

Separation of the radiolysis products of *o*-iodobenzoic acid

The expected products of radiolysis of *o*-iodobenzoic acid are benzoic acid, mono- and di-hydroxybenzoic acids and diphenic acid. For the study of the radiation chemistry of *o*-iodobenzoic acid the separation and quantitative determination of all these aromatic acids were required.

Many authors¹⁻³ have studied the chromatographic behaviour of mono- and di-hydroxybenzoic acids in different solvents and have determined the respective R_F values, but no method has been reported which yields a clean separation of all the acids we were concerned with and which would permit their subsequent quantitative determination.

A method has been developed, which permits the separation and semi-quantitative determination of all these acids by means of ascending chromatography, using a single solvent consisting of benzene-propionic acid-water (2:2:1)³. The acids were detected by four different methods: spraying; ultraviolet illumination; in the case of benzoic acid, which does not react with the developers used in this solvent, detection was performed by automatic scanning or by autoradiography, in comparison with ¹⁴C-labelled standards; diphenic acid was detected by two-dimensional chromatography using two different solvents (see Table I).

Experimental

Samples containing 10 μ l of 10⁻³ M solutions of the acids were spotted on Whatman No. 1 chromatography paper. After a run of 4-5 h, during which time the solvent

TABLE I

SUMMARY OF R_F VALUES OBTAINED FOR THE DIFFERENT ACIDS AND OF THE METHODS OF DETECTION

<i>Acid</i>	R_F	<i>Method of detection</i>
Benzoic	0.98	Scanning; autoradiography
Salicylic	0.93	U.V., spraying with diazotised <i>p</i> -nitroaniline (DPNA) + K ₂ CO ₃ solution ⁴
<i>m</i> -Hydroxybenzoic	0.77	U.V., DPNA spray
<i>p</i> -Hydroxybenzoic	0.83	U.V., DPNA spray
2,3-Dihydroxybenzoic	0.55	U.V., DPNA spray
2,4-Dihydroxybenzoic	0.40	U.V., DPNA spray
2,6-Dihydroxybenzoic	0.15	DPNA spray
3,4-Dihydroxybenzoic	0.25	Ammoniacal silver nitrate spray ⁴
3,5-Dihydroxybenzoic	0.11	DPNA spray
Diphenic	0.06	Two-dimensional chromatography using first the propionic acid-benzene-water system, then the paper is rotated 90°, introduced into butanol-benzene-ammonia buffer (80:5:15) ⁵ solvent and sprayed with alcoholic methyl red in phosphate buffer ⁵

travelled a distance of about 30 cm, the paper was dried and the acids detected by the above-mentioned methods.

The R_F values are averages for the results obtained in 10 determinations. The relative standard deviation calculated from 10 determinations was 3.0 %.

Applications

As seen from Table I the differences between the R_F values of the individual acids are large enough to permit identification on the same paper strip. If quantitative determination is required, the strips may be cut at the respective R_F values, the acids eluted and their U.V. absorbance determined with a spectrophotometer.

*Ministry of Defence, Atomic Energy Commission,
Soreq Research Establishment, Yavne (Israel)*

MARIANNA MANTEL
ESTHER KUSHNIR

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Chromatography on ion exchange papers

XVII. The adsorption of metal ions on cation and anion resin papers from nitric acid

The adsorption of metal ions on cation exchangers from hydrochloric acid, perchloric acid and sodium perchlorate was studied by column methods¹ as well as ion exchange papers techniques^{2, 3}, and it was observed that above a certain concentration of acid (or salt) the adsorption of metal ions again increases.

As no data for the adsorption of metal ions from nitric acid were available the present study was undertaken. It was further considered of interest to compare cation and anion resin papers for a wider range of ions than studied previously⁴.

The technique used here was identical to that used in previous studies²⁻⁴, the nitrate form of the anion resin paper (SB-2) being prepared by equilibrating the chloride form with an ammonium nitrate solution. The cation (SA-2) resin paper was employed in the hydrogen form. When the metal ions were spotted on the dry anion resin paper and developed with HNO_3 , some ions (notably the rare earths) were prone to double spotting, one spot moving with the liquid front. This could be avoided by running the developing solvent for 3 cm over the paper and spotting the metal ions behind the liquid front.

The results obtained are shown in Fig. 1 where the metal ions are arranged in the order of the periodic table. The following points of interest were noted:

(1) There is an increase in adsorption above 6 N HNO_3 on the cation resin paper of the same magnitude as was observed in perchloric and hydrochloric acids.

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