

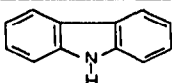
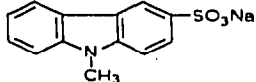
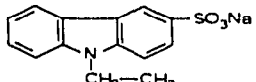
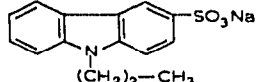
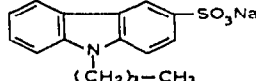
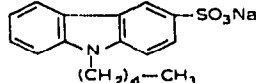
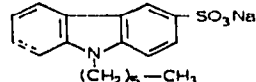
The separation of sodium salts of sulphocarbazole acids by thin-layer chromatography

Carbazole is a basic substance for organic derivatives which are important in the chemical industry. Large quantities of carbazole occur in and are obtained from coal tar. Sodium derivatives of sulphocarbazole are widely used in industry, and a rapid and simple method of identifying them during processing is desirable. Apart from that, the relationships between R_f and the molecular structure of the compounds examined is very interesting. The present paper deals with the conditions for the separation of these substances by adsorption thin-layer chromatography.

Experimental

The separation of the above substances was examined on a series of adsorbents using different solvent systems in order to establish an optimal chromatographic system. The most suitable adsorbent for the separation of these compounds was Silica Gel GF 254 (Merck) coated on glass plates. The adsorbent layers were prepared as suggested by the manufacturers¹ and the plates were activated by heating at 135° for 2 h.

TABLE I
CHROMATOGRAPHED SUBSTANCES

No.	Structural formula	Chemical name
1		Carbazole
2		Sodium salt of <i>n</i> -methyl sulphocarbazole
3		Sodium salt of <i>n</i> -ethyl sulphocarbazole
4		Sodium salt of <i>n</i> -propyl sulphocarbazole
5		Sodium salt of <i>n</i> -butyl sulphocarbazole
6		Sodium salt of <i>n</i> -pentyl sulphocarbazole
7		Sodium salt of <i>n</i> -hexyl sulphocarbazole

The solutes (concentration, 0.04 mole/l) were spotted with calibrated 4- μ l pipettes. Table I presents a list of the compounds examined and their structure formulae.

The chromatograms were developed using 1- and multi-component solvent systems by the ascending technique. The following 1-component solvents were used as the mobile phase: (1) water, class AB* according to the classification proposed by PIMENTAL AND McCLELLAN^{2,3}; (2) methanol, class AB; (3) propanol, class AB; (4) acetone, class B; (5) chloroform, class A; (6) trichloroethylene, class A; (7) benzene, class N; (8) toluene, class N; (9) carbon tetrachloride, class N.

Ethyl acetate-methanol-formic acid-pyridine (80:10:10:10) and (75:7.5:7.5:10), ethyl acetate-methanol-formic acid-piperidine (80:10:10:10) and ethyl acetate-methanol-formic acid-morpholine (80:10:10:10) were used in multi-component solvent systems. All the solvents were dried with silica gel⁴, heated at 300°. The results are presented as chromatographic spectra for 1- and multi-component solvent systems.

Results

Separation by adsorption TLC depends on the type of substance used, on the solvent or solvent system, and especially on the adsorbent because of the importance of the differentiation of strengths involved in the chromatographic system consisting of adsorbent-separated substance-solvent.

Fig. 1 presents the results of the separation of substances using a 1-component mobile phase.

As shown no separation of the chromatographed substances takes place. The highest R_F values are obtained for the solvents of class AB according to EWELL *et al.*² and PIMENTAL AND McCLELLAN^{2,3}. The lowest R_F values are obtained using the mobile phase of classes A, B and N. The course of the carbazole spectrum deviates from that of its derivative, which can probably be explained by the lack of a steric effect in this substance.

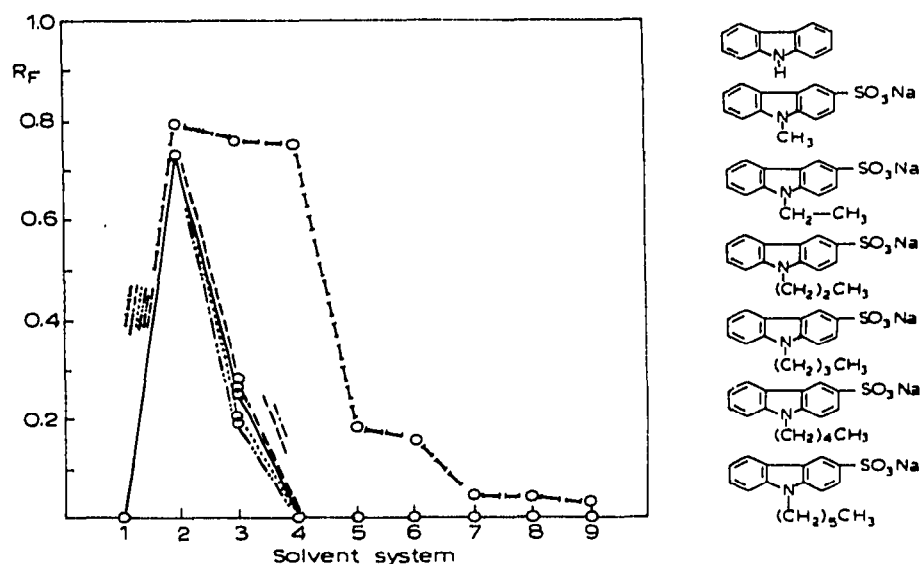


Fig. 1. Chromatographic spectra of carbazole and derivatives of sulphocarbazole obtained using a 1-component mobile phase: 1 = water; 2 = methanol; 3 = propanol; 4 = acetone; 5 = chloroform; 6 = trichloroethylene; 7 = benzene; 8 = toluene; 9 = carbon tetrachloride.

Sodium derivatives of sulphocarbazole differ only by the quantity of CH_2 -groups; all the substances have active SO_3Na -groups and a nitrogen atom and can react with OH-groups on the silica gel surface by forming a hydrogen bond of a different energy. Apart from these reactions, others can occur which are typical of this kind of adsorption (specific or nonspecific interaction⁵⁻⁸).

No strong interactions from the 1-component mobile phases used are observed due to the strong adsorption of the substances on the silica gel surface and, consequently, to the lack of more differentiated adsorption capacities, probably affected by the formation of hydrogen bonds between a nitrogen atom and OH-groups on the silica gel surface. Thus, the compounds examined show similar R_F values (see Fig. 1).

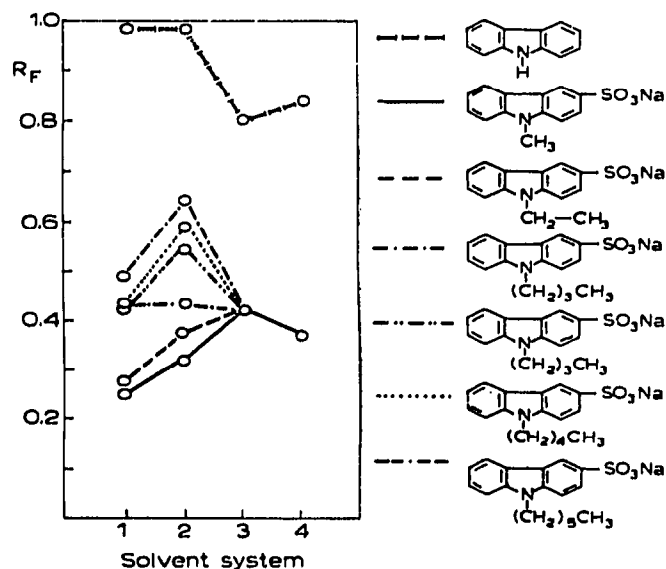


Fig. 2. R_F values of carbazole and of derivatives of sulphocarbazole obtained using a multi-component mobile phase: 1 = ethyl acetate-methanol-formic acid-pyridine, (80:10:10:10); 2 = ethyl acetate-methanol-formic acid-pyridine, (75:7.5:7.5:10); 3 = ethyl acetate-methanol-formic acid-piperidine, (80:10:10:10); 4 = ethyl acetate-methanol-formic acid-morpholine, (80:10:10:10).

The results obtained using a multi-component solvent system as the mobile phase are presented in Fig. 2. Using a multi-component mobile phase, differentiations of adsorption capacities of separate components of this mixture are observed. Consequently, different actions occur between the solvent molecules and the substances separated, on the one hand, and the adsorbent and the substances, on the other.

As the properties of the components of the mobile phase differ, a competitive displacement of the adsorbed molecules from the adsorbent surface will take place, which results in the separation of the chromatographed compounds. As shown in Fig. 2, a satisfactory separation of the compounds takes place only in one case using ethyl acetate-methanol-formic acid-pyridine (75:7.5:7.5:10) as mobile phase. This phenomenon is associated with the adsorption of a substance from multi-component solvent systems which is still not well understood. The above results show that a

satisfactory separation of the derivatives of sulphocarbazole acid is available using a mixed solvent of definite structure, as a mobile phase.

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Received October 21st, 1969

J. Chromatog., 46 (1970) 204-207

CHROM. 4482

A quantitative analysis of sorbose by thin-layer chromatography in the presence of some frequently occurring monosaccharides

Thin-layer chromatography is utilized extensively for the analysis of substances including sugars and sugar mixtures. Several authors¹⁻¹⁰ have described solvent systems which separate the various components of monosaccharide mixtures containing, among others, sorbose, by using variously prepared layers. The R_F values given by the authors show that the determination of sorbose is most frequently disturbed by fructose and mannose and more rarely by glucose and xylose. The best separation of sorbose from the other monosaccharides by unidirectional development has been published by WALDI⁸ and by LATO and co-workers^{9,10}. The two-dimensional chromatograms published by FIGGE³ also show a good separation; however, it is well known that for quantitative determinations the unidimensional technique is more preferable.

Our present paper describes a method which allows the separation of sorbose from some frequently occurring monosaccharides by unidirectional development and its quantitative analysis.

Separation

30 g of Kieselgel G are mixed with 70 ml of 1/15 M phosphate buffer (pH 7), and a layer 0.25 mm thick is prepared from this mixture. The plates are then dried for 1 h at 100° after spreading. On the plates divided into strips are applied 25-50 μ g of the aqueous solution of the following monosaccharides in 10 μ l: sorbose, glucose,

J. Chromatog., 46 (1970) 207-210