## III,8. *n*-HEXANE (*Hydrocarbon from Grignard Reagent*)

This preparation illustrates the preparation of a liquid hydrocarbon from a Grignard reagent. The Grignard reagent from *n*-hexyl bromide may be decomposed either with dilute sulphuric acid or with solid ammonium chloride; the latter gives a somewhat better yield.

$$CH_3(CH_2)_4CH_2Br + Mg = CH_3(CH_2)_4CH_2MgBr$$
$$2CH_3(CH_2)_4CH_2MgBr + H_2SO_4 = 2CH_3(CH_2)_4CH_3 + MgBr_2 + MgSO_4$$
$$2CH_3(CH_2)_4CH_2MgBr + NH_4Cl = 2CH_3(CH_2)_4CH_3 + NH_2MgBr + MgClBr$$

Fit a 500 or 750 ml. three-necked flask with a mercury-sealed stirrer, a 100 ml. dropping funnel and an efficient double surface condenser (Fig. *II*, 7, 11, *a*); place calcium chloride or cotton wool guard tubes on the funnel and condenser respectively. Arrange the flask so that it can be heated in a bath of hot water. Place 12·0 g. of magnesium turnings (1), 100 ml. of sodium-dried ether and a crystal of iodine in the flask. Weigh out 82·5 g. (70·5 ml.) of dry *n*-hexyl bromide (Section **III,37**) and introduce it into the separatory funnel. Run in about 10 g. of the *n*-hexyl bromide into the magnesium and ether. Set the stirrer in action. Warm the flask by surrounding it with hot water; remove the hot water immediately reaction sets in. Add the remainder of the bromide slowly and at such a rate that the reaction is under control. Continue the stirring until most of the magnesium has passed into solution (about 4 hours). Add 27 g. of A.R. ammonium chloride, and leave the reaction mixture overnight. Cool the flask in ice and add slowly a large excess of dilute hydrochloric acid; the precipitate will dissolve completely. Separate the upper ethereal layer, and wash it successively with dilute hydrochloric acid and water; dry with anhydrous magnesium or calcium sulphate. Distil the ethereal solution through an efficient fractionating column (*e.g.*, a Hempel column filled with $\frac{1}{4}''$ glass rings or $\frac{1}{8}''$ porcelain rings; a modified Hempel column; a 30 cm. all-glass Dufton column; or a Widmer column—see Sections **II,15** and **II,17**). After the ether has passed over, *n*-hexane will distil at 67–70° (13–14 g.).