

not shown in the figure. If desired the water jackets may be blown on to the pump, but in practice I find it preferable to attach the water jackets by means of rubber stoppers as described. The pumps may be constructed of any glass resistant to temperature change. The use of ordinary soda glass or lead glass is not to be recommended. Pyrex glass may be employed but I have found that another glass¹ works much more readily than pyrex glass while at the same time it has as low a coefficient of expansion. This glass reduces slightly in the ordinary blow-pipe flame and is somewhat less fusible than ordinary soda glass and requires a larger supply of air. Simple glass blowing operations can be carried out in an ordinary blow-pipe flame. If it is desired to avoid reduction or to carry out more difficult operations in glass blowing, it is preferable to employ oxygen in finishing the operation.

Any good water-jet pump is satisfactory as supporting pump. In practice I have found it convenient to construct my own pump using G-702-P glass for the purpose. The effectiveness of the water-jet pump depends largely on the relative diameter of the nozzle and the delivery tube. A nozzle having an interior diameter of 2 mm. and a delivery tube having an interior diameter of 5 or 6 mm. will give a pump which operates very well, and, if necessary, it is easy to construct a pump which operates at a pressure below 40 pounds. The delivery tube should be simply a straight tube about 20 to 25 cm. long. It is essential that the delivery tube and the nozzle be carefully lined up, as the pump will not operate properly if the jet strikes the side of the delivery tube. A bottle should be introduced between the water-jet pump and No. I supporting pump and it is also convenient to introduce a manometer. To start the pumping operation, the apparatus to be exhausted is joined to tube W by means of a ground joint or by cement. The water-jet pump is then put in operation and when the pressure has fallen to about 25 mm., the burners are lighted. If these burners have been previously adjusted, the pump requires no further attention. The pump will begin to operate within several minutes after lighting the burner. I have been able to exhaust a volume of about 1500 cc. to less than 10^{-4} mm. of mercury in about 10 minutes.

WORCESTER, MASS.

NOTE.

An Improved Hydrogen Chloride Generator.—Of the numerous methods employed to generate hydrogen chloride for lecture table experiments, and for investigations, none are entirely satisfactory, the chief objection to most of them being that the quantity of gas evolved cannot easily be controlled, or is not uniform. A careful quantitative study of all

¹ Manufactured by the Corning Glass Works of Corning, N. Y., G-702-P'.

the methods in use developed the fact that the one which introduced hydrochloric acid (sp. gr. 1.18) under sulfuric acid (sp. gr. 1.84) gave the best results.

After considerable experimenting the apparatus pictured in Fig. 1 was found to be very satisfactory. It consists of a large separatory funnel, of one-liter capacity, with the stem cut off short. The tall, narrow form, as shown, gives the best results. It is fitted with a two-holed rubber stopper. One of the holes contains a Y-tube, one limb of which is provided with a plug in order that air may be introduced into the train; the other limb is the gas exit. The second hole contains the piece marked A. This consists of a small dropping funnel sealed to a capillary tube about 40 cm. in length. It is important that this tube be a capillary so that it will fill with acid, and thus give the hydrostatic pressure necessary to force the hydrochloric acid into the sulfuric acid. The spent acid is drawn off at B. The entire apparatus is supported by a ring stand.

The apparatus described behaves splendidly. The flow of gas is perfectly uniform at all times. The rate of flow is so easily controlled by the stopcock C, that any amount of gas that will ordinarily be needed, can be delivered. The gas is dry. The apparatus, even when producing a rapid stream, does not heat up to any extent. The piece is easily charged and discharged. The spent acid is only diluted, does not contain much hydrochloric acid and can be used for other purposes in the laboratory. The danger of sulfuric acid spray being carried over is greatly reduced. When the stopcock is closed the gas flow ceases at once and when the stopcock is opened the gas flow starts immediately. On the whole, the apparatus is more satisfactory than any of the others tried.

In actual use the first 100 cc. of hydrochloric acid gave off 32.7 g. of gas, the second 100 cc. gave off 27 g. of gas, and the third 100 cc. gave only 2 g. This indicates that equal parts of the two acids are the best proportions to use. If much more than this proportion of hydrochloric acid be added the gas will continue to come off for a time after the stopcock is closed.

It was observed that when 200 cc. of hydrochloric acid were introduced into 200 cc. of sulfuric acid, 59.7 g. of hydrogen chloride were expelled;

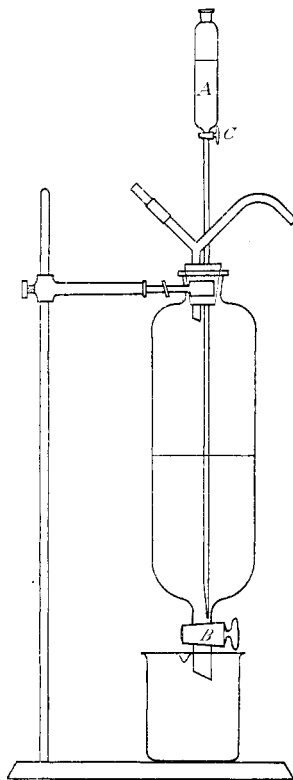


Fig. 1.

when 200 cc. of sulfuric acid were added to 200 cc. of hydrochloric acid only 49 g. were liberated. If each experiment had been given ample time to reach equilibrium the two values would, of course, be the same. In the former case equilibrium was reached very much sooner than in the latter, and it is due principally to this fact that the first method is so much superior to the second.

It has been observed¹ that dry hydrogen chloride acts strongly upon rubber, producing, among other things, various sulfur compounds. After the rubber has been exposed for some time, however, it is only slightly attacked. For accurate work, nevertheless, the rubber stopper should be replaced with a glass part.

O. R. SWEENEY.

CHEMICAL LABORATORY,
OHIO STATE UNIVERSITY.

[CONTRIBUTION FROM THE LABORATORIES OF THE ROCKEFELLER INSTITUTE FOR MEDICAL RESEARCH.]

ON NITRO- AND AMINOPHENOXYACETIC ACIDS.

BY WALTER A. JACOBS AND MICHAEL HEIDELBERGER.

Received June 27, 1917.

In the course of synthetic work which was undertaken in connection with investigations to be published later, it was found necessary to prepare a large number of substituted aminophenoxyacetic acids and their derivatives. A perusal of the literature showed that only a few of the simpler members of this series had been described, so that the preparation and study of these substances became a separate undertaking. We wish, therefore, to present the results of this work in the present communication.

At first we adopted the procedure of starting with the appropriate nitrophenol, converting this into the nitrophenoxyacetic acid, and then into the desired amino derivative. In preparing the nitrophenoxyacetic acids it was found most satisfactory to employ the method used by Kym² for the preparation of *p*-nitrophenoxyacetic acid. This involves heating the dried sodium salt of the nitrophenol with ethyl chloroacetate and saponifying the nitro ester so obtained. Although this method is somewhat more time-consuming than the reaction between the nitrophenol and chloroacetic acid in boiling alkaline solution it proved to be more economical in the case of the less easily accessible nitrophenols, since the yields of the esters were practically quantitative.

Later in the work an attempt was made to prepare the nitrophenoxyacetic acid from 4-nitroguaiacol, but no reaction took place between the dried sodium salt and ethyl chloroacetate. It was found, however, that

¹ THIS JOURNAL, 36, 2366 (1914).

² Kym, *J. prakt. Chem.*, [2] 55, 113 (1897).