FURTHER SMALL-SCALE ORGANIC PREPARATIONS

H. HOLNESS, M.Sc., F.R.I.C.
and
C. MURDOCH, B.Sc.

PITMAN

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by

H. HOLNESS, M.Sc., F.R.I.C.
Senior Lecturer in Chemical Engineering, College of Technology, Manchester, 1

and

C. MURDOCH, B.Sc.
Part-time Lecturer in Organic Chemistry, South-West Essex Technical College, Walthamstow, London, E.17

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PREFACE

Twenty Small-Scale Organic Preparations (for "A" level) already published (Pitman, 1958) was confined to the preparation of compounds drawn entirely from the aliphatic series for, at the time it was written, certain examining bodies did not demand a knowledge of the aromatic series of compounds.

Since that time, however, syllabuses have been altered and this book is the natural extension of the previous one; it is confined to preparations drawn from the aromatic series of compounds.

The same simple pieces of apparatus are used as were used for the aliphatic preparations but, since many of the compounds prepared are solids, one extra piece—a small Hirsch funnel—has been used. The preparations are arranged alphabetically as before so as to avoid any suggestion that they form a progressive course of instruction.

The two books, this and *Twenty Small-Scale Organic Preparations*, are together intended to give a comprehensive series of organic preparations, suitable for both the "Advanced" and "Scholarship" Levels of the General Certificate of Education as well as for the Ordinary National Certificate in Chemistry.

In selecting the preparations described in this book the following aims were in mind—

1. To introduce the student to compounds representing as many classes as possible.

2. To cover a number of the better known "named" reactions.

3. To show the preparation of isomers where different routes are necessary.

All the preparations have been tested on the bench and the times and yields quoted are those representative of an "average" student.

We have included in the book details of the Lassaigne sodium fusion test, thereby fulfilling, in the two books, all the needs for an elementary course in practical organic chemistry.

We should like to place on record our thanks to the many students who have helped us in arriving at this final form of the preparations described.

College of Technology, Manchester. H. H.

South-West Essex Technical College, London, E.17. C. M.

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APPARATUS

IN addition to the apparatus in general use for inorganic qualitative analysis using the semi-micro technique,* the preparations described in the following pages need a few simple pieces of apparatus normally available in any laboratory and a set of five pieces of special apparatus. These are described below—

Simple Apparatus from the Laboratory

a 100-ml beaker.
a 250-ml conical flask (as used for titrations) fitted with a two-holed rubber bung carrying delivery tubing arranged to form a steam generator.
a 240°C thermometer.
a 600-ml beaker or similar vessel for use as a water bath.
a stirring rod—4 in. by 5 mm.
a 3-in. clock glass.
a 4-in. length of 5-mm glass tubing.
a 4-in. length of 7-mm glass tubing bent at right angles.
a 3-in. calcium chloride U-tube.
a 9-in. length of 5-mm glass tubing bent at right angles 2 in. from one end.
a 6-in. length of rubber tubing, $\frac{3}{16}$ in. diameter.
a 1-in. length of thick-walled rubber pressure tubing, $\frac{1}{8}$ in. internal by $\frac{7}{16}$ in. external diameter.
a 75-ml porcelain evaporating basin.
a tripod and gauze.
a retort stand, clamp and boss.
a No. 5 (29 mm) porcelain Hirsch funnel.

* For a description, see Holness: *Inorganic Qualitative Analysis by Semi-Micro Methods* (Pitman).

*Special Apparatus**

1. a pear-shaped flask, 30–40 ml capacity.
2. a 7-in. long "cold finger" condenser fitting into (3).
3. a jacket with a side-arm.
4. a twin-necked adaptor head.
5. a 9-ml graduated receiver tube with side-arm.

These are either fitted with B.14 ground joints, or are made to accommodate the same sized corks thereby achieving complete interchangeability. The five pieces are shown in Fig. 1 and the versatility of the apparatus is illustrated in Figs. 2–9.

With this apparatus all the basic processes of organic preparative chemistry can be carried out as the preparations described in the succeeding pages show. The yields obtained in some cases do not permit the product to be redistilled but the quantity obtained is adequate for the student to carry out the usual tests on the substance. Should a particular product be required in a purified state then the separate yields from each student can be "pooled" and purification done as a class experiment.

* Available from The Eureka Scientific Co. Ltd., Ilford Lane, Ilford, Essex.

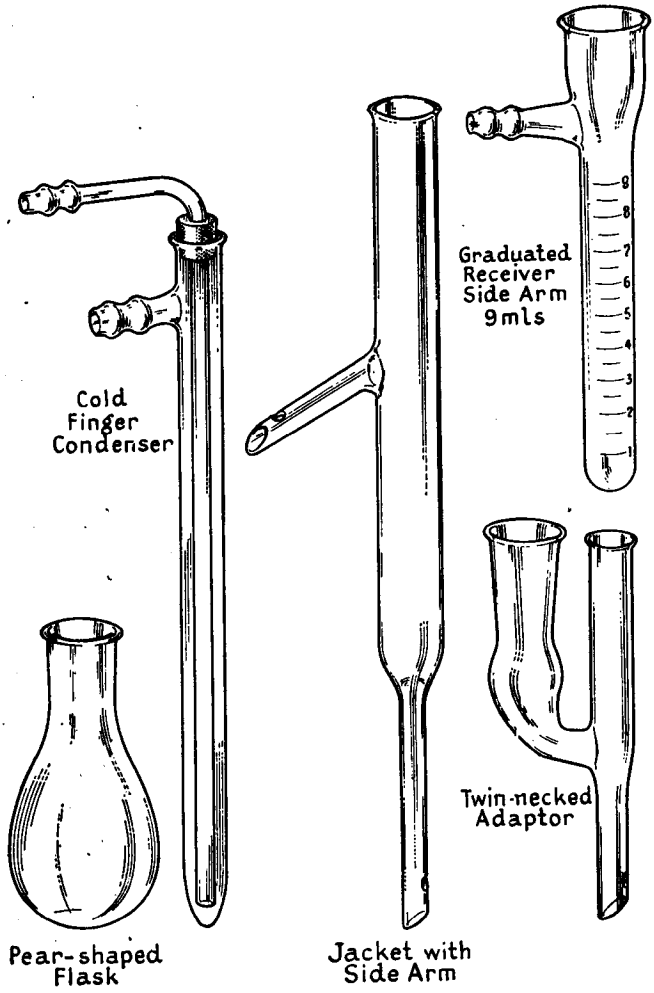
Cold
Finger
Condenser
Graduated
Receiver
Side Arm
9mls
Twin-necked
Adaptor
Pear-shaped
Flask
Jacket with
Side Arm

FIG. 1

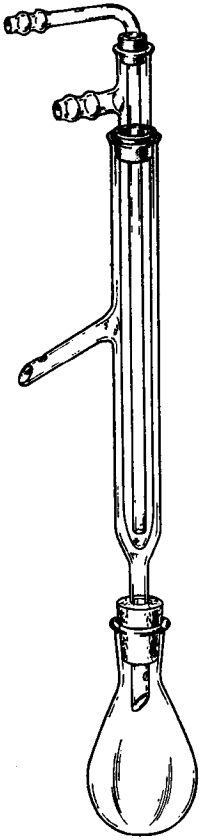

FIG. 2

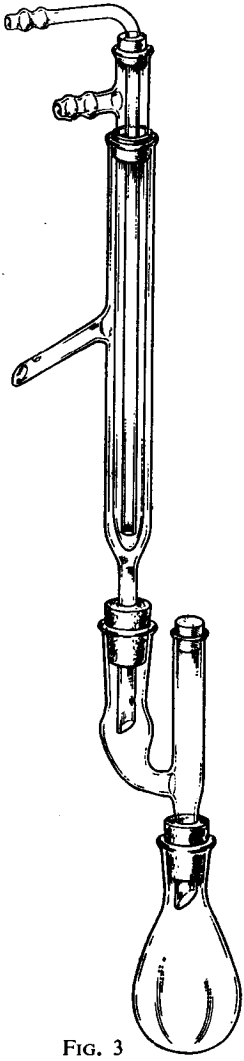

FIG. 3

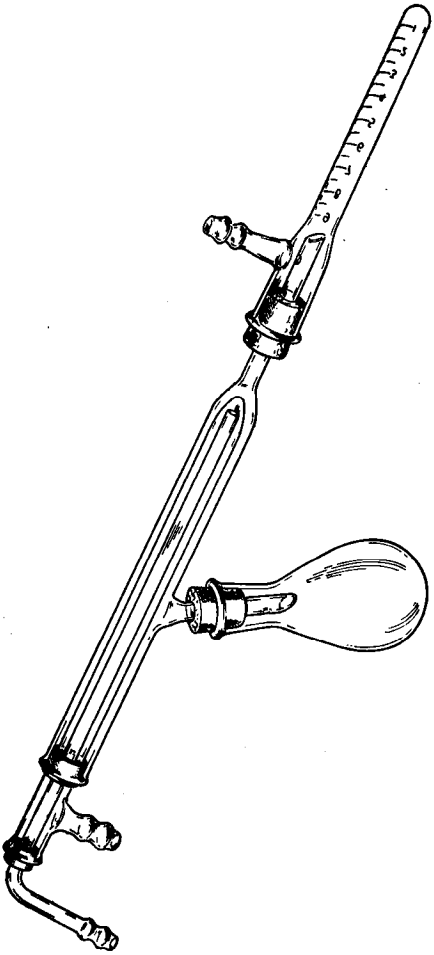

FIG. 4

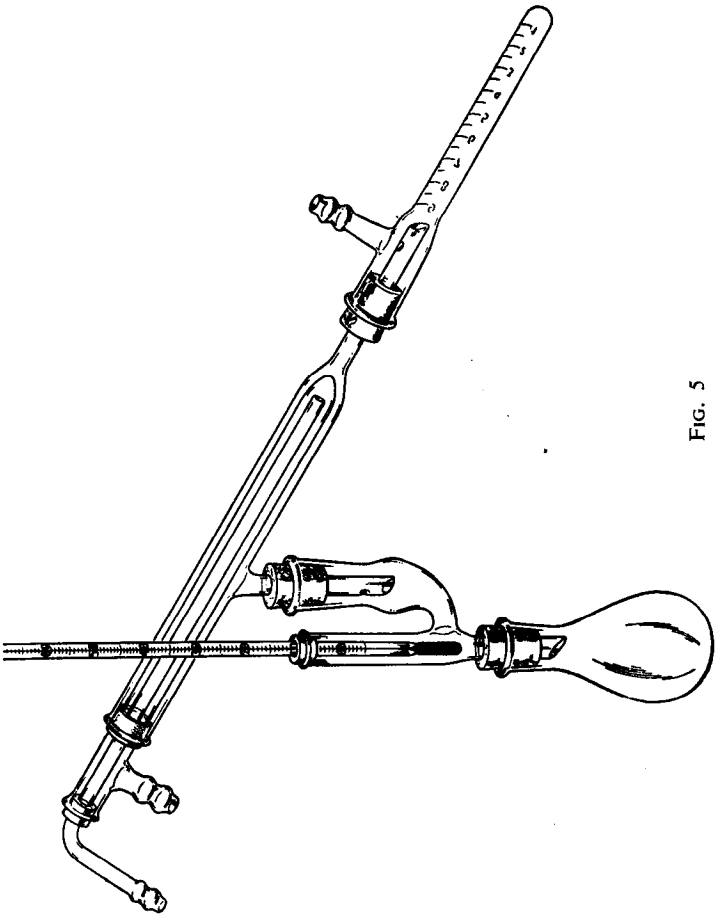

FIG. 5

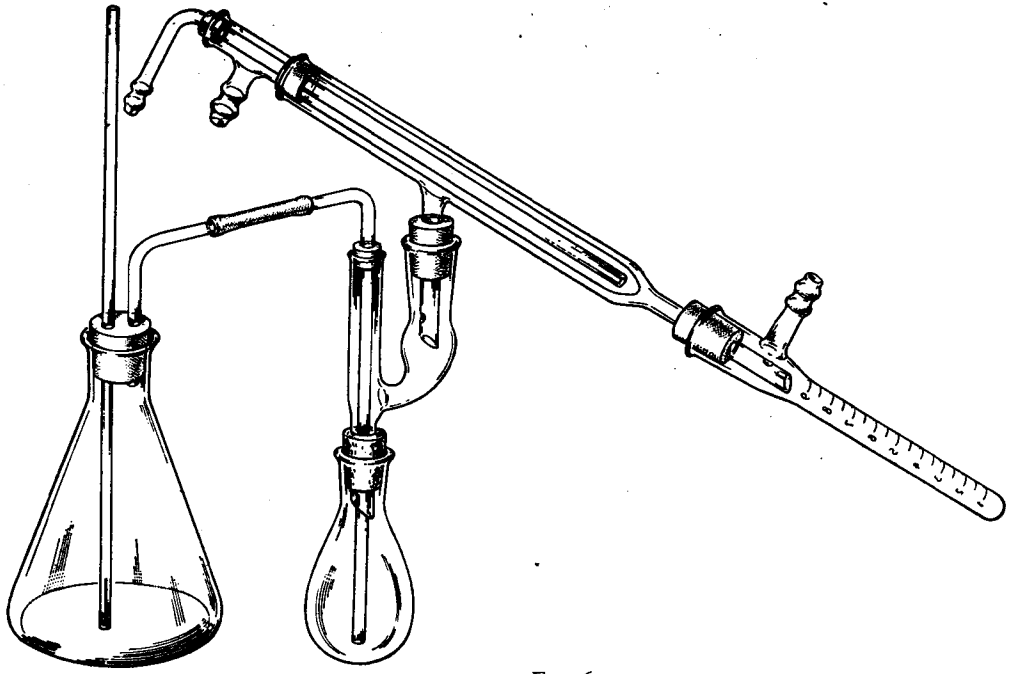

FIG. 6

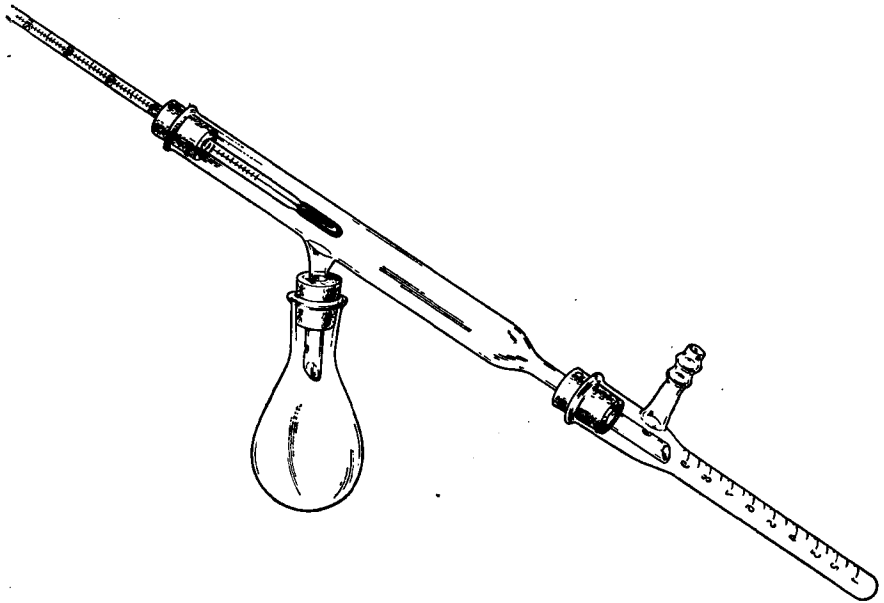

FIG. 7

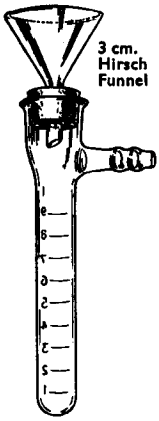

FIG. 8

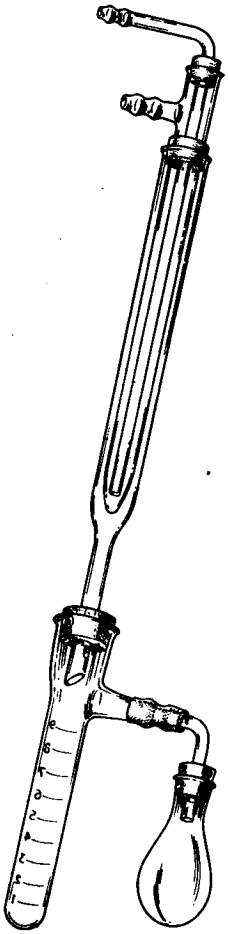

FIG. 9

ACETANILIDE

($C_6H_5.NH.CO.CH_3$; M. Pt. 113°C)

1 ml Aniline
1·5 ml Acetic Anhydride

Put into the dried flask 1 ml of aniline and add to it, cautiously and with gentle swirling, 1·5 ml of acetic anhydride. Attach the flask to the condenser system arranged for refluxing (Fig. 2, p. 4) and heat in a bath of boiling water for 15 min.

After heating, pour the contents of the flask into 70 ml of cold water held in the 100-ml beaker and warm the water until the crude acetanilide dissolves. Now cool the beaker in ice water and filter the fine crystals, using the Hirsch funnel and receiver tube (Fig. 8, p. 9). Wash with 10 ml of cold water, drain and press the crystals between filter paper to dry them. (*Time:* 70 min. *Yield:* 0·7 g of purified compound.)

ACETOPHENONE

($C_6H_5.CO.CH_3$; M. Pt. 20°C; B. Pt. 203°C)

3 g Anhydrous Aluminium Chloride
7·5 ml Benzene (dried over sodium)
2 ml Acetyl Chloride

Put into the dried flask 3 g of finely ground, anhydrous aluminium chloride, add 7·5 ml of pure benzene previously dried over sodium and mix well. Fit the flask with adaptor head and condenser arranged for refluxing (Fig. 3, p. 4) and attach a length of rubber tubing to the side-arm of the condenser jacket. To the other end of this rubber tubing fasten a funnel with its mouth in a beaker containing a little water and arranged so that the subsequently-evolved hydrochloric acid fumes are dissolved without the risk of a "suck back."

Put the flask in a bath of cold water and, by means of the dropping pipette, introduce in 0·5 ml portions through the narrower neck of the adaptor head, 2 ml of acetyl chloride. Immediately replace the cork after each addition and allow the vigorous reaction to subside before adding the next portion. After each addition gently swirl the flask to mix the contents.

Raise the temperature of the water bath to 50°C and maintain it at this temperature for 30 min, swirling the flask at intervals to keep the reactants well mixed. After this period of heating, cool the flask in water and then pour the contents carefully into 20 ml of water held in the 100-ml beaker. Cool the beaker in water and mix the two liquid layers using the dropping pipette for the purpose—fill the pipette with some of the upper benzene layer, then lower the tip into the aqueous layer and vigorously eject the contents; repeat this procedure several times to ensure a thorough mixing. Allow the mixture to separate and, using the pipette, remove and discard the lower aqueous layer. Wash the benzene layer with 10 ml of a

5 per cent aqueous solution of sodium hydroxide using the dropping pipette and the technique described above. Remove and discard the lower aqueous layer, add two or three small pieces of anhydrous calcium chloride to the benzene layer and allow it to dry for 15 min.

Dry the flask and pour into it the dried benzene solution, fit the flask with adaptor head and thermometer and arrange it for distillation (Fig. 5, p. 6). Distil off the benzene and, when it has all been removed, heat the flask strongly to drive over the acetophenone as quickly as possible. Collect in a dry semi-micro test-tube the fraction boiling in the range 198–203°C. (*Time:* 105 min. *Yield:* 1·4 ml.)