standard deviation and the 95 % confidence limits of the single determinations are also reported. The reported data agree with theoretical values.

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Received September 22nd, 1967

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Thin-layer and gas chromatography of trimethylsilyl ethers of glycols

Trimethylsilyl derivatives have proved useful in the chromatographic identification of many organic compounds containing replaceable hydrogen atoms[1]. We describe the following procedures which we have employed in identifying a number of cyclic glycols and phenylethylene glycols as metabolic products of unsaturated compounds.

A solution or extract containing the glycol was placed in a glass-stoppered 15-ml conical centrifuge tube and evaporated to dryness. The residue was dissolved in 0.14 ml of pyridine, after which 40 μl of hexamethyldisilazane and 20 μl of trimethylchlorosilane were added, mixing after each addition. The tube was then stoppered and maintained at an appropriate temperature for an appropriate period of time. If an elevated temperature was required, the glass-stoppered top of the tube was wrapped with aluminum foil to prevent seepage of condensate, shaken gently in a thermostatted water bath, and cooled to room temperature before opening. The reaction mixture could be kept overnight at room temperature without loss of sensitivity. Aliquots of the reaction mixture were directly injected in the gas chromatograph, or else the contents of the tube were evaporated to dryness under a stream of dry nitrogen passed through a capillary tube extending to just above the surface of the liquid. The residue was leached with 0.5 ml of hexane, and aliquots of the extract used for thin-layer or gas chromatography. If it was desired to concentrate the extract, evaporation was performed under nitrogen.

For thin-layer chromatography, Eastman Chromagram sheets No. 6060 (coated with silica gel containing a fluorescent indicator) were activated at 110° for 15 min. Spots were applied without using an air stream for drying. Ascending chromatography was carried out with heptane. The sheets were then dried at room temperature and the spots located under U.V. (2537 Å) light.

TABLE I

PREPARATION AND CHROMATOGRAPHY OF TRIMETHYLSILYL ETHERS OF GLYCOLS

Glycol	*Silylation*		*Thin-layer chromatography*		*Gas chromatography*			
	Time (min)	*Temperature (°C)*	R_F	*Sensitivity (μg)*	*Column*[a]	*Conditions*[b]	*Retention time (min)*	*Sensitivity (μg)*
cis-1,2-Dihydroxyindane	45	70	0.10 (1)[c] 0.19 (2) 0.28 (3)	10	I	A	14.6	0.5
trans-1,2-Dihydroxyindane	45	70	0.22 (1)[c] 0.36 (2) 0.45 (3)	10	I	A	15.8	0.5
cis-1,2-Dihydroxycyclohexane	60	room	—	—	I	B	8.6	0.25
trans-1,2-Dihydroxycyclohexane	60	room	—	—	I	B	9.6	0.25
Hydrobenzoin	60	room	0.55	10	II	C	18.0	0.5
Isohydrobenzoin	60	room	0.30	10	II	C	19.0	0.5
Phenylethylene glycol	60	room	0.34	100	I	C	3.6	0.1

[a] Column I: 2% OV-1–3% OV-17 on Chromosorb W (AW-DMCS), 60/80 mesh; column II: 5% OV-17 on Chromosorb W (AW-HMDS), 60/80 mesh.

[b] Temperatures of column, flash heater, and detector are, respectively: (A) 120°, 215°, 175°; (B) 80°, 185°, 160°; (C) 130°, 235°, 190°.

[c] The solvent front was run 12 cm from the origin, the sheet was dried, and the spots were visualized under U.V. light. The sheet was then returned to the development tank and the solvent again run 12 cm. After drying and location of spots, the process was repeated a third time.

Gas chromatography was carried out with a F & M model 400 apparatus equipped with a flame ionization detector, using 4-ft. glass columns and helium carrier gas at 100 ml/min.

Conditions for the silylation reaction with a number of glycols and details of chromatography are shown in Table I. The derivatives of both *cis–trans* pairs could be separated by gas chromatography, and those of *cis*- and *trans*-1,2-dihydroxyindane by thin-layer chromatography (the dihydroxycyclohexane derivatives were not visible under U.V. light). The ethers of the racemic and *meso* pair represented by the hydrobenzoin isomers were separated by both thin-layer and gas chromatography. In most cases the limit of sensitivity in gas chromatography was the same whether the reaction mixture or the extract was injected. This was not true for the phenylethylene glycol derivative, which was poorly extractable. The overall sensitivity was therefore very poor when the extract of this derivative was chromatographed in either way. We were unable to find satisfactory conditions for thin-layer chromatography after direct spotting of the reaction mixtures.

This work was supported by U. S. Public Health Service Grant No. UI 00453 (formerly OH 00251).

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Received September 25th, 1967