NOTES.

Molecular Dissymmetry due to symmetrically placed Hydrogen and Deuterium. The α-Pentadeuterophenylbenzylamine Problem. By G. R. CLEMO and G. A. SWAN.

THE reported resolution of α-pentadeuterophenylbenzylamine (Clemo and McQuillen, J., 1936, 808) has been criticised on the ground that the hexadeuterobenzene used for the preparation of the base was impure (m. p. − 1°); and Adams and Tarbell (*J. Amer. Chem. Soc.*, 1938, **60**, 1260) state that they failed to resolve the base. As practically pure hexadeuterobenzene is now readily available here (Clemo and Robson, this vol., p. 429), we have examined the question afresh; but have failed this time to resolve the amine. The hydrogen *d*-tartrate had specific rotation $[\alpha]_D^{18^\circ} + 12{\cdot}4^\circ$, which remained virtually constant on repeated crystallisation of the salt from alcohol–light petroleum. After ten recrystallisations of the tartrate, the base was recovered, and found to be optically inactive. From this base, the oxalate was prepared; and this also was inactive. In this connection, however, attention should be drawn to the failure to resolve the comparable case of 4-methylbenzhydrylamine (this vol., p. 1958), and the problem at issue cannot therefore be considered to be settled.

The experimental conditions were the same as those used previously (J., 1936, 808) except where otherwise stated below.

Pentadeuterobenzophenone.—The hexadeuterobenzene used had m. p. 5·5°. On recrystallisation from light petroleum the pentadeuterobenzophenone had m. p. 47° (Found : C, 83·1; water, 58·7. Calc. for $C_{13}H_5D_5O$: C, 83·4; water, 50·8%). The oxime had m. p. 142° (Found : C, 77·6; water 49·2. Calc. for $C_{13}H_6D_5ON$: C, 77·2; water, 51·4%). α-Pentadeuterophenylbenzylamine was distilled; b. p. 135—140°/1 mm.

α-Pentadeuterophenylbenzylamine Hydrogen d-*Tartrate.*—When rapidly heated, this melted at 179—180° (Found : C, 60·1; water, 56·9. Calc. for $C_{13}H_8D_5N,C_4H_6O_6$: C, 60·3; water, 52·1%. Calc. for $C_{13}H_{13}N,C_4H_6O_6$: C, 61·3; water, 51·4%). 2 G. of this salt were crystallised ten times from alcohol–light petroleum, about 90% being recovered in each crystallisation. The m. p. remained constant; and the specific rotation only varied within the limits of experimental error, its average value being $[\alpha]_D^{18^\circ} + 12·4^\circ$ ($\alpha = + 0·278^\circ$, $l = 2$, $c = 1·120$ in ethanol).

Recovery of α-Pentadeuterophenylbenzylamine.—After the hydrogen *d*-tartrate had been repeatedly crystallised, the base was recovered; it gave $[\alpha]_D^{18^\circ} = 0·00^\circ \pm 0·02^\circ$ ($l = 0·5$, $c = 9·36$ in ethanol). α-Pentadeuterophenylbenzylamine oxalate had m. p. 204—205°; and gave $[\alpha]_D^{18^\circ} = 0·00^\circ \pm 0·01^\circ$ ($l = 2$, $c = 0·349$ in ethanol).

We thank Imperial Chemical Industries, Ltd., for a grant towards the purchase of " heavy " water; and one of us (G. A. S.) thanks the Department of Scientific and Industrial Research for a maintenance grant.—UNIVERSITY OF DURHAM, KING'S COLLEGE, NEWCASTLE-UPON-TYNE. [*Received, November 7th,* 1939.]

The Addition of Magnesium Iodide to Camphor and Terpene Derivatives.

By SYDNEY T. BOWDEN and THOMAS F. WATKINS.

THE observation that an ethereal solution of anhydrous magnesium iodide on treatment with camphor deposits a beautifully crystalline addition compound induced us to examine the behaviour of the halide with other terpene derivatives containing a carbonyl group.

Addition Compound with Camphor.—A mixture of dry ether (40 c.c.), magnesium powder (0·6 g.), and dry iodine (2·5 g.) was boiled under reflux (calcium chloride guard-tube) until the solution was almost colourless; it was then filtered with adequate protection against the intrusion of moisture. On addition of a solution of camphor (3 g.) in a few c.c. of ether, the addition *compound* was deposited in well-formed crystals (becoming light brown on standing), which were separated by filtration in a stream of dry air [Found : MgI_2 (by gravimetric estimation of the halogen), 37·1; $(C_2H_5)_2O$ (by loss in weight at 56°/20 mm. in a slow stream of nitrogen), 11·3. $5C_{10}H_{16}O,2MgI_2,(C_2H_5)_2O$ requires MgI_2, 37·1; $(C_2H_5)_2O$, 10·1%. The result for ether is appreciably high owing to partial sublimation of camphor under the conditions]. When the substance was slowly heated, it melted at 108°, then congealed to a mass which became deeply coloured as the temperature was raised, and finally melted at 176°.

Similar experiments with 3-bromocamphor showed that no reaction occurs on boiling the ethereal solution with magnesium iodide for 8 hours.

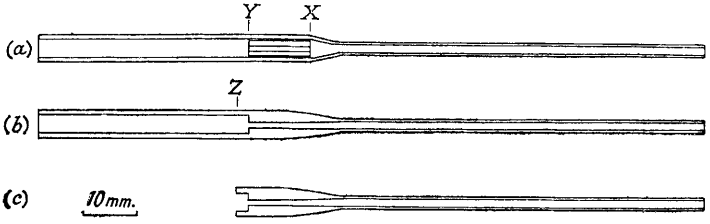
Addition Compound with Carvone.—The addition *compound* of carvone and magnesium iodide was precipitated as a brownish-red oil when ethereal solutions of the components were mixed at the ordinary temperature. The oil was washed with ether; it solidified after being kept for 24 hours in a desiccator containing activated silica gel. It melted at 85°, then congealed, and finally melted at 125°; at 200° decomposition occurred with visible evolution of iodine vapour [Found : MgI_2, 44·1; $(C_2H_5)_2O$, 7·9. $2C_{10}H_{14}O,MgI_2,(C_2H_5)_2O$ requires MgI_2, 44·6; $(C_2H_5)_2O$, 7·2].

Fenchone and magnesium iodide yielded a similar oily addition compound, which would not crystallise.

Addition Compound with Santonin.—Treatment of an ethereal solution of magnesium iodide with santonin led to separation of yellow crystals of the addition *compound*; it began to decompose before melting at 175°. The compound was analysed after removal of the solvent at 56° in a slow stream of nitrogen [Found : MgI_2, 35·9. $2C_{15}H_{18}O_3,MgI_2$ requires MgI_2, 36·1%).—TATEM LABORATORIES, UNIVERSITY COLLEGE, CARDIFF. [*Received, October 25th,* 1939.]

A Useful Micro-immersion Filter. By CHRISTINA C. MILLER.

To make the micro-immersion filter illustrated in (c), two pieces of Jena Geräte or Pyrex glass were required, namely, a 12 mm. length of capillary tubing of 3·5 mm. external diameter and 1 mm. bore, cut off square, and a 150 mm. length of tubing of 1 mm. wall, into which the capillary fitted *closely*. The longer piece of tubing was drawn out at one end to form a capillary of 2·5 mm. external diameter and 70 mm. length, and the thick capillary tube was pushed in at the other end as far as it would go (a). The pair of tubes was then steadily rotated and heated at X in a small and not too hot blowpipe flame, so as to fuse together the two pieces of glass with the minimum of deformation. The heating was gradually extended towards Y until the whole of the capillary tube was sealed in without deforming the flat end at Y. Thereafter the size of the flame was increased a little, the capillary heated more strongly towards X, and the tube, after slight blowing, drawn out to give the appearance shown in (b); the whole was annealed. Finally, the tube was cut sharply at Z, and the piece of wider tubing removed, leaving not more than 2 mm. projecting from the capillary. If the break was irregular, the tube was carefully ground flat on fine carborundum and fire-polished, without, however, causing the glass to collapse. The final form was that shown in (c).



For filtrations the filter was provided with a disc, 3·5 mm. in diameter and 1·5 mm. thick, cut with a clean, sharp cork-borer from a Whatman ashless filtration accelerator. The disc was pressed gently into position with a flat-ended glass rod, moistened with water, and attached to a suction apparatus. It was unnecessary to suck in filter paper fibre suspended in water, as there were no crevices.

For the determination of silica and the " mixed oxides " in 10 mg. portions of insoluble silicates, I prefer this filter to the immersion filters fitted with rolls of filter paper, as used by Schwarz v. Bergkampf (*Mikrochem.*, Emich Festschrift, 1930, 268) and Thurnwald and Benedetti-Pichler (*Mikrochem.*, 1932, **11**, 200), and also to that devised by King (*Analyst*, 1933, **58**, 325). The filters with rolls of paper have a small filtering surface; the washing of King's filter is apt to be troublesome if the outer glass tube does not fit closely round the capillary tube. The filter described has the relatively large filtering surface of King's filter, the filter disc is readily removed by easing it out with a thin platinum wire, and, finally, only a small amount of liquid is required for washing it.

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