

Fig. 1.

and serve the double purpose of giving rigidity to it, and of connecting the electric circuit with the wire system shown in Fig. 2.

Owing to the small current required in making extractions with ether, it was not thought advisable to provide for heating less than six flasks at a time. The resistance wire is made of No. 22 nichrome alloy which affords a resistance of 0.937 ohm to the foot.

When the apparatus is connected with the 110-volt circuit, the strength of current obtained and the length of wire through which the current passes at

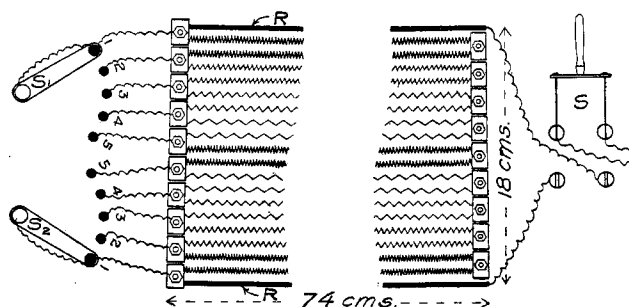


Fig. 2.

each step as the switches are moved from position 1 to position 5 are as follows:

Position of switch.	Number of strands in circuit.	Total resistance. Ohms.	Length of wire. Cm.	Current. Amp.
1	18	110.0	3578	1.00
2	14	73.3	2384	1.50
3	10	55.0	1789	2.00
4	6	40.0	1301	2.75
5	2	27.5	895	4.00

By changing the switches one at a time, twice the number of steps in the variation of the current may be obtained.

The inner vessel is water-tight and may be used as a liquid bath if desired. When used as a water bath with the top plate removed, the water becomes heated to a temperature of about 42° with the switches in position 1. This causes the ether to distil at almost exactly the same rate as when the apparatus is used as an air bath with plate P in place. When it is desired to heat more than six flasks at a time, this can be conveniently done by removing the top plate, in which case the apparatus can be more economically used as a water bath.

The minimum temperature obtained with the top plate removed and glycerine in the inner box is 60°. This increases to 80° with the plate in place, while with the switches in position 5 a maximum temperature of 135° may be obtained.

The apparatus as thus described may be obtained from the Central Scientific Co. at a very moderate cost.

The advantages of the apparatus may be stated as follows:

(1) The flasks rest on a hot metallic plate, and are surrounded by a heated atmosphere in consequence of which a very low current is sufficient for extractions with ether.

(2) The temperature of the apparatus is capable of being changed between considerable limits which enables the same apparatus to be used for making extractions not only with ether, but also with acetone, chloroform, carbon tetrachloride, and other solvents commonly used in making extractions. The same feature enables the apparatus to be heated up quickly at first then used for making extractions with ether.

(3) After the extraction is complete, the solvent may be evaporated from the flasks without danger of ignition by replacing them in the apparatus and turning on a low current.

(4) With the top plate removed the apparatus may be used as a hot plate for making slow evaporations, and for other purposes.

(5) If so desired it may be used as a liquid bath by placing water, or some other high boiling-point liquid, in the inner box.

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## TWO NEW PIECES OF CHEMICAL APPARATUS.

- I. Apparatus for the continuous extraction of liquids with immiscible solvents lighter than water.
- II. Apparatus for quantitative reactions which depend on the measurement of an evolved gas.

By R. F. BACON AND P. B. DUNBAR.

Received September 26, 1911.

### I. CONTINUOUS LIQUID EXTRACTOR.

This apparatus was designed originally for use in the extraction of lactic acid from ketchup and other fruit products. Its principal advantages are com-

pactness, the elimination of ground joints and stoppers, and practically complete condensation. Seven of the extractors may easily be placed side by side on a 24-inch hot plate. They have been made for the authors at a cost of \$1.50 each, but may be constructed in the laboratory by any one having ordinary skill in glass-blowing.

The apparatus consists of four parts: (1) A jacket flask; (2) an extraction thimble; (3) an ordinary Gooch funnel; (4) a condenser.

(1) The jacket flask (Fig. 1) A is made of glass tubing 2 inches in diameter and approximately one-sixteenth of an inch thick; it is 20.5 inches long and is enlarged to a diameter of about 3 inches at its lower or sealed end, as shown in the illustration. (2) The extraction thimble (Fig. 1) B is an ordinary test tube having a diameter of 1.5 inches and a capacity of 100 cc. when filled to within 1.5 inches of the top. One-fourth of an inch from its top and on opposite sides of the tube are placed two holes about one-fourth inch in diameter. (3) The Gooch crucible funnel is (Fig. 1) C; those used in this laboratory are 8.5 inches long. When dense liquids are to be extracted it is sometimes necessary to increase the length of the funnel. The lower end of the stem is ground at an angle of 45 degrees. (4) The condenser (Fig. 1) D is designed to hang loosely in the jacket flask. Its details are evident from the drawing. A simpler form of condenser, which is just as efficient, may be made by sealing one end of a 1 3/4-inch tube and drawing it to a point.

The open end is flared somewhat to permit the tube to hang in the jacket. The condenser is closed by a two-holed rubber stopper, through which pass the in-flow and outflow tubes.

#### OPERATION.

Place from 100 to 150 cc. of ether in the jacket flask A; put the liquid to be extracted (100 cc.) in the test tube B, insert the funnel in the same tube and suspend it in the jacket flask, about 3 inches above the bottom, with a copper wire which passes through the holes in the test tube and is hooked over the rim of the jacket flask. Insert the condenser in the top of the jacket flask. The condensation is usually so perfect that no ether vapors escape into the room. The condensed ether drops from the point of the condenser into the funnel and is carried to the bottom of the test tube whence it flows up through the liquid and overflows at the top.

The efficiency of the extractor may be increased by the use of a glass spiral attached to the stem of the Gooch funnel, as described by Kempf.<sup>1</sup>

#### II. APPARATUS FOR THE MEASUREMENT OF AN EVOLVED GAS.

The apparatus represented in Fig. 2 was devised to give in a compact and easily manipulated form an apparatus which will allow of the measurement of an evolved gas without first sweeping out all air or other indifferent gases. It consists of a graduated funnel tube, A, a reaction chamber, B, an absorption tube, C, filled with glass beads, a eudiometer, D, and a leveling tube, E. A heating coil may be wrapped around the reaction chamber B when desired. The absorption tube C, which is sealed onto the eudiometer, fits into the reaction chamber B by means of a ground joint at F.

The manner of using the apparatus may best be illustrated by one of the reactions which can be advantageously carried out in it. Spica<sup>2</sup> estimates citric acid from the carbon monoxid evolved by decomposing this acid with strong sulphuric acid at 100° C. He runs air-free carbon dioxid through a flask containing the citric acid until all air is displaced from the apparatus. He then adds concentrated sulphuric acid, heats to 100° C., drives over the evolved carbon monoxid with a stream of air-free carbon dioxid, and collects the gas in a eudiometer over a sodium hydrate solution. The method as carried out by him is accurate, but requires considerable care and attention to remove the air from the apparatus completely, to insure that all evolved carbon monoxid is swept over into the eudiometer, and to prevent the strong sodium hydrate solution from sucking back into the reaction flask containing carbon dioxid and sulphuric acid. In the apparatus proposed by us no attempt is made to remove the air before reaction, the volume of evolved gas being simply measured by the increase in volume of the total gases after reaction. Thus the Spica method as carried out in this apparatus is as follows:

Place about 0.2 gram of citric acid in the reaction chamber B, open the stopcock X and bring the liquid (in this case a strong sodium hydrate solution) to the zero mark G in the eudiometer tube by raising or lowering the leveling tube E. The air in the apparatus is then at atmospheric pressure. Close X. To A add about 15 cc. of concentrated sulphuric acid. Lower the leveling tube so that the air in the apparatus is under reduced pressure. By carefully opening the stopcock X run in exactly

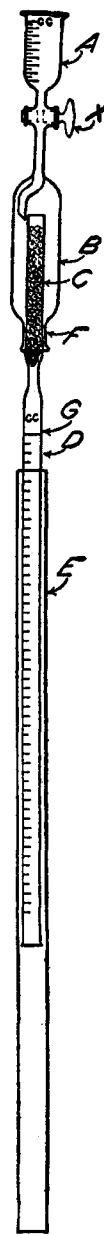


Fig. 2.

<sup>1</sup> Chem. Ztg., 34, 1365 (1910); C. A., 5, 1350 (1911).

<sup>2</sup> Chem. Ztg., 34, 1141 (1910).

10 cc. of the sulphuric acid. Close X and heat to about 100° C. by means of the heating coil until the reaction is complete. Let the apparatus stand until the absorption of other gases (sulphur dioxide, etc.) is complete and it has reached room temperature. Bring the gas in the apparatus to atmospheric pressure by means of the leveling tube E. The gas reading in the eudiometer minus 10 cc. (for the added sulphuric acid) equals the volume of the evolved gas at the existing temperature and pressure. The apparatus has been used in this laboratory with accurate results to estimate citric acid by the Spica method and for the estimation of amino acids by the Van Slyke method,<sup>1</sup> which depends on the evolution of nitrogen, by the action of nitrous acid on the amino acids. The apparatus is readily cleaned by taking it apart at the ground joint F.

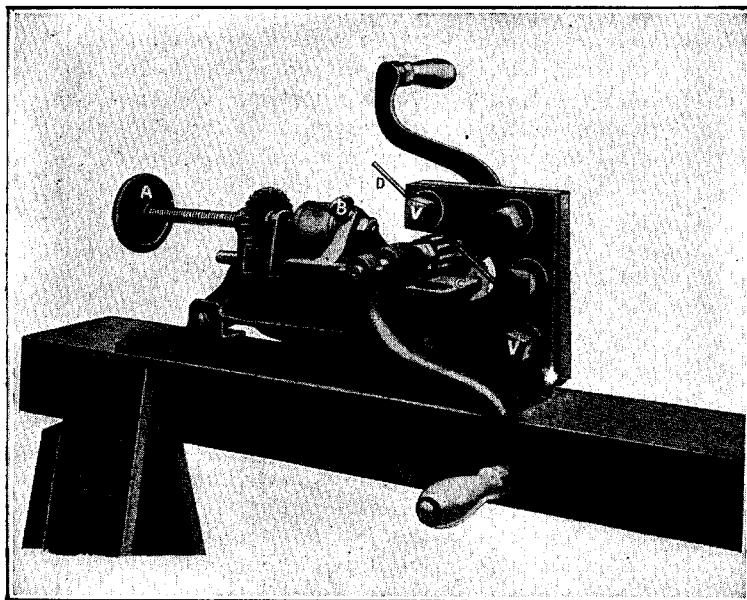
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#### LABORATORY MILLING MACHINE FOR SAMPLING STEEL.

By CHAS. MORRIS JOHNSON.

Received November 1, 1911.

In certain kinds of steel the writer encountered much difficulty in getting samples of sufficiently



small mesh for the determination of carbon by the direct method described by him several years ago.

("The Chemical Analysis of Special Steels"—John Wiley & Sons, 1909.)

The trouble was confined to thin sheets, wire, hack saw steel, band saw steel, razor blades, resistance ribbon, nails and small samples of all kinds that are irregular in shape and difficult to hold in the drill press vise.

The machine shown in the illustration afforded a successful means of avoiding various time-consuming expedients.

The sample D of wire, for example, is held in the vise V-V. The millings are taken up by means of a cutter, made of the best high-speed steel, and are caught on a piece of cardboard at C.

The automatic feeding device at A is hastened in its action by tightening the screw at B.

If desired, where a large number of samples are to be milled, a small motor can be used, and a pulley can be attached instead of one of the handles.

The milling cutters can be sharpened several times.

It is desirable, and well worth the small amount of time involved, to anneal all samples received, if they are not already in a softened state. This operation can be done in a half hour's time by heating the sample to 800° C. (bright red), quenching at once in water and then holding at 620°-630° C., for 20 minutes (low red). The annealing of 13 per cent. manganese steels has also been previously described in "Analysis of Special Steels, etc." together with the chemical tests for perfect annealing.

The millings obtained by this laboratory tool are not sifted, as they are just right for direct determination of carbon by combustion in oxygen.

If the sample submitted is sufficiently rigid, it does not need to be held by both ends, as in the case of the sample of wire.

This machine, as illustrated, has been in use in the writer's laboratory for a year. Since its introduction, the samplers no longer dread the appearance of wire and steel ribbon, this work being now a mere matter of easy routine.

The first cost was a bagatelle compared to the saving of labor in one month. Several copies of this milling tool are now in use in other laboratories of this Company.

LABORATORY OF PARK WORKS,  
CRUCIBLE STEEL COMPANY OF AMERICA,  
PITTSBURGH.

## ADDRESSES.

#### RECENT DEVELOPMENTS IN BAKELITE.<sup>2</sup>

By L. H. BAKELAND.

Received November 13, 1911.

My subject involves the use of two main raw materials, phenol or carboic acid, which is found in

<sup>1</sup> *Ber. d. chem. Ges.*, [3] 43, 3170 (1910).

<sup>2</sup> Read at joint meeting of New England Section, Amer. Chem. Soc. and Soc. Chem. Industry, Boston, Nov. 10, 1911.

gas tar, and formaldehyde. The latter is now manufactured by the oxidation of wood-alcohol.

I should state that the name of formaldehyde is used in a rather broad sense. I never have seen real formaldehyde, CH<sub>2</sub>O, and very few chemists have had occasion to examine it. It is a gas which can be made to liquefy and solidify by the intense arti-