NOTES.

The Action of Alkylmagnesium Iodides on Triphenylphosphine Dichloride. By BERTIE KENNEDY BLOUNT.

THE action of methyl- and ethyl-magnesium iodides on triphenylphosphine dichloride under normal conditions has been found to proceed according to the equation $(C_6H_5)_3PCl_2 + RMgI = [(C_6H_5)_3PR]I + MgCl_2$, giving triphenylalkylphosphonium iodides (R = Me or Et).

In view of these results, the substance of m. p. 162—163° (J., 1931, 1894) isolated from the products of Grignard and Savard's reaction, which is stated by these authors (*Compt. rend.*, 1931, **192**, 592) to give triphenyldialkylpentaphosphines, $(C_6H_5)_3PR_2$, has been re-examined and identified as impure triphenylethylphosphonium iodide, the anomalous mixed m. p. being due to the considerable depression in m. p. which the compound shows after standing for a time in the air. The corresponding methyl derivative does not behave in this way.

To a Grignard solution, prepared from methyl iodide (3·55 g.), was added a benzene suspension of triphenylphosphine dichloride, prepared from triphenylphosphine (1·31 g.). Heat was evolved and an orange-coloured precipitate separated, which was finally replaced by a white gelatinous solid. After refluxing gently for 2 hours, the mixture was shaken with water and dilute hydrochloric acid, and the heaviest of the three layers produced was separated, dissolved in alcohol, filtered, and gradually treated with ether. Triphenylmethylphosphonium iodide separated in a crystalline condition, m. p. 176—181° (1·05 g., 52%). After two further precipitations with ether and a final crystallisation from alcohol–ligroin, it melted at 182—183°, alone or mixed with triphenylmethylphosphonium iodide prepared from triphenylphosphine and methyl iodide (Found: C, 56·4; H, 4·5. Calc. for $C_{19}H_{18}IP$: C, 56·4; H, 4·5%).

By using similar conditions, with ethyl iodide instead of methyl iodide, a product, m. p. 164—165°, was finally obtained, shown by its m. p., mixed m. p., and general properties to be triphenylethylphosphonium iodide.

The author wishes to thank the Ramsay Memorial Fellowship Trustees for a Fellowship.—THE DYSON PERRINS LABORATORY, OXFORD. [*Received, November 20th*, 1931.]

Resolution of r-*Phenylmethoxyacetic Acid into its Optically Active Components.* By DOUGLAS JOHN CRUICKSHANK PIRIE and ISOBEL AGNES SMITH.

THE lævorotatory form of the acid has been prepared from lævorotatory mandelic acid by McKenzie (J., 1899, **75**, 753), but there is no reference in the literature to the resolution of the racemic acid. The resolution is an easy one and serves for the preparation of the dextrorotatory acid.

105 G. of hydrated quinine (1 mol.) and 46 g. of *r*-phenylmethoxyacetic acid (1 mol.) were dissolved by warming in 552 c.c. of a mixture of equal volumes of ethyl alcohol and water. The solution was cooled in the ice-chest for 5 hours, whereupon 90 g. of a salt crystallised in rosettes of needles. This salt, on being recrystallised from 300 c.c. of the alcohol-water mixture, gave 70 g. of salt (after drying in air on porous plate for 1 day) giving in ethyl alcohol ($l = 1$, $c = 2{\cdot}208$) $\alpha_D - 1{\cdot}92°$, whence $[\alpha]_D - 87{\cdot}0°$. After two further crystallisations, the salt weighed 60 g. and gave in ethyl alcohol ($l = 1$, $c = 2{\cdot}12$) $\alpha_D - 1{\cdot}74°$, whence $[\alpha]_D - 82{\cdot}1°$. The quinine salt was decomposed by dilute sulphuric acid, and the solution extracted with ether. 16 G. of dextrorotatory phenylmethoxyacetic acid (m. p. 64—65°) were obtained, having in ethyl alcohol ($l = 1$, $c = 1{\cdot}716$) $\alpha_D + 2{\cdot}6°$, whence $[\alpha]_D + 151{\cdot}5°$. This value, which did not increase on crystallisation of the acid from petroleum (b. p. 80—100°) to which a little benzene had been added, is in agreement with that ($[\alpha]_D - 151{\cdot}1°$) of McKenzie (*loc. cit.*) for the lævorotatory acid.—UNIVERSITY COLLEGE, DUNDEE. UNIVERSITY OF ST. ANDREWS. [*Received, December 4th*, 1931.]

A Convenient Method of preparing Anhydrous Aluminium Bromide. By RONALD P. BELL.

ALUMINIUM turnings are placed in a flask with a ground-in reflux condenser and covered with carbon disulphide. Rather less than the theoretical quantity of bromine, dissolved in 3—4 vols. of carbon disulphide, is added gradually through the condenser, with water-cooling if necessary. The mixture is warmed on the water-bath until the drops in the condenser are no longer coloured by bromine (½—1 hr.). The dark brown liquid is decanted from the solid residue (or if necessary filtered through glass wool) into a dry flask, which is immediately placed in a desiccator, and the carbon disulphide removed at room temperature (water-pump with a calcium chloride tube). The solid which separates at first is brown (probably owing to the presence of a compound with

carbon disulphide), but becomes almost white after several hours. The purity of the aluminium bromide, determined by the Volhard method, is 99·4—99·9%. The violence of the reaction with water was moderated by first dissolving the bromide in nitrobenzene, as described by Kaveler and Monroe (*J. Amer. Chem. Soc.*, 1928, **50**, 2421).

Since bromine is known to react with carbon disulphide at high temperatures, and this reaction might be accelerated by the presence of aluminium bromide, the product was tested for sulphur compounds by evaporating a portion to dryness with concentrated nitric acid, dissolving the residue in dilute hydrochloric acid, and testing the solution for sulphate: only a slight opalescence was formed on addition of barium chloride solution.—UNIVERSITY INSTITUTE OF PHYSICAL CHEMISTRY, COPENHAGEN, DENMARK.
[*Received, November 9th*, 1931.]