kurzer Zeit hat sich konstanter Druck eingestellt, und die Ampulle ist gleichzeitig erwärmt worden. Durch Druck oder leichten Schlag auf den Kopf N des Stabes S wird die Ampulle zertrümmert. Der verdampfte Inhalt wird von dem von oben eintretenden Gasstrom erfasst und durch die Bohrungen der Hülse und die Rillen auf die Säule gespült. Eine Rückdiffusion nach oben wird durch den bei E eintretenden Gasstrom verhindert. Nach Beendigung des Versuchs wird der Hahn V geschlossen und dann der Stab herausgezogen. Um ein Ausströmen des bei E eintretenden Schleppgases zu vermeiden, wird die Vorrichtung mit einem Blindstab verschlossen.

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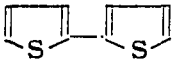
Eingegangen den 9. März 1964

J. Chromatog., 15 (1964) 538–540

Gas chromatography in the separation and identification of isomeric bithienyls

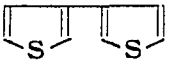
The reported methods for the separation and identification of isomeric bithienyls and polythienyls are lengthy and are not sensitive to low concentrations. SEASE AND ZECHMEISTER[1] separated bithienyl from terthienyl and other polythienyls by chromatographing on alumina from solvent solutions. WYNBERG AND BANTJES[2] used chromatography on basic alumina together with U.V. and I.R. absorption bands and mass spectral analysis for the separation and identification of the isomeric bithienyls.

In the radiation chemistry of thiophene a rapid, direct and highly sensitive method for the detection and analysis of radiation induced isomeric bithienyls was needed. Preliminary work with gas chromatography using thermistor and thermal conductivity detectors did not separate the three isomeric bithienyls (I, II, III).



2,2′-Bithienyl
(I)

2,3′-Bithienyl
(II)



3,3′-Bithienyl
(III)

A flame ionization detector employing a 6 ft. ¼ in. O.D. stainless steel column packed with 15 % silicone gum rubber (SE-30) coated on Chromosorb W (60–80 mesh) also did not separate the three isomers. However, a Carbowax 20 M column described below gave good separation of the three bithienyls.

Experimental

The bithienyls were separated using an FM Model 720 Chromatograph with a Model 1609 flame ionization detector. The 6 ft. ¼ in. O.D. stainless steel column was packed with 15 % by weight of Carbowax 20 M on Chromosorb W (80–100 mesh).

The experimental conditions were:
Carrier and gas flow: helium 130 ml/min; hydrogen 58 ml/min; air 640 ml/min.
Injection port temperature: 260°.
Detector temperature: 205°.
Column temperature: 220°.

Injections of 1 μl of a mixture of thiophene and isopropanol solutions of the isomeric bithienyls were made. The concentration of the three bithienyls present in 1 μl of isopropanol–thiophene solvent mixture are shown in Table I. The isomeric bithienyls

TABLE I

GAS CHROMATOGRAPHY OF ISOMERIC BITHIENYLS ON CARBOWAX 20 M

Bithienyl	*Retention time (min)*	*Concentration (μg in 1 μl solvent)*	
		used in Fig. 1	*lowest amount detectable*
2,2′-	6.6	0.96	0.028
2,3′-	7.6	1.0	0.029
3,3′-	9.2	0.92	0.027
(*Solvents*)			
Isopropanol	0.5		
Thiophene	0.6		

used were purified and characterized by Dr. H. WYNBERG, as described in ref. 2. Trace amounts of the isomers were present in each of the bithienyls. In addition, solvent dilutions of the isomeric bithienyls were made containing approximately 3 and $8 \cdot 10^{-8}$ g of each compound per μl.

Results and discussion

Fig. 1 shows the excellent separation of the three isomeric bithienyls obtained with the Carbowax 20 M column. The lowest concentration of bithienyls detectable was approximately $3 \cdot 10^{-8}$ g with an attenuator setting of 10 × (Table I). There was no improvement in the resolution of the three isomers by using programmed temperature

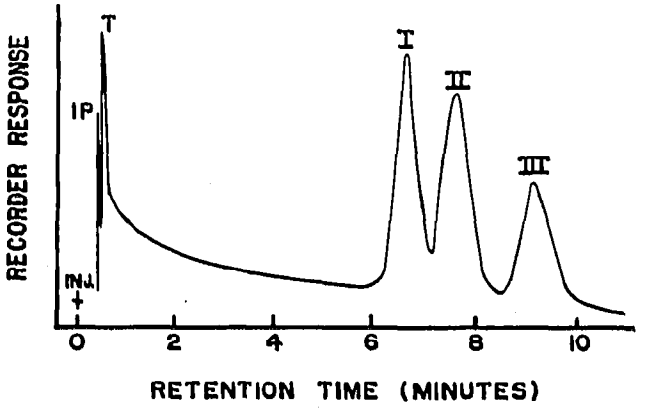


Fig. 1. Gas chromatogram of solvent solution of isomeric bithienyls. Column = 15% Carbowax 20 M on Chromosorb W (80–100 mesh). Column temperature = 220°. Injection port temperature = 260°. Helium flow = 130 ml/min. Sample size 1.0 μl. Attenuator setting = 200 ×. Peak I = 2,2′-bithienyl. Peak II = 2,3′-bithienyl. Peak III = 3,3′-bithienyl. IP = isopropanol. T = thiophene.

chromatography (120° start and 10° per min). Isothermal chromatography at 188°, 220° and 240° gave good separation of the isomers. The sharpest peaks were obtained at a temperature of 240°.

A flame ionization detector with the Carbowax 20 M column gave the best separation of isomeric bithienyls with a number of substrates studied (tricresyl phosphate, silicone gum rubber SE30 and Apiezon L grease). The Carbowax column gave excellent separation and identification of low concentrations of the isomeric bithienyls produced in the radiation chemistry of thiophene. The column material should be useful in the separation and identification of other isomeric heterocyclic compounds.

Acknowledgement

The author thanks Dr. H. WYNBERG for supplying the isomeric bithienyls. Appreciation is expressed to Drs. H. GISSER AND W. MCNEILL for review of the manuscript and to Mr. L. CELLI for experimental work.

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SIGMUND BERK

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Received March 3rd, 1964

J. Chromatog., 15 (1964) 540–542

Chromatography of fat-soluble food dyes on thin starch layers with stationary non-polar phases

Of the fat-soluble food dyes only the natural ones are permitted in moist countries. The use of synthetic fat-soluble dyes became restricted since Japanese research workers found them to be carcinogenic, especially Butter Yellow[1,2]. For this reason colouring of foods with these synthetics was prohibited in several countries, including Czechoslovakia[3]. The strict restriction, or even prohibition of these substances calls for a suitable, and above all, rapid analytical method of detection, or as the case may be, even for determination, of these substances in food inspection and research work.

Chromatographic methods based on adsorption[4] or on partition[5–7] are mostly used for the detection of fat-soluble food dyes. A classical system of identification of lipophilic food dyes has been described by THALLER AND SCHELLER[8] and JAX AND AUST[9]. These methods are based on pre-separation into groups by adsorption column chromatography; the components are then identified by means of partition paper chromatography. As the adsorption material alumina, various clays, etc., were employed. The use of partition paper chromatography which a stationary non-polar phase (paraffin oil) was first described by LINDBERG[5]. For the stationary phase other substances such as *n*-lauryl alcohol, oleic acid, diacetylethylene glycol monostearate[10],