gives the black alkali in terms of sodium carbonate in 100 cc. of water.

It is necessary in all cases to use an excess of the standard sodium carbonate, as the calcium and magnesium carbonates, which are precipitated in an amorphous form, are quite soluble. The presence of the excess of sodium carbonate decreases their solubility, due to the presence of the common ion CO_3.

The method is of peculiar value in the examination of irrigating waters, for not only does it show the character of a water as to black alkali, but if the examination shows a water to contain an excess of the soluble salts of calcium and magnesium, the results are scarcely less valuable, for these soluble salts under proper conditions of irrigation are an antidote for black alkali. Hence, if a soil which already contains black alkali is irrigated with water containing an excess of hardness and proper methods of applying the water are used, favorable results are to be anticipated. The application of this principle was first demonstrated, by Hilgard, with the alkali soils of California, upon which he applied gypsum as an antidote for black alkali.

SUMMARY.

(1) Black alkali is not a definite compound but may consist of the carbonates and bicarbonates of any of the alkalies.

(2) Only the alkalinity due to the carbonates and bicarbonates of the alkalies should be expressed as black alkali, and not the alkalinity due to the carbonates and bicarbonates of the alkaline earths.

(3) While not strictly correct, custom and practical considerations sanction the expression of black alkali in terms of sodium carbonate.

(4) The method described yields satisfactory results for black alkali and also for the antidote for black alkali, *i. e.*, soluble salts of calcium and magnesium, which for irrigation purposes should be expressed in terms of calcium sulphate.

A DELIVERY FUNNEL FOR INTRODUCING LIQUIDS UNDER INCREASED OR DIMINISHED PRESSURE.

BY T. J. BRYAN.

Received October 25, 1905.

THE dropping funnel has replaced the thistle tube in cases where a gradual delivery of liquids gives advantage over addi-

tions in quantity. So long as the gases and vapors in the distilling or generating flask have uninterrupted access to the air, there is no difficulty in regulating the delivery to any desired speed by the use of a dropping funnel. At times, however, it becomes necessary (as in cases of the collection or absorption of the gases evolved, or of the protection of the distillate from access of air, etc.) to introduce the use of Woulff bottles, gas washing-bottles, etc., which, as the process of distillation or generation continues, brings about an increase of pressure in the generating flask. This increase of pressure often reaches a point at which it exceeds that produced by the column of liquid in the dropping funnel, and upon opening the stop-cock, not only does the liquid not run down into the flask, but the gases and vapors rise through the funnel, and escape. Attempts to avoid this difficulty, have been made either by filling the tube of the funnel before the operation is commenced,[1] thus increasing the height of the column of liquid; or as is frequently practiced, by inserting this filled tube in a test-tube containing more of the liquid to be delivered. The first method is disadvantageous, especially when the end of the tube is placed in the liquid in the flask, because bubbles of gas or vapor gradually replace the liquid in the tube and necessitate blowing in the top of the funnel or an interruption of the process, to reestablish the desired conditions. The second method, furthermore, is disadvantageous because a quantity of the liquid remains in the test-tube and does not come in contact with the liquid in the flask. Both devices have in all cases the additional disadvantage, that the excess of internal pressure must be less than that of the column of liquid in the funnel.

An apparatus which provides at all times for the same pressure on the upper and lower surfaces of the liquid in the funnel, will evidently solve the above difficulties, for on opening the stop-cock the liquid would then fall by its own weight. Neumann[2] invented an apparatus which intermittently establishes these conditions, and which for some time allows of continuous flow only because of the pressure of a stream of gas delivered into the upper part of the funnel. But Neumann never intended that his apparatus should be used for continuous delivery, for that

[1] Gattermann: "Die Praxis des organischen Chemikers," 1894, p. 156.

[2] J. pr. Chem. [2] 38, 89.

would defeat the very end for which the apparatus was devised, *i. e.*, the introduction of gas into the reaction flask.

It is apparent that a bent tube leading from the flask to the top of the funnel, and properly fitted in rubber stoppers, brings about the desired equality of pressures and admits of either continuous or intermittent delivery from the funnel. The inconvenience of introducing several tubes through one cork; the difficulty of introducing a fresh supply of liquid into the dropping funnel during the reaction with the attendant loss of gases and vapor; and the danger of breaking the apparatus, have led the writer to design the delivery funnel pictured in Fig. 1. It con-

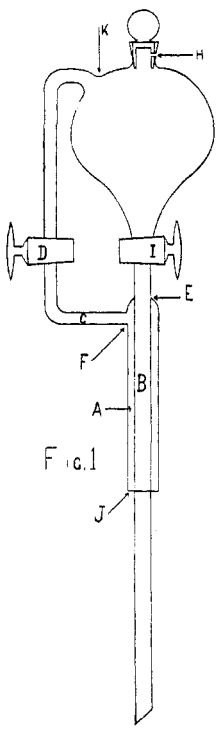

FIG. 1

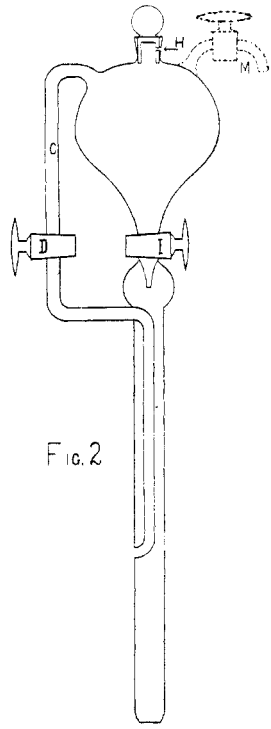

FIG. 2

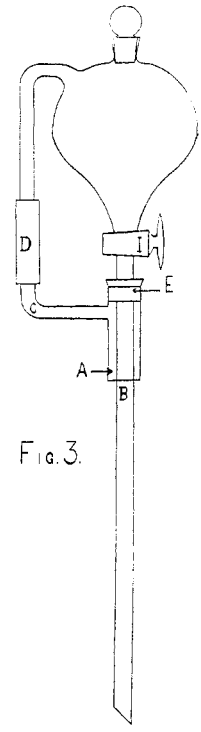

FIG. 3.

sists of a dropping funnel whose tube B passes through and is sealed to the tube A at E. The tube C with a glass stop-cock at D is fused into openings in the upper part of A at F and in the upper part of the funnel at K. The funnel is made with air vent in the stopper.

It is evident from the figure that the working of the apparatus is very simple. The funnel is fitted into the reaction flask by means of a stopper passed over the tube A. When stop-cock I and air vent H are closed, and stop-cock D is open, the same pressure which exists in the reaction flask is exerted through the opening J and the tubes A and C to the upper surface of the liquid in the funnel; and on opening stop-cock I, the same pressure being exerted through B upon the lower surface of the liquid, it flows down through B under a pressure dependent only on the specific gravity of the liquid, the height of its column, and the capillarity of the tube B, which last consideration may be neglected in this connection. The speed of delivery may be regulated by the stop-cock, and the gas pressures on the upper and lower surfaces of the liquid will always remain equal and will allow of either continuous or intermittent delivery so long as a single drop of liquid remains in the funnel. On closing D and I and removing the stopper a fresh supply of liquid may be introduced into the funnel.

In the construction of the apparatus, the lower part of tube B should be of a small bore, for if the capillarity of the opening T in tube A is greater than that of B, a column of liquid formed from condensed vapors is liable to collect in B, somewhat decreasing the pressure in the upper part of the funnel. To further avoid condensation in tube B, its length should be limited to four inches, which gives all the latitude ordinarily desired in regulating the depth to which B shall extend in the flask.

This apparatus though designed by the writer for delivery under increased pressure, is found to be a still greater convenience in working under diminished pressure. In fractional distillation in vacuum, it is frequently desirable to use a very small flask and to distil comparatively large amounts of liquid. If this delivery tube is used, the apparatus may be exhausted while H and I are closed and D is open; it will then be found that on opening I the delivery of the liquid in the funnel can be regulated with the same ease as the flow from an ordinary separatory funnel open at both ends to the air. This quiet delivery instead of the violent spurting which accompanies all attempts to put an ordinary dropping funnel to a similar use, will recommend itself to all who give it a trial. If it is desired, the funnel may be refilled by closing both stop-cocks opening the air vent and removing the stopper. After

filling, it will be necessary to replace the stopper, close the air vent, open the stop-cock D and exhaust before proceeding with the distillation.

Fig. 2 is a modification of Fig. 1 fitted with a Walter's dropping device. The use of the side tube M with its stop-cock, is recommended when it is desirable either to replace air in the apparatus with some other gas, or when it is undesirable to discontinue the distillation in vacuum after refilling the funnel. In the latter case after filling, the funnel and upper part of C are exhausted by attaching a pump to M while D, I and H remain closed; then M is closed, D opened and the conditions are the same as before the flow was interrupted.

A cheaper form of the same apparatus, represented in Fig. 3, gives good results in cases where the reagents used do not attack rubber. This apparatus differs from that in Fig. 1, in that the tubes A and B are joined by means of a rubber stopper instead of being sealed, and the tube C is broken at D and the stop-cock replaced by rubber tubing and a Hofmann's clamp. The length of the tube A is just sufficient to allow its passage through a stopper, thus minimizing the condensation of vapors in A, and the distance that the tube is inserted in the reaction flask is changed by sliding the tube B through the stopper E and adjusting the length of the rubber tube at D. This modification of the delivery tube may be made from a common dropping funnel by any one of ordinary ability at glass-blowing, and is especially adapted to use by students, as the flexibility given by the rubber stopper at E and by the rubber tube at D, reduces the probability of breakage in unskilful hands.

THE UNIVERSITY OF ILLINOIS,
URBANA, ILLINOIS, October 23, 1905.

SOME OBSERVATIONS ON THE PREPARATION OF METALLIC CALCIUM BY ELECTROLYSIS.

BY S. A. TUCKER AND J. B. WHITNEY.

Received October 30, 1905.

THE method for the preparation of this metal used was that devised by J. H. Goodwin,[1] and the attempt was made to improve the yield of the metal if possible.

The apparatus used was similar to that employed by Goodwin,

[1] Proc. Am. Phil. Soc. (Phil.) 43, 381.