A PLATINUM CRUCIBLE FOR CARBON COMBUSTIONS.

By John V. R. Stehman.

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Shimer[1] has shown that a platinum crucible provided with a special water-cooled stopper and rubber gasket, may be used as successfully as the more expensive platinum tube and gas-consuming furnace for carbon combustions.

The prevalent idea regarding his form of apparatus, seems to be a fear of the rubber gasket causing error by burning directly or by becoming hard and brittle, allowing small pieces to drop into the crucible, when the stopper is pushed into place.

In the following form of apparatus designed by the writer, no rubber gasket or washer is used, and, as in Shimer's apparatus, in place of the furnace, a Bunsen burner or blast-lamp is used, and air in place of oxygen.

It consists, as seen in the drawing, of three parts, *A*, *B* and *C*, also an asbestos washer, *d*. *A* is a water-cooled brass cup. *B* is the cup casing or crucible support, also made of brass. Part *C* is the platinum crucible, provided with a flange around the top, and was remodeled from a 30 cc. crucible. This special shape increases its length and allows over 1 inch to be brought to a full red heat when placed over the burner.

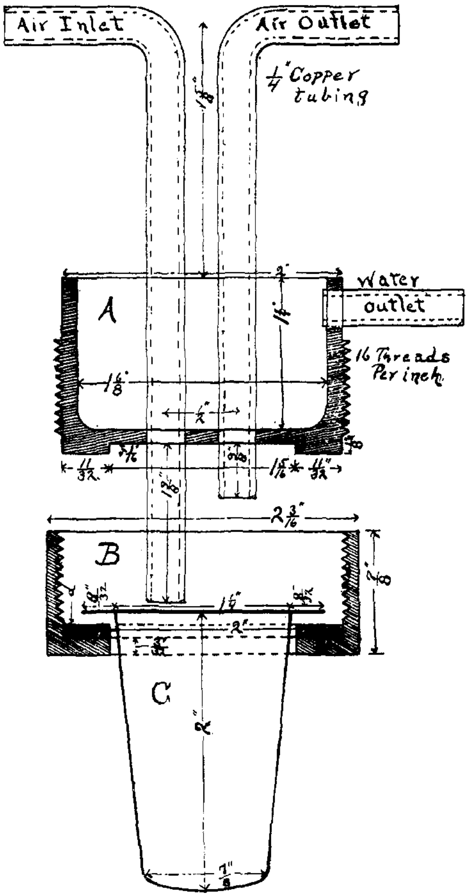
Part *d* is an acid-washed asbestos washer, made from a piece of asbestos wick yarn, of such a length as to allow the beveled ends to lap for about one-half inch. It is moistened and laid in place on the flange of casing *B*, then pressed into place by screwing cup *A* down upon it, the crucible being left out of place. This will force out the excess of moisture in the washer and flatten it. When the washer presents an even surface and has filled the flange of *B* full, it is ready to receive the crucible.

The crucible is now dropped into place, its flange resting upon the washer, and the cup *A* screwed down, using a gentle but firm pressure. That part of the cup *A*, which rests partly upon the flange of the crucible and partly upon the asbestos washer, is turned true, and polished, and coming in contact with the crucible flange and moist asbestos washer insures an air-tight joint. If water is now allowed to circulate through the cup at about 120 cc.

[1] This Journal, 21, 557.

per minute the washer will remain moist when the crucible is heated with the blast-lamp, the casing and cup being protected by the asbestos board.

Two washers may be used, one above and one below the crucible



flange, or the flange of the crucible may rest directly upon the casing flange and the washer placed on top of the crucible flange. The author has used a pure rubber washer, letting it rest directly upon the flange of the crucible, which in turn rested upon the casing flange of *B*, but it was found that the blank determinations were always high, that is 0.0008 gram, but were constant, if care was taken not to heat the crucible for more than about five-eighths of an inch from the bottom, to a red heat.

Once in place it is not necessary to remove the crucible, and a large number of determinations can be made, requiring no removal of the crucible, or rearrangement of the washer.

The cup *A*, it will be seen, carries an outlet and an inlet tube for air, brazed into place. It is also provided with an outlet tube for water, and by connecting this tube to a glass tube, giving to the water sufficient fall, it will act as a siphon and carry the water away rapidly. The inflow is obtained by allowing a glass tube attached to some source of supply, preferably the tap, to rest directly upon the bottom of the cup, and having the supply so regulated that the outflow will carry it off.

The train for the combustion of carbon as used with this apparatus consists of the following parts:

(1) Air supply obtained from furnace blast or aspirated from bottles.

(2) Potassium hydroxide bulbs containing potassium hydroxide of 1.40 sp. gr.

(3) A small guard tube to catch any drops of potassium hydroxide that might find their way from the potash bulbs.

(4) Combustion crucible, closed by a special water-cooled cup and casing and resting upon an asbestos board. The bottom part of the crucible projects 1¼ inches through the board and is heated by a flame placed directly beneath it. The whole arrangement is supported by a tripod.

(5) Copper oxide tube; a brass tube 12 inches long and ⅜ of an inch in diameter, containing granular copper oxide in that part of the tube which is subjected to redness. This tube is water-jacketed, as recommended by Dr. Sargent.[1] If these water-coolers are made over copper tubes, large enough in diameter to allow the copper oxide tube to slide freely through them, but not too loosely, it will be found a simple matter to replace the copper oxide tube at any time. These coolers are best supplied with water directly from the tap, the outlet of one cooler being connected to the inlet of the other by glass tubing and rubber connections, and the waste water carried to a suitable drain pipe, near at hand. The copper oxide tube rests upon a tripod and over it is

[1] This Journal, **22**, 277.

placed a piece of fire-brick, cut so as to form an arch over the tube, and retain the heat. The copper oxide tube is heated for a length of 3 inches to a good red heat, and if the Bunsen flame will not do this, a blast is used allowing it to strike the fire-brick first and curl around the tube.

(6) The next part of the train, is that recommended by Job and Davies,[1] and consists of a U-tube, 6 inch form, containing in the arm nearest the copper oxide tube, thoroughly dehydrated cupric sulphate, and in the other, thoroughly dehydrated cuprous chloride.

(7) A silver sulphate tube containing 10 cc. of silver sulphate solution.

(8) A calcium chloride tube, filled with freshly and thoroughly dehydrated granular calcium chloride.

(9) The potash absorption bulb, same style as used by Dr. Sargent[2] and Job[2] and Davies.

(10) A guard tube of calcium chloride.

The solution of steels is obtained by the use of an acidified solution of double chloride of copper and potassium. The filtration of the carbon is effected as follows. A glass rod, small enough in diameter to pass through the stem of a carbon filtering tube ⅞ by 3 inches, is flattened out at one end as recommended by Shimer.[3] Upon this flattened end is supported a platinum perforated disk smaller than ⅞ inch, so that it just about clears the sides of the tube when dropped into place. The glass rod and platinum or porcelain disk are put into place in the carbon filtering tube, a layer of dry asbestos previously washed and ignited is made upon the disk, and a little short-fiber asbestos suspended in water is then poured upon it, until a felt of about ⅛ inch or more is formed.

The filtering is done upon this bed, using suction. When the washings are completed, the suction is turned on hard and the felt sucked dry. The platinum disk carrying the asbestos felt and carbon residue is then pushed to the top of the funnel tube, and as a general thing it will be found to wipe the sides of the tube free from carbon; when near the top it is pushed forward with a sudden jerk, carrying it out of the tube. It may then be placed upon a watch-glass, put in the oven and dried. The asbestos felt

[1] This Journal, **22**, 791.

[2] *Loc. cit.*

[3] *Loc. cit.*

and carbon should be transferred from the tube to the watch-glass just as soon as the felt has been freed from excess of water, otherwise the carbon may dry upon the sides of the filtering tube and cannot be wiped clean when the asbestos plug is pushed forward, and this will necessitate the use of a little ignited asbestos to wipe the sides of the tube, the only objection to any form of filtration for carbon which necessitates a transference of the carbon residue.

With such an arrangement 6 or 8 carbons might be filtered at one time, or in the time it generally takes to filter one, by simply having 6 or 8 tubes suitably connected to a source of suction. With a little practice it will be found that it is not often necessary to clean out the tube with an extra piece of asbestos.

After the carbon plug is dry, it is transferred, carbon side down, to the platinum crucible and it is best, as recommended by Shimer,[1] to have a thin platinum disk in the bottom of the crucible. The platinum disk used for filtering is removed from the asbestos felt before it is introduced into the crucible, a very easy operation as the felt in drying turns up at the edges so that it may be picked up with forceps and transferred to the crucible. The felt carrying the carbon is pressed against the bottom and sides of the crucible, and if it was found necessary to wipe the sides of the filtering tube with ignited asbestos this too is added, keeping it close to the sides of the crucible. If now a platinum disk, the same as used in filtering, is dropped upon the felt it will greatly aid in keeping the heat in the bottom of the crucible, although it is by no means necessary.

The asbestos washer is now moistened with a little water (about 5 or 6 drops), as is also the contact flange of the cup, using the wet finger; the cup is now screwed into place and drawn up tight. The apparatus is placed on the asbestos board, over the tripod, the water connections made, and the air-inlet and air-outlet tubes connected to the absorption train. The copper oxide tube having in the meantime been heating, air is now turned on and allowed to flow at the rate of 4 bubbles per second for five minutes, when the weighed potash bulb is introduced in the train and the Bunsen flame is brought under the combustion crucible; ¾ to 1 inch may easily be brought to a good red heat. The author has been using

[1] *Loc. cit.*

fifteen minutes for a combustion and fifteen for aspiration, with an air flow of 4 bubbles per second during combustion and 5 during aspiration, and combustions may be made in less time than this, as the combustion is completed in from ten to fifteen minutes, and it is simply a matter of washing all the carbon dioxide from the train in as short a time as possible and using the least amount of air.

Blank determinations as obtained with this apparatus do not run over 0.0005 gram, and have been as low as 0.0002 gram. The results obtained on standard samples, the carbon of which was determined by combustion in platinum and porcelain tubes, checked very closely and a series of determinations on the same sample were very concordant.

The following changes might be made in the apparatus: (1) It could be made of aluminum. (2) The upper outside face of both cup and casing might be knurled, affording a better hold in putting together. (3) The cup might be closed and provided with water inlet and the outlet tube connected to the water-jacket of the copper oxide tube, thereby reducing the number of water connections. (4) The air inlet tube should be bent a little towards the center and away from the walls of the crucible. The original cost of the above form of apparatus need not exceed $5, not of course including the crucible, and can be made in any machine shop.

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OCCURRENCE OF SALICYLIC ACID IN FRUITS.[1]

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FOR the past twelve months or more, tests for salicylic acid in various fresh fruits have been carried on in the laboratory of the Montana Experiment Station with the result of showing its almost constant presence in extremely small quantity.

So far as we know the only similar work has been done by Portes and Desmouliere[2] who report its presence to the extent of

[1] Read at the November meeting of the New York Section of the American Chemical Society.

[2] *J. Pharm. Chim.*, **14**, 342.