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Note

Separation of benzyl compounds by gas-liquid chromatography

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The gas chromatography of benzyl compounds has not been studied quantitatively, although some have been included in studies of various aromatic compounds¹⁻³. Hishta and Bonstein⁴ separated mono-, di- and tribenzylamines using a Carbowax 1500 column. The present investigation was concerned with the quantitative separation of benzyl compounds on nine different liquid stationary phases, and a general equation relating the specific retention volumes with the reciprocal of the column temperature was derived.

EXPERIMENTAL

Apparatus

A Beckman GC-45 gas chromatograph equipped with a flame-ionization detector was used, together with a Beckman 10 recorder with a 1-mV capacity. Nitrogen was used as the carrier gas at an optimal flow-rate of 30 ml/min at N.T.P. Stainless-steel columns (6 ft. \times $\frac{1}{4}$ in. O.D.) were used. Each column was packed with 20% (w/w) of stationary phase on 80-100-mesh Chromosorb W. The column temperature was controlled to within $\pm 0.08^\circ$.

Materials

All of the benzyl compounds were obtained from Fluka (Buchs, Switzerland) in the purest available grades. The purities were checked.

Sampling

A mixture containing 0.25 g of each benzyl compound was prepared. The sample sizes ranged from 0.2 to 0.4 μ l and injections were made with a 1- μ l Hamilton syringe. Each injection for an individual compound or mixture was repeated twice and the results were reproducible to within $\pm 1\%$.

RESULTS AND DISCUSSION

The retention data for benzyl compounds at different column temperatures (90-180°) were expressed in the form of specific retention volumes^{5,6}, V_g^0 , and these values were further processed by linear regression so as to obtain the constants a and b of the equation

$$\log V_g^0 = (a/T) - b \quad (1)$$

TABLE III

HEATS OF SOLUTION ($-\Delta H$) OF BENZYL COMPOUNDS (kcal/mole) IN DIFFERENT LIQUID STATIONARY PHASES

Compound	OV-1	OV-3	OV-11	OV-25	DEGS	DEGGLU	DEGA	EGTCP	Carbowax 6000
Benzyl chloride	10.052	10.256	10.390	10.520	10.052	10.549	10.396	8.809	5.654
Benzylamine	13.182	10.256	11.172	11.101	13.182	13.116	13.233	11.885	8.864
Benzyl alcohol	12.998	10.730	10.965	11.101	12.998	12.772	12.590	11.885	9.842
Benzyl acetate	12.627	11.736	12.063	12.343	12.343	13.448	13.572	11.885	11.526
Benzyl methyl ketone	13.147	11.767	12.584	12.801	13.147	13.483	13.528	12.918	13.074
Benzyl cyanide		12.365	12.450	12.849					
Dibenzyl ether	13.708	13.972	15.428	15.254	13.708	14.696	14.801	22.163	13.072
Dibenzylamine	15.365	14.485	15.342	15.109	15.365	16.058	15.747	24.371	15.457
N-Benzylaniline	15.365	14.485	15.657	15.683	15.365	15.354	16.480		16.300
Benzyl phenyl ketone	15.117	14.530	15.777	15.837	15.117				16.300
Benzyl benzoate	15.550	14.725	16.181	15.839	15.550				17.266
Benzyl salicylate		15.209	15.427	15.574					

number of CH_2 groups in the latter, but there are appreciable variations in the ΔH values of benzyl compounds that contain two phenyl rings. On Carbowax 6000, the variation in ΔH values ranged between 5 and 17 kcal/mole, depending on the structure of the benzyl molecules and whether these molecules are able to reduce the internal association of this liquid phase⁷.

The results in Tables I and II indicate that the best separation was achieved with OV-11 and DEGGLU as liquid stationary phases.

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