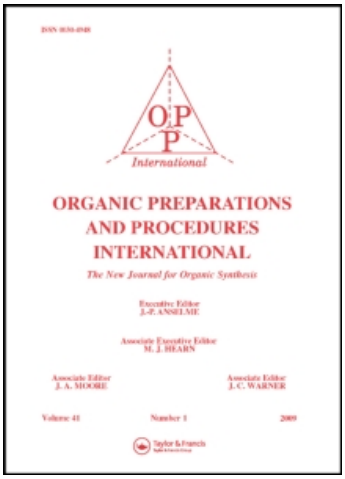

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A SIMPLIFIED APPARATUS FOR GENERATION OF DIAZOMETHANE

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A SIMPLIFIED APPARATUS FOR GENERATION OF DIAZOMETHANE

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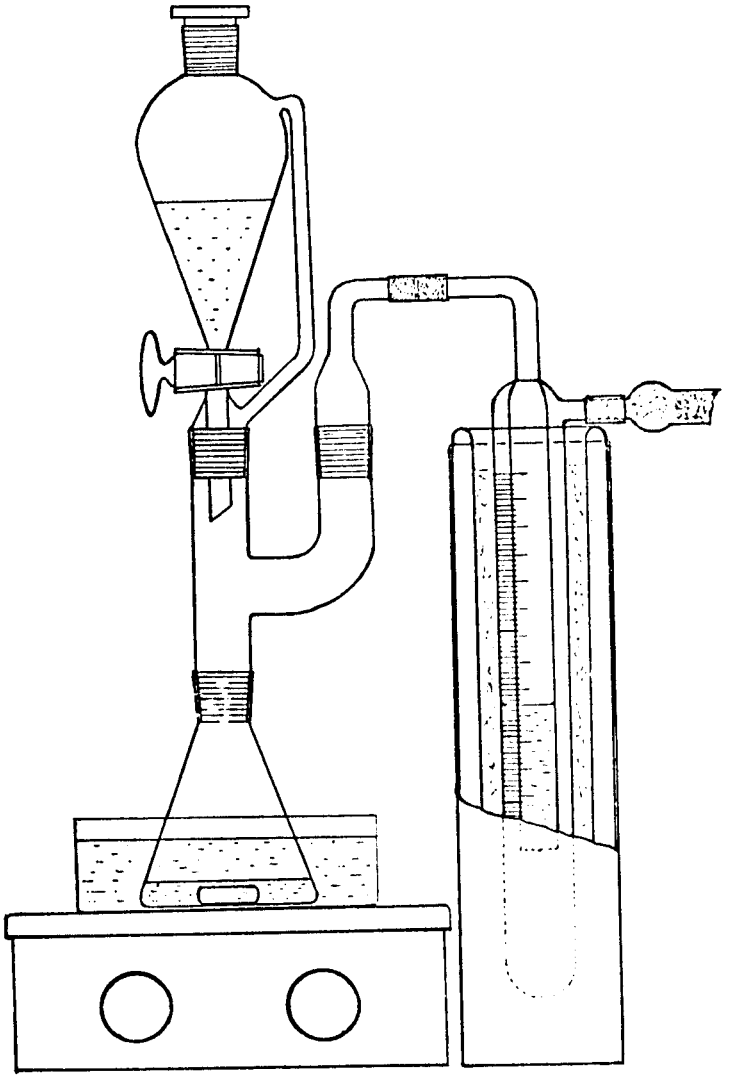
In 1980, a new apparatus for laboratory preparation of diazomethane was described by the author of this paper.[1] Its main feature was a special Dry Ice reflux condenser fitted with a Teflon^{R} stopcock and an overflow trap. The apparatus was adopted and is being manufactured by Aldrich Chemical Company, which is now offering a microapparatus for the preparation of small amounts of diazomethane. The use of the fairly expensive special Dry Ice reflux condenser in the aforementioned apparatus can be avoided in a modified apparatus which is shown in Fig. 1 and which can be assembled from conventional ground glass equipment (Kem Kit).

The apparatus consists of a 125 ml ground glass Erlenmeyer flask immersed in a water bath and fitted with a Teflon^{R} coated magnetic stirring bar. A Y piece inserted into the neck of the flask holds a pressure-equalizing separatory funnel and an adapter connected by means of Tygon^{R} tubing to a Dry Ice trap immersed in a Dry Ice-acetone bath in a Dewar flask and protected from moisture by a Drierite guard tube. The ground glass joints should be lightly greased and should not be moved or rotated during the operation. The procedure for the preparation of diazomethane is essentially the same as described in the literature.[1,2]

Caution. Diazomethane is toxic and potentially hazardous (see warning in ref. 1). It is essential to grease all the ground glass joints when the apparatus is assembled and not to move them during the operation.

Scratched and etched flasks and all sharp edges must be avoided (glass tubes used must be flame-polished). The apparatus should not be exposed to strong light, and the operation should be carried out behind a glass shield or a hood door with safety glass. It is imperative to wear rubber gloves and face shield or safety goggles during the whole procedure.



A solution of 21.4 g (0.1 mol) of Diazald^{R} (Aldrich Tradename) in 125 ml of ether is added dropwise into a stirred solution of 9 g (0.136 mol) of commercial potassium hydroxide* in 15 ml of water, 35 ml of Carbitol (diethylene glycol monoethyl ether) and 10 ml of ether while the water bath is heated to 60°-70°. When all the solution has been added, an additional portion of 30 ml of ether is added from the separatory funnel into the flask and heating is continued until no more ether distils. The ethereal solution is collected in a Dry Ice trap without any loss of diazomethane or ether. The whole operation requires 30-45 minutes and about 0.5 kg of Dry Ice. If a graduated Dry Ice trap is used, the desired amount of the ethereal solution of diazomethane can be measured directly into reaction vessels for subsequent reactions. Approximately 60% yield of diazomethane based on Diazald^{R} was obtained in a series of experiments.

The advantage of the apparatus described in this paper over that described in Organic Syntheses[2] is in the more complete and faster condensation of the diazomethane solution. This shortens considerably the time necessary for generation of larger amounts of diazomethane. Compared with the author's apparatus described earlier,[1] the present design does not require special equipment but can be improvized from current laboratory parts. The use of Dry Ice traps of different volumes makes the procedure suitable for the generation of 0.02-0.2 mol of diazomethane.

REFERENCES

1. M. Hudlicky, J. Org. Chem., 45, 5377 (1980).

2. T. J. De Boer and H. J. Backer, Org. Syn. Coll. Vol. 4, 250 (1963).

*Commercial potassium hydroxide is a hemihydrate which contains 85-86% of KOH.