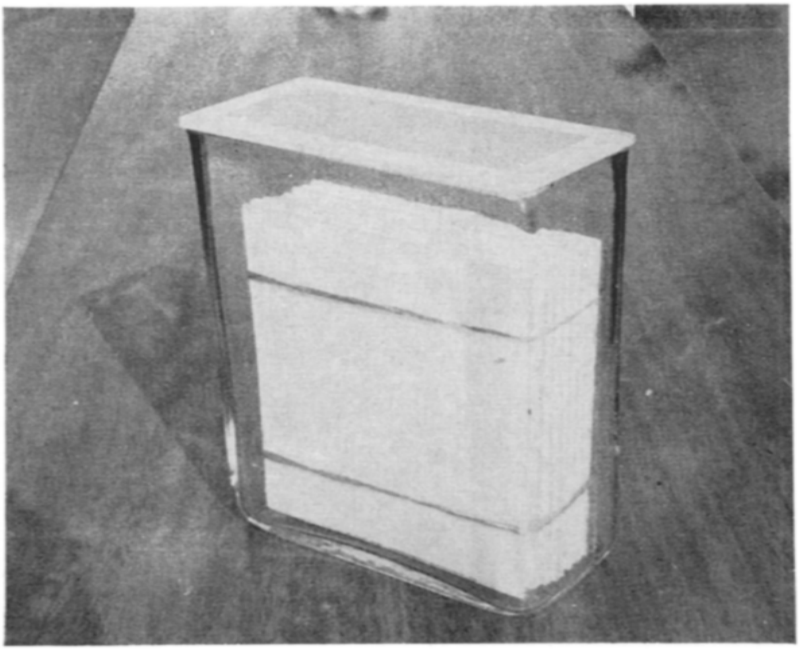

Fig. 4.

If the stacks become very large and heavy, it may be advisable to have two wire hooks for lifting them into and out of the tank. It is evident that the technique is also suitable for the simultaneous development of two-dimensional plates.

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[1] N. NYBOM, *Nature*, 198 (1963) 1229.

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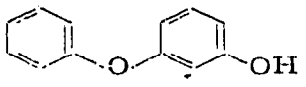
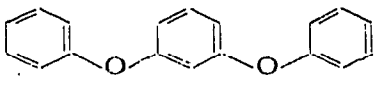
Separation of polyphenyl ethers by thin-layer chromatography

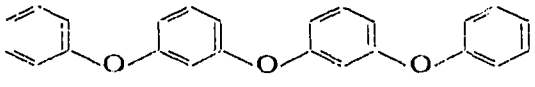
A multiple development technique

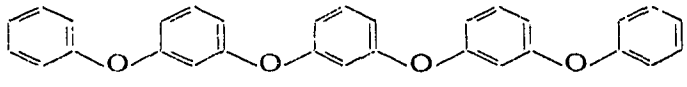
During an investigation of the mechanism of the oxidation at high temperatures of polyphenyl ethers, a method was needed for separating the oxidation products. Among the model compounds chosen for study were the following: *m*-phenoxyphenol (I), *m*-diphenoxybenzene (II), bis-(*m*-phenoxyphenyl) ether (III), and *m*-bis-(*m*-phenoxyphenoxy)-benzene (IV).

Initial attempts at separating a mixture of the above compounds by column chromatography on silica gel with various solvents were unsuccessful, so thin-layer chromatography was attempted in hopes of finding suitable conditions for the separation. It was found that a single elution and development of the above compounds with the most suitable solvent system did not give satisfactory separation but that multiple


(I)


(II)


(III)


(IV)

development of thin-layer chromatograms provided efficient separation of a mixture of I, II, III, and IV.

The *m*-phenoxyphenol and bis-(*m*-phenoxyphenyl) ether were commercially available. The *m*-diphenoxybenzene and *m*-bis-(*m*-phenoxyphenoxy)-benzene were kindly supplied by Dr. GLENN R. WILSON of this laboratory and were distilled before use. The chloroform, cyclohexane, and benzene used were reagent-grade and were used without further purification. A 1 % (by wt.) solution in chloroform of each of the above compounds was prepared by dissolving 0.377 g of the compound in exactly 25.00 ml of chloroform. A synthetic mixture of I, II, III, and IV was prepared by mixing 5 ml of each of the 1 % chloroform solutions.

Thin-layer chromatograms were prepared in the usual manner. They consisted of a 250-μ layer of Silica Gel G on a 20 × 20 cm glass plate applied with a fixed thickness spreader (Research Specialties Company, Richmond, Calif.).

Five separate plates were carefully spotted with a Lang-Levy micropipette (10 λ) with each of the above solutions including the synthetic mixture. This permitted easy comparison of the distance traveled by the pure compounds with the distance traveled by the compounds in a mixture and the identification of the compound in the mixture.

The chromatoplates were activated in an oven at 105–110° for 0.5 h before each development except in the one case noted below. It was found by application of the "Micro Circular Technique"[1], that the best development solution was 5 % benzene – 95 % cyclohexane. The development chambers were standard size and the larger walls were covered with Whatman No. 1 filter paper saturated with the benzene–cyclohexane solution to minimize "edge effects" and the "fringe phenomenon".

The two methods described below were used to determine the effects of multiple development on the separation of the polyphenyl compounds.

Method A

Four plates, activated and spotted identically were developed simultaneously. After drying in air, one plate was sprayed with a fluorescent indicator (Aldrich Chemical Co., Fluorescent indicator for hydrocarbons) and viewed under U.V. light. The distances traveled for each pure compound and for each compound in the mixture were recorded. The remaining three plates were activated at 110° for 0.5 h and were subjected to another development. Again one plate was used to determine the distances traveled. The third plate was *not* reactivated before an additional development to determine whether reactivation was necessary to significantly increase the distances traveled. The fourth plate was activated before a third and fourth development.

Method B

A single chromatoplate, after activation, was spotted with the above solutions and developed in the same manner. However, instead of spraying with fluorescent indicator, the location of each of the compounds was determined by treatment with iodine vapor. Once the spots were located and distances traveled tabulated, the iodine was allowed to evaporate from the chromatoplate. After the iodine had completely evaporated, the plate was reactivated at 110° for 0.5 h and redeveloped. The procedure was repeated three times and the distance traveled by each spot was recorded after each iodine treatment.

In all developments, the solution was allowed to travel from one end of the chromatoplate to a line drawn across the plate at a distance of 10 cm from the position of the spots. Since the distance between the spots and the line to which the solvent travels decreases with each development, one cannot use "R_F" values to refer to the distances traveled by the spots except for the very first development. The easiest way to record data in multiple developments is simply to note the total distance the spot has traveled from its original position. These values for the pure compounds I, II, III, and IV from method A and B are recorded in Table I.

TABLE I

DISTANCES TRAVELED ON MULTIPLE DEVELOPMENT (cm × 10^{-1})

Compound	Development number							
	Method A				Method B			
	1	2	3*	4	1	2	3	4
I	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
II	0.25	0.47	0.50	0.65	0.25	0.45	0.60	0.68
III	0.17	0.33	0.36	0.46	0.15	0.30	0.40	0.50
IV	0.10	0.18	0.22	0.30	0.10	0.19	0.26	0.32

* Chromatoplate not activated before final development.

The values of the distance traveled for the compounds in the synthetic mixture are recorded in Table II.

A slight impurity was found in "pure samples" of I and II after the four development. It had traveled about 0.08 cm and therefore was neither of the higher phenyl ethers being studied.

The correlation between the two methods is good considering the experimental error. The multiple development technique significantly increases the separation for the compounds separated. While no system could be found that cleanly separated these compounds with a single development, two developments were sufficient for clean separation. It seems necessary to activate the plates after each development since development 3, Method A, showed very little change in the distance traveled while a similar activated plate, development 3, Method B, showed significant changes.

It should be mentioned that the polyphenyl ethers are base fluids for high temperature lubricants and because of their high thermal stability there is little

TABLE II

DISTANCES TRAVELED ON MULTIPLE DEVELOPMENT OF SYNTHETIC MIXTURE (cm × 10^{-1})
The braces denote overlap of spots; a is about 20% overlap, b is about 50% overlap

Compound		Development number							
		Method A				Method B			
		1	2	3*	4	1	2	3	4
I		0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
II	a	0.23	0.48	0.48	0.64	0.24	0.43	0.57	0.65
III	a	0.14	0.29	0.30	0.43	0.20	0.27	0.38	0.45
IV	b	0.09	0.16	0.17	0.30	0.10	0.13	0.22	0.29

* Chromatoplate not activated before final development.

danger of reaction or rearrangement on heating to 110° in the presence of silica gel. Other systems may not be so obliging.

These results have been used to obtain an efficient separation of the above compounds and similar polyphenyl ethers in column chromatography.

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[1] E. STAHL, *Chemiker Ztg.*, 82 (1958) 323.

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Die Anwendung der Dünnschichtchromatographie zur Trennung des 2,5,7,8-Tetramethyl-2-(β-carboxyäthyl)-6-hydroxychromans und des Tocopheronolactons von α-Tocopherylchinon und einigen Tocopherolen

Untersuchungen von SIMON und Mitarbeitern[1] über den Wirkungsmechanismus des α-Tocopherols zeigten, dass das Tocopheronolacton [Lacton des 2-(3-Hydroxy-3-methyl-5-carboxypentyl)-3,5,6-trimethylbenzochinons] ein Umwandlungsprodukt des α-Tocopherols im tierischen Organismus darstellt. Von MARTIUS UND FÜRER[2] wird das 2,5,7,8-Tetramethyl-2-(β-carboxyäthyl)-6-hydroxychroman als eine sehr wahrscheinliche Abbaustufe des α-Tocopherols durch β-Oxydation angesehen. Beide genannten Verbindungen sind von WEICHET und Mitarbeitern[3] bereits synthetisch hergestellt worden*. Wegen ihrer grossen Bedeutung sollte geprüft werden, welche Möglichkeiten bestehen, 2,5,7,8-Tetramethyl-2-(β-carboxyäthyl)-6-hydroxychroman, Tocopheronolacton, α-, γ-, δ-Tocopherol und α-Tocopherylchinon mit Hilfe der Dünnschichtchromatographie voneinander zu trennen. Die Ergebnisse sind in Tabelle I zusammenge-

* Herrn Dr. WEICHET vom Institut für Pharmazie und Biochemie, Prag, danke ich herzlich für die Überlassung von Substanzproben.