quite easily made, either from a piece of glass tube or a solid glass rod, as may be preferred.

When made from a piece of tube, the end is closed by rotating in the flame and is slightly blown out. It is then reheated at the extreme tip and when the pressure on the inside is reduced, a small dimple, or bellshaped depression is formed in the end. When made from a glass rod the depression may be formed with a piece of pointed hard carbon, care being exercised in either case to form the depression exactly in the end of the rod, the size and depth of the depression being rather unimportant. For quantitative work it should be of such size that any adhering precipitate can be easily removed with a small piece of filter paper.

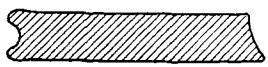

Fig. 1.—A boiling-stirring rod to prevent bumping.

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NORMAL BUTYLBENZENE

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During a study[1] of certain derivatives of normal butylbenzene it became necessary to prepare a considerable quantity of that material. Of the two methods available for the preparation, the Fittig and the Grignard, the former was chosen due to the more readily available materials.

Descriptions and comments regarding this reaction found in the literature do not encourage attempts to conduct the operation on a moderately large scale in the laboratory, yet it may be so carried out that the high yield and simplicity of operation leave little to be desired.

Experimental.—A copper kettle, the size of a gallon (3.8 liter) pail, was made of heavy material and fitted with the tinned iron friction ring and cover of such a pail. Three holes were cut in this cover and connections about 2 cm. in length and diameter soldered in place. The outlets were fitted with a dropping funnel, a thermometer well, and a reflux condenser (preferably an all-metal one) and the kettle was placed in a bath of running water so that it was immersed to within 5 cm. of the top.

Four hundred and sixty g. of sodium was prepared by rolling under a cement lawn roller, or slicing into pieces 2 mm. thick, and placed in the

[1] This study is directed towards the determination of the disinfectant power of phenol as influenced by groups placed in the various positions about the ring. The writers are indebted to Treat B. Johnson of Yale University for opening the field to them.

kettle. Sufficient dry ether[2] was added to cover the sodium (about 1 liter being necessary) and allowed to stand until any evolution of hydrogen had ceased. There was then added through the funnel a mixture of 1170 g. each of *N*-butyl bromide[3] and bromobenzene at the rate of 100 g. per hour. The temperature remained at or below 20° depending on that of the bath. No refluxing occurred. If refluxing had commenced it would have indicated too vigorous an action and the danger of loss of control. After two days the reaction was considered completed.

The recovery of the product was carried out in one of two ways, the second producing the better yields.

1. The water was removed from the bath and 300 cc. of alcohol added through the funnel as rapidly as refluxing would permit. Refluxing was continued for three hours by the use of hot water in the bath, while the remaining bits of sodium were decomposed with water. Sufficient water was added to bring the sodium bromide into solution and the contents of the kettle were removed to a 5-liter flask where the upper oily layer was washed with water. The product was dried over calcium chloride and fractionated.

2. The contents of the kettle were extracted with several portions of benzene and the combined extracts fractionated.

The extractor was made from a bell jar with neck, supported neck down over a 1-liter flask. The two were connected by a 1 cm.-bore glass tube fitted with cork stoppers. In the jar were placed a coarse wire gauze and over this a loosely fitting filter paper bag. On the open top was set a round-bottom flask fitted as a condenser.

The fractionation was effected with a Clarke-Rahrs type of column[4] electrically heated to about 150°, the crude product being collected between 160° and 185°. A second fractionation gave pure *n*-butylbenzene, boiling at 181–184°; yield, 775–825 g.

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[2] Conveniently prepared by the cautious treatment of ether under a reflux condenser with half of its weight of concd. sulfuric acid, and subsequent distillation.

[3] "Organic Syntheses," John Wiley and Sons, Inc., New York, N. Y., **1923**, Vol. I, p. 5.

[4] Clark and Rahrs, *Ind. Eng. Chem.*, **15**, 349 (1923).