NOTES.

Molecular Solution Volumes of Optical Isomers. By WILLIAM H. BANKS.

PATTERSON and LAMBERTON (this vol., p. 1453) compare molecular solution volumes derived from density measurements on solutions of the optically active isomers of *iso*butyl tartrate. The differences obtained for their solutions in *l*-menthyl acetate are of the order of 0·05 c.c. and are considered to be significant, being several times as great as the probable error of experiment. A consideration of the experiment shows, however, that these differences may be of the same order as error arising from temperature uncertainty.

The effect of uncertainties in density Δd_2 and concentration Δp on the molecular solution volume ϕ may be derived from the authors' equation and is (in their nomenclature)

$$\Delta\phi = \pm 100M \,.\, \Delta d_2/pd_2^2 \mp 100M\{1/d_1 - 1/d_2\}\Delta p/p^2$$

which on substitution of appropriate quantities ($M = 262$; $p = 5{\cdot}5\%$, say; $d_1 = 0{\cdot}925$, $d_2 = 0{\cdot}932$) becomes

$$\Delta\phi = \pm 5483\Delta d_2 \mp 7{\cdot}06\Delta p.$$

It is clear that errors in p of 10^{-3} and less (the authors record values to four decimal places) need not be considered. An uncertainty in density, however, of 1×10^{-5} must produce an uncertainty in ϕ of 0·055 c.c. In reference to the second and more accurate series of measure-

ments it is stated that most interpolated values for the temperature 20° were within 1×10^{-5} unit of a mean density–temperature line. That this order of accuracy may reasonably be considered the limit is to be expected from possible temperature uncertainties. A calculation from the authors' data indicates that the *l*-menthyl acetate solutions have a temperature coefficient of about 8×10^{-4} unit/degree, so an uncertainty in temperature of 0·01° will introduce an uncertainty of 8×10^{-6} unit in density. Such an uncertainty in temperature is not unreasonable, since the authors use a thermometer graduated only to 0·05°.

It would seem difficult to attach significance to such consistent differences as are apparent, since these are derived from a comparison of a mean value of only three values for the *l*-isomer with a mean of five for the *d*-isomer, of which one value, VI, is of the same order as those for the *l*-isomer.—BATTERSEA POLYTECHNIC. [*Received, September 30th*, 1937.]

Dipole Moments and Molecular Structure. The Dipole Moments of p-*Hydroxyazobenzene and its Derivatives compared with those of Phenol and its Derivatives.* By FRANK LOUIS WARREN.

THE moments of *p*-hydroxyazobenzene and phenol and their respective derivatives have been compared, for, since azobenzene has zero moment, *p*-hydroxyazobenzene should have the same moment as that of phenol, the moment in both cases being due to the hydroxy-group. The data obtained for *p*-hydroxy- and *p*-methoxy-azobenzene confirm those of Bergmann and Weizmann (*Trans. Faraday Soc.*, **1936**, **185**, 1318) and are therefore not given in detail; those for phenyl benzoate and *p*-benzoyloxyazobenzene are tabulated below. The final table shows the close agreement between the moments of the corresponding benzene and azobenzene derivatives, and supports the above authors' conclusion that *p*-hydroxyazobenzene in benzene solution exists almost wholly in the azo-form.

Experimental.—The apparatus used for measuring the dielectric constant was that described by Sugden (J., **1932**, 768), with a 210 kc./sec. quartz crystal controlled oscillator in place of the dynatron circuit, and a Sullivan dual-range (65—355; 320—1270 μμ F.) variable air condenser fitted with a double vernier to give a more open scale, as recommended by Sugden. The measuring cell (Farmer and Warren, J., **1933**, 1300) had a capacity of 203 μμ F. with air as dielectric, and a calibration correction of only 0·18 μμ F. (Sugden, *loc. cit.*). The electronic polarisations were calculated from $[R_L]_D$ 67·85 for azobenzene (Duval, *Compt. rend.*, **1911**, **153**, 874) and Eisenlohr's figures (*Z. physikal. Chem.*, **1895**, **16**, 218), except for *p*-methoxyazobenzene, for which the averages of Bergmann and Weizmann's measurements (*loc. cit.*) were employed.

Solutions in benzene.

Phenyl benzoate, m.p. 71°.

f_2.	$\epsilon^{30°}$.	$d_4^{30°}$.	$P_{1,2}$.	P_2.
0	2·2621	0·86836	26·619	
0·003734	2·2826	0·87025	27·018	133·4
0·007993	2·3040	0·87260	27·436	128·9
0·010940	2·3203	0·87417	27·748	129·8
0·014903	2·3438	0·87619	28·193	132·3

P_2 (mean), 131·1; P_E (calc.), 55·7; $P_2 - P_E$, 75·4.

p-Benzoyloxyazobenzene, m.p. 138°.

f_2.	$\epsilon^{30°}$.	$d_4^{30°}$.	$P_{1,2}$.	P_2.
0·003691	2·2867	0·87177	27·162	174·3
0·007246	2·3105	0·87496	27·688	174·2
0·007606	2·3137	0·87538	27·750	175·1
0·012855	2·3474	0·87991	28·510	173·7

P_2 (mean), 174·3; P_E (calc.), 97·8; $P_2 - P_E$, 76·5.

	$\mu \times 10^{18}$.
Phenol	1·70 (*b*), 1·61 (*c*)
Anisole	1·2 (*d*)
Phenyl benzoate	1·92

	$\mu \times 10^{18}$.
p-Hydroxyazobenzene	1·62, 1·62 (*a*)
p-Methoxyazobenzene	1·3, 1·29 (*a*)
p-Benzoyloxyazobenzene	1·93

(*a*) Bergmann and Weizmann (*loc. cit.*). (*b*) Smyth and Morgan (*J. Amer. Chem. Soc.*, 1927, **49**, 1030); Williams (*Physikal. Z.*, 1928, **29**, 174). (*c*) Donle and Gehrckens (*Z. physikal. Chem.*, 1922, *B*, **18**, 316). (*d*) Donle and Volkert (*ibid.*, 1930, *B*, **8**, 60); Hojendahl ("Studies in Dipole Moments," Thesis, Copenhagen, 1928; *Physikal. Z.*, 1929, **30**, 391); Estermann (*Z. physikal. Chem.*, 1928, *B*, **1**, 134).

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The Cyclisation of 2-(β-1′-*Naphthylethyl*)-Δ^2-cyclo*pentenone*. By STANLEY H. HARPER.

COHEN and COOK (J., 1935, 1570) were unable to cyclise 2-(β-1′-naphthylethyl)-Δ^2-*cyclo*pentenone by aluminium chloride in either benzene or nitrobenzene to 3′-keto-1 : 2 : 3 : 4-tetrahydro-1 : 2-*cyclo*pentenophenanthrene. It was thought that the use of phosphoric oxide might lead to the required ketone. The product isolated, however, was 1 : 2-*cyclo*pentenophenanthrene. The unsaturated ketone (6·3 g.) was heated with phosphoric oxide (13 g.) at 130° for 30 mins., considerable resinification occurring. The product was decomposed with ice and sodium hydroxide, and extracted with ether. After removal of the solvent, the residue gave on vacuum distillation 0·54 g. of crystalline material, which recrystallised from aqueous acetone or ethyl alcohol in clusters of shining plates, m. p. 134°, not depressed on admixture with an authentic specimen of 1 : 2-*cyclo*pentenophenanthrene (Kon, J., 1933, 1081; Harper, Kon, and Ruzicka, J., 1934, 124) (Found : C, 93·4; H, 6·5. Calc. for $C_{17}H_{14}$: C, 93·6; H, 6·4%). The identity was further confirmed by comparison of the picrate and the trinitrobenzene derivative. The unsaturated ketone was recovered unchanged from a similar experiment using syrupy phosphoric acid at 130° for 5 hrs.

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