# A NEW PORTABLE GAS GENERATOR.

By A. W. Browne and M. J. Brown.

The numerous forms of apparatus designed for the generation on a laboratory scale of gases obtainable by the action of a liquid (usually an acid) upon a solid substance, may be divided into four classes[1].

(1) Generators in which a surface of the entire mass of acid is permitted to come into contact with the solid substance, and in which no provision whatever is made for the separation of the spent solution from the fresh acid.

(2) Generators in which the acid and solid are brought together as above, but in which the higher density of the spent solution is employed in effecting a partial separation of this solution from the fresh acid.

(3) Generators in which the acid is caused to drop gradually upon the solid and in which no provision is made for the automatic replacement of the consumed acid.

(4) Generators in which the acid drops upon the solid, but in which automatic replacement of the consumed acid facilitates generation of the gas at approximately constant pressure.

An advantage often possessed by generators of the first and second classes over those of the third and fourth is the comparative simplicity and inexpensiveness of their construction. Over those of the third class, they frequently possess also the additional advantage of supplying the gas at a more nearly constant pressure. Generators of the third and fourth classes, on the other hand, are as a rule more economical than the others in that they more efficiently accomplish the neutralization of the acid. The operation of recharging apparatus of the third and fourth classes with fresh acid, or of discharging the spent solution, may in general be performed with greater convenience than is possible in the case of apparatus of the first and second classes. In fact, the operations of charging and discharging, conducted either automatically or semi-automatically in certain forms of apparatus may readily be performed without interrupting the generation of gas.

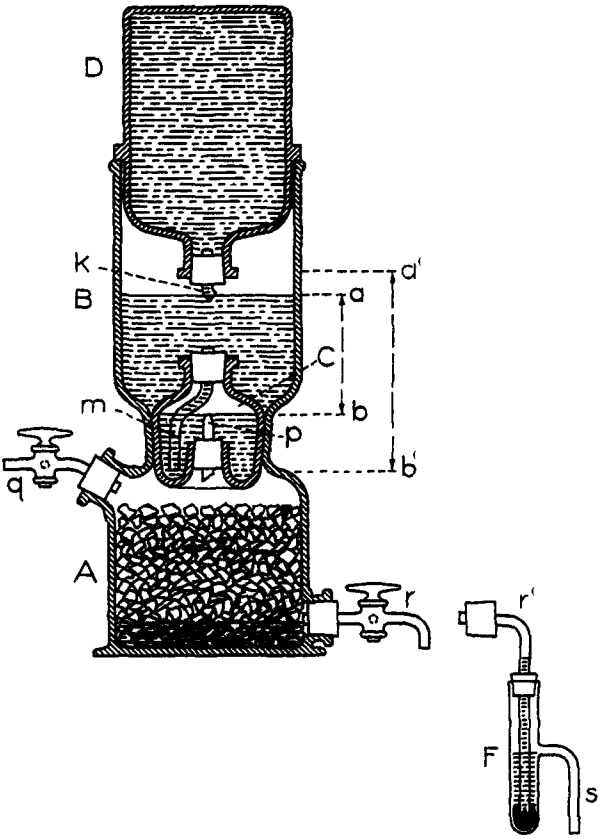
In the attempt to devise a comparatively simple desk generator of the fourth class, the authors have constructed the apparatus[2] shown in the accompanying sketch. This generator, which may be regarded as a portable modification of the larger apparatus designed by Browne and Mehling[3], consists essentially of the tubulated glass cylinder AB, into the constriction of which is ground the glass receptacle C, and into the top of

[1] The generators designed by Kipp, Wollny (Z. anal. Chem., **24**, 214 (1885)), Ostwald (Ibid., **31**, 180-186 (1892)) and Parsons (This Journal, **25**, 231-236 (1903)), might serve respectively as typical representatives of the four classes.

[2] The apparatus was manufactured for the authors by Messrs. Peters and Rost, Berlin.

[3] This Journal, **28**, 838-845 (1906).

which loosely fits the flanged bottle D[1]. The lower compartment A of the cylinder is filled with the solid substance (for example, with calcium carbonate, zinc or ferrous sulphide) which is introduced either through the upper tubulus, or in case the pieces of solid are unusually large,




through the constriction before C has been put in place. The receptacle C is provided with two one-hole rubber stoppers. Through the upper stopper passes a glass tube of 6 mm. inner diameter; through the lower stopper passes a shorter tube tapered at the upper end to a diameter of about 1.5 mm., and beveled at the lower end to prevent the retention of a column of liquid by capillary attraction. Before C is finally set into position the ground surface is thoroughly covered with a thin coating of grease composed of two parts of hard paraffin to ten parts of vaselin.

When C is set into position, care must be taken not to press down upon it so hard that great difficulty will later be experienced in removing it. On

[1] Parsons, loc. cit.

the other hand, care must be taken to have it fit securely, in order to prevent it from being temporarily forced out of position by an abnormally sudden increase of pressure in the apparatus. After the stopcocks *q* and *r* have been closed, acid is poured into B until it reaches the level *a*. The bottle D is filled with acid and is put into position.

When the generator is set in operation by opening the stopcock *q*, acid flows from B through *m* into C and thence through *p* into A, where it comes into contact with the solid substance. So long as *q* is left open, the acid continues to drop intermittently upon the solid. The acid in *B* can never fall below the level *a*, however, since new acid is automatically fed from D to replace that which has been consumed. The bottle D is provided with a rubber stopper through which passes a short, slightly bent piece of glass tubing *k*. This has the double purpose of retarding the outflow of acid from D at the moment of setting the bottle in position over B, and of preventing the occasional entrance into D of gas bubbles forced out of C through *m* after the apparatus has been subjected to abnormal usage. This second purpose might have been equally well accomplished by bending *m* at right angles above the rubber stopper, in which case it would have been possible to dispense with *k* and the rubber stopper through which it passes; but by leaving *m* as shown in the figure, the recharging of the apparatus with solid is made feasible, even when B, C and D are partly filled with acid. In case it is desired to recharge with solid under these conditions, it is necessary only to lift out D, and to insert into the upper end of *m* a long glass rod tipped with a short piece of rubber tubing, in order to prevent acid from dropping into A when the rubber stopper is removed.

When the stopcock *q* is closed after the generator has been in operation for some time, the excess of gas forces the acid out from C through *m* into B, raising the acid in B slightly above the level *a*, and lowering the acid in C below the level *b*. The pressure prevailing under these conditions is therefore somewhat greater than the normal pressure of the apparatus. When the acid in C has been completely displaced by the gas, the pressure will reach the magnitude represented by the line *a'b'*. Any additional gas that may be generated will now pass upward through *m* and bubbling through the acid in B will finally escape beneath the loosely fitting flange of D. The volume of excess gas thus generated after the stopcock *q* has been closed depends upon several factors: (1) upon the diameter of the opening in *p*; (2) upon the promptness with which the acid acts on the solid, and (3) upon the speed with which the gas is withdrawn from the apparatus. (1) The greater the diameter of the opening in *p*, the larger will be the amount of acid automatically introduced into A to compensate the reduction in pressure brought about by withdrawing

a certain amount of gas from the apparatus. If the stopcock $q$ is closed directly after the automatic addition of a large quantity of acid, a comparatively large excess of gas will be evolved. (2) If the action of the acid upon the solid is prompt, the influx of acid will be quickly checked and the excess of gas generated will be comparatively small. (3) If large amounts of gas are suddenly withdrawn from the apparatus, acid will be supplied with greater rapidity than if the apparatus is less severely taxed. In short, the conditions favoring the generation of a minimum excess of gas are obtained when the aperture in $p$ is as small as possible, when the action of the acid upon the solid is as prompt as possible, and when the gas is drawn from the apparatus with a speed not exceeding certain limits and fluctuating as little as possible. It is found in actual practice, however, that if the aperture is of a diameter less than one mm. capillary attraction may prevent the free flow of acid except when the pressure of the column of acid is appreciably higher than the pressure prevailing in A. With regard to the amount of gas that may be drawn from the apparatus, it may be said that so long as the demand is nearly constant and within the maximum capacity of the generator, the gas is furnished at a fairly constant pressure, although a momentary rise of pressure sometimes amounting to several centimeters of water may occasionally be noticed when the acid falls upon the solid. To obtain the steadiest stream of gas, the speed should be checked (either by partially closing the stopcock, or by introducing a short piece of capillary tubing[1] between the generator and the delivery tube) to about four bubbles per second. If the consumption of gas is large and very irregular, the pressure at the time of first opening the stopcock will of course be somewhat higher than the normal working pressure of the apparatus, owing to the raising of the acid surface in B, and the lowering in C, to which reference has already been made. By a combination of circumstances, resulting for example when the aperture in $p$ has been made too large, when unusually large volumes of gas are drawn from the apparatus, and when care has not been taken to press C firmly into place when setting up the apparatus, it is possible that the pressure in $A$ may rise so suddenly that the receptacle C may be forced momentarily out of position. This need occasion no alarm, however, as the receptacle falls at once into place again and may at leisure be pressed firmly into position with the aid of a heavy glass rod after D has been temporarily removed. The spent solution collects in A and may be drawn off as desired through the tube $r$. If it is desirable, however, to permit the apparatus to operate for days at a time without attention, recourse may be had to the automatic device shown in the lower right-hand corner of the sketch. This device is entirely similar in principle to the mercury trap employed

[1] Parsons, loc. cit.

by Browne and Mehling[1], and has given complete satisfaction in this laboratory throughout an extended period of time. It consists of the bent tube *r* which slants downward from the apparatus at every point (to avoid trapping of air bubbles) and the lower end of which dips to any desired depth beneath the mercury contained in the tube F. The spent solution finally flows through the side tube *s*. A more complete description of the contrivance will be found in the article of Browne and Mehling, together with a drawing lettered similarly part for part with the sketch shown in the present article.

The apparatus has been tested in several ways during the past year in this laboratory. In addition to the test of ordinary usage, it has been subjected to several special tests with a view to ascertaining in an approximate way (1) the upper limit of its normal working capacity, (2) its ability to furnish a slow current of gas at constant pressure for a considerable length of time, without receiving any attention and (3) the variation in pressure to which the gas is subject when delivered under varying conditions. The results obtained were briefly as follows: (1) When the opening in *p* was 1.5 mm. in diameter, it was found possible to obtain without difficulty a current of carbon dioxide (from marble and 6 N hydrochloric acid) delivered at a rate of about one liter of gas in two minutes. After the gas had been delivered at this speed for thirty minutes, *q* was closed in order to ascertain whether the excess of gas generated under these conditions would be more than sufficient to fill the receptacle C. The experiment showed that the excess was just about sufficient to fill C. Substantially similar results were obtained in the generation of hydrogen sulphide (from ferrous sulphide and 6 N hydrochloric acid). In the generation of hydrogen (from zinc and 6 N sulphuric acid) the excess of gas produced on closing *q* after the gas had been flowing at the rate of one liter in five minutes for a period of over half an hour was not sufficient to fill C. (2) A current of carbon dioxide (from marble and 6 N hydrochloric acid) at the rate of four bubbles per second was delivered for a continuous period of seven and one-half days without interruption, and without the slightest need of attention. After the completion of this experiment, in which the mercury trap was employed, the only attention required by the generator in order to prepare it for another entirely similar run was the refilling of the bottle D. Moreover, the recharging of D obviously causes no interruption of the current of gas. (3) The variation of pressure which arises from the alternate generation and utilization of an excess of gas stored in C is inherent in generators of this type. The variation is very much greater when the gas is irregularly withdrawn from the apparatus, and reaches a minimum when a slow steady stream of gas is required. In the seven

[1] loc. cit.

day test of the generator, for example, the pressure was substantially constant except for a slight momentary increase experienced whenever a fresh supply of acid dropped upon the solid substance.

## Summary

The apparatus described in the preceding pages possesses the following advantages:

(1) It is capable of furnishing a moderately rapid current of gas at substantially constant pressure.

(2) The spent acid is automatically discharged (by means of the accessory device shown in the figure) without changing the pressure in the apparatus.

(3) A new charge of acid may be introduced at any time without interrupting the generation of gas, by simply filling the bottle D and setting it into position.

(4) A new charge of solid may be introduced without removing the acid in B and C, provided the bottle D is temporarily removed, and a glass rod tipped with a short piece of tubing is pressed into the tube *m*.

(5) No surface of acid or of spent solution is directly exposed to the air, and consequently no fumes are given off.

(6) The acid can not be forced back by the generation of an excess of gas so as to overflow from the apparatus.

(7) In proportion to the desk space it occupies, it has an unusually large available capacity for both acid and solid.

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## A CONSTANT PRESSURE GAS GENERATOR FOR USE OVER A WIDE RANGE OF PRESSURE.

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In the preceding article a portable modification of the Browne-Mehling gas generator[1] has been described. Entirely similar in principle to this device is the apparatus described in the following paragraphs. This apparatus has been employed by the authors for the generation of gases at approximately constant pressures of from 5 mm. to 270 mm. of mercury. In order to avoid unnecessary repetition and to emphasize the similarity in principle existing between this and the other generators devised in this laboratory, the accompanying sketch is lettered similarly part for part with the sketches in the articles to which reference has already been made. The apparatus consists essentially of two Fresenius calcium chloride towers placed base to base and connected by means of a long rubber tube. By raising the tower BD any desired pressure may be obtained and the pressure will in any case be approximately constant,

[1] This Journal, **28**, 838-845 (1906).