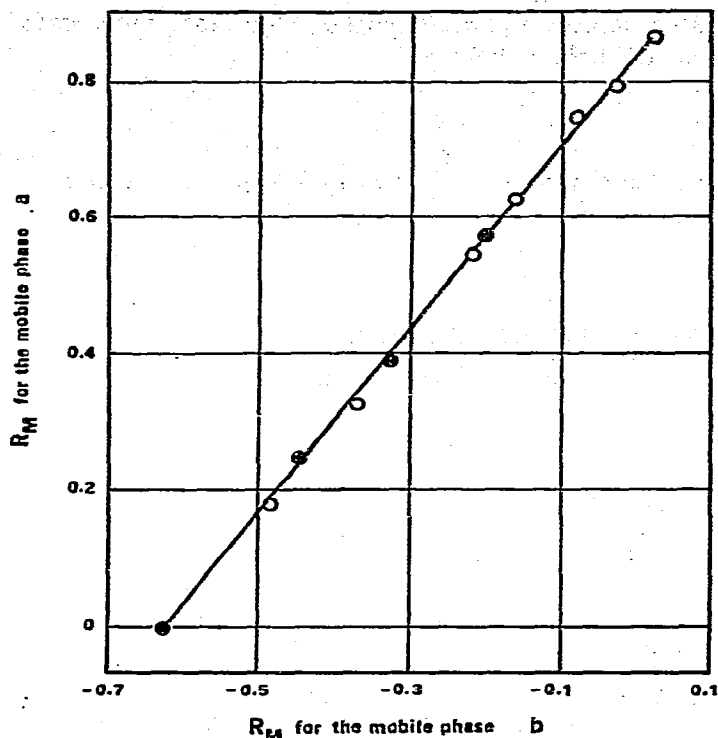


Fig. 1. R_M values for ferrocene compounds in primary alcohols (○) and secondary alcohols (●) as mobile phases.

REFERENCES

- 1 K. Schögl and A. Mohar, *Monatsh. Chem.*, 93 (1962) 861.
- 2 K. Schögl and H. Egger, *Monatsh. Chem.*, 94 (1963) 376.
- 3 R. E. Bozak and J. H. Fukuda, *J. Chromatogr.*, 26 (1967) 501.
- 4 A. Dormon and J. Decombe, *Bull. Soc. Chim. Fr.*, 1968, 3673.
- 5 P. Reich-Rohrwig and K. Schlögl, *Monatsh. Chem.*, 99 (1968) 1752.
- 6 H. Falk, O. Hofr and K. Schlögl, *Monatsh. Chem.*, 100 (1969) 624.
- 7 M. J. Nugent, R. Kummer and J. H. Richards, *J. Amer. Chem. Soc.*, 91 (1969) 6141.
- 8 M. Shigma, M. Tsunashima, H. Kono, J. Motoyama and K. Hata, *Bull. Chem. Soc. Jap.*, 43 (1970) 841.
- 9 D. T. Haworth and T. Liu, *J. Chromatogr. Sci.*, 14 (1976) 519.
- 10 K. Tanikawa and K. Arakawa, *Chem. Pharm. Bull.*, 13 (1965) 926.
- 11 O. E. Ayers, T. G. Smith, J. D. Burnett and B. W. Ponder, *Anal. Chem.*, 38 (1966) 1606.
- 12 C. Pomier and G. Guiochon, *Chromatographia*, 2 (1969) 346.
- 13 A. Ratajczak and B. Czech, unpublished results.
- 14 A. Ratajczak, B. Misterkiewicz and B. Czech, *Bull. Acad. Pol. Sci., Ser. Sci. Chim.*, 25 (1977) 27.
- 15 K. Schlögl, *Monatsh. Chem.*, 88 (1957) 601.
- 16 F. S. Arimoto and A. C. Haven, *J. Amer. Chem. Soc.*, 77 (1955) 6295.
- 17 N. Weliky and E. S. Gould, *J. Amer. Chem. Soc.*, 79 (1957) 2742.
- 18 E. B. Sokolova, G. P. Chalykh, T. A. Malikova, L. B. Sevastyanova and O. A. Nemchinova, *Zh. Obshch. Khim.*, 43 (1973) 1333.
- 19 Wu. Kuan-Li, E. B. Sokolova, I. E. Chlenov and A. D. Petrov, *Dokl. Akad. Nauk SSSR*, 137 (1961) 111.

- 20 G. Marr, B. W. Rockett and A. Rushworth, *J. Organometal. Chem.*, 16 (1970) 141.
- 21 A. Sonoda and J. Moritani, *J. Organometal. Chem.*, 26 (1971) 133.
- 22 K. Yamakawa, H. Ochi and K. Arakawa, *Chem. Pharm. Bull.*, 11 (1963) 905.
- 23 K. Yamakawa and M. Hisatome, *J. Organometal. Chem.*, 52 (1973) 407.
- 24 D. S. Trifan and R. Backsai, *Tetrahedron Lett.*, 13 (1960) 1.
- 25 M. Rosenblum, A. K. Banerjee, N. Danieli, R. W. Fish and V. Schlatter, *J. Amer. Chem. Soc.*, 85 (1963) 316.
- 26 B. Czech and L. Drobek, unpublished results.