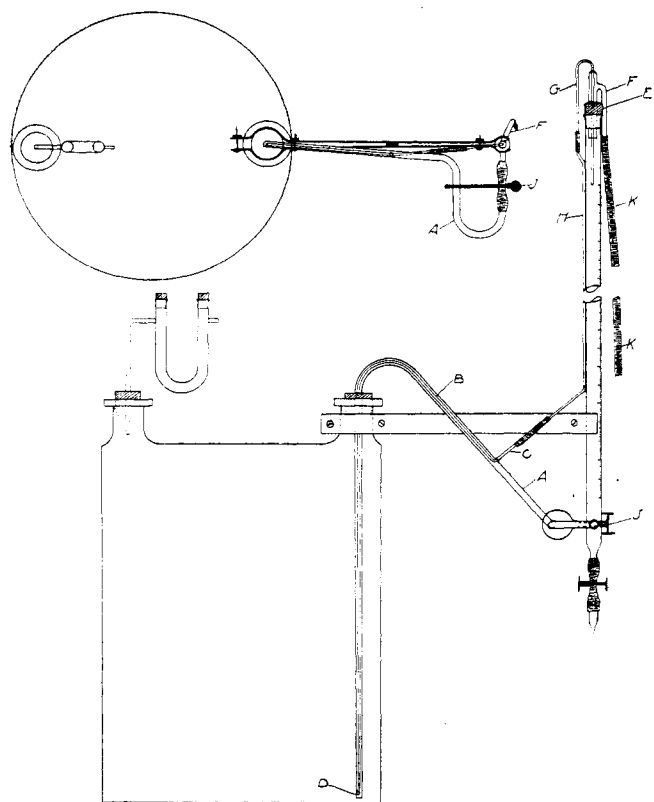


which is fitted a double tube. The outer tube is connected by a short T tube, *F*, bent downward and fitted with a rubber tube by means of which the operator sucks up solution to fill the burette. The inner or



overflow tube *G* is fused into the outer and bends downward, connecting by means of a short rubber tube, with a small glass tube, *H*. This tube extends down the back of the burette and joins, by means of another short rubber tube, with the tube *C*. The overflow tube *G* may be adjusted by sliding up or down in the cork *E* so that the lower end of it is on the same level as the zero mark on the burette. It is seen that when the liquid above the zero mark on the burette is allowed to siphon back it will drain off all the liquid above the zero mark.

In order to fill the burette, the operator opens the pinch-cock *J* and sucks through the rubber tube *K* till the liquid is above the zero mark on the burette. He then allows the excess to automatically siphon back through the overflow tube into the main supply.

Instead of filling the burette by suction, a rubber bulb may be attached to the bottle so that the burette can be filled by air pressure. For small supply bottles this method is excellent but for larger ones it is not so satisfactory.

6358 ELLIS AVENUE, CHICAGO, ILLINOIS

### A KJELDAHL DISTILLATION APPARATUS

By J. M. PICKEL

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Mr. Holmes describes what is evidently an excellent form of Kjeldahl distillation apparatus.<sup>1</sup> We have

<sup>1</sup> THIS JOURNAL, 6 (1914), 1010.

been using for eight or ten years in the laboratory of the North Carolina Department of Agriculture a distillation apparatus of the writer's designing, which would seem to be simpler, more flexible and less costly than that of Mr. Holmes'. This apparatus has the following features, some of which are, so far as known to the writer, distinctive:

1—The connecting bulbs and condensing tubes are in one piece, thus eliminating annoying rubber connections. (In the case of a copper still, the whole thing is in one piece.)

2—The condensing tubes are not clamped, screwed or attached, in any way, to other parts of the apparatus, and not enclosed within any other part of the apparatus. (To be sure, during distillation, there is connection with flask and receiver.)

3—In consequence of items 1 and 2, any condenser can, without interfering with any other condenser, be instantly removed and another dropped into its place; any condenser can, therefore, without disturbing its neighbors, be repaired, or flushed out with water

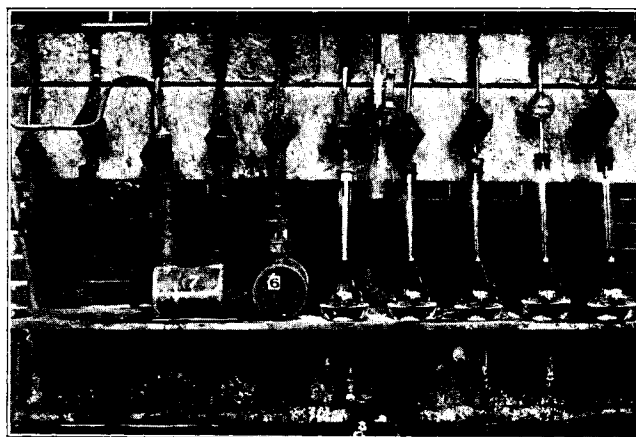


FIG. 1—BACK VIEW OF KJELDAHL DISTILLING APPARATUS

under a tap, or air forced into it under water (with a bicycle pump, for example), this latter operation disclosing by air bubbles even the tiniest leak (all of our condensers are thus tested when first put into commission and at suitable intervals thereafter).

4—The condensers adjust themselves automatically to distilling flasks of different lengths.

5—In case one cares to set or hang the apparatus against a wall, thus throwing distilling flasks and receivers on one and the same side, the condensers are easily adjusted to that arrangement; but the receivers are placed *above*, not *below*, the distilling flasks.

6—The distilling flasks, their support and heaters (Bunsen burners) are suspended over a trough.

7—In consequence of arrangement 5 and 6, if, during distillation, a distilling flask breaks, as not infrequently happens, the contents of it drop, not on the table, nor on or into a receiver, but into the trough which can be easily flushed out with water.

8—Regurgitation of liquid from the receiver back into the distilling flask is precluded.

In Fig. II, upper part, are the receivers (flat bottom

Erlenmeyer flasks of 250 and 300° cc. capacity), connected up with the condensers. The stopper of each receiver carries two bead tubes, in addition to the glass tube that connects, by means of rubber tube, with the condenser. This glass tube and the rubber tube could be dispensed with—the rubber stopper in that case being slipped over the end of the condenser. The condenser, or its glass extension, does not touch the

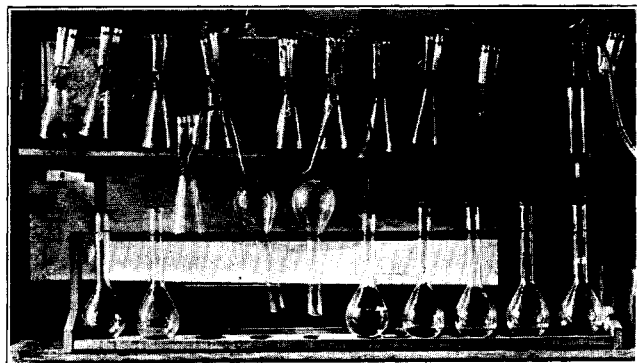


FIG. II—FRONT VIEW OF KJELDAHL DISTILLING APPARATUS

liquid in the receiver. To begin the distillation, introduce the standard acid and indicator into the receiver, but no water. Connect up with the condenser, then fill the interstices of the beads with distilled water.

In lower part of Fig. II are shown:

(a) A never-gets-out-of-order carrier, or support, for Kjeldahl flasks.

(b) A device (see the flask at the extreme right) which, by mere pressure on a pinchcock, drops into the flask (simultaneously washing its neck) the requisite quantity of water (about 100 to 125 cc.) to dilute the digestate.

(c) A distillation flask, marked 10, connected up with condensing tube and receiver. The condensing tube rises from the connecting bulb, passes through a slot (see Fig. I), over the edge of the water tank, into the water; along under the water and rises over the front edge of the tank on which it rests; the only other point of support to the condensing tube is the stopper in the distilling flask. In case one desires to bring the receiver on the same side with the distilling flasks—the condensing tube, after dipping three or four inches into the water, bends back through a slot over the same side of the tank (see the fifth flask from the right in Fig. I, thus connected up with its receiver).

**DIMENSIONS OF THE CONDENSING TUBE AND CONNECTING BULB**—The bulb (copper or block-tin), greatest diameter 6 or 7 cm., height 8 to 10 cm.; soldered to the lower end of bulb so as to give good drainage back into the flask, is a tube (preferably block-tin) about 6 mm. ( $\frac{1}{4}$  inch) inside diameter, length 7 to 8 cm.; projecting 2 to 3 cm. into the top of the bulb, is the condensing tube (block-tin), diameter inside about 6 mm. length from bulb to top of first bend, 10 to 12 cm. top of first bend to middle of second bend about 20 cm., thence to middle of third bend about 14 cm., thence to middle of fourth bend about 18 cm., thence to end about 6 to 7 cm.; all of these measurements, except

those of the bulb, of the tube below the bulb and of the tube above the bulb to the first bend, can, without detriment, be diminished by about half, *provided* that, in order to use interchangeably flasks of different lengths, there must be sufficient length of tube between the first and second bends to prevent lifting too much of the tube out of the water in using flasks with extra long necks. With plenty of cooling water, a block-tin tube  $\frac{1}{4}$  inch inside diameter and 8 or 10 inches (20 to 25 cm.) under water affords ample condensing surface.

The cooling tank has the following measurements: length (for twelve condensers), 153 cm.; depth, 20 cm.; width at top 26.5 cm., at bottom 21 cm. In case receivers and distilling flasks are on the same side, the tank need be only 4 or 5 cm. wide.

But after the distillation is over, these condensers cannot be easily steamed out? If the steaming out is intended to clean out traces of ammonia, it is entirely superfluous in the case of these condensers—there is no ammonia to clean out; if it is intended to clean out any fixed alkali, that is something steam cannot do; that is a function of water; and these condensers can easily be carried to a tap, water run in at the receiver end and out through the bulb end. However, if one insists on steaming out, each of these condensers can easily and cheaply be furnished with its individual tank, each little tank with a stoppered

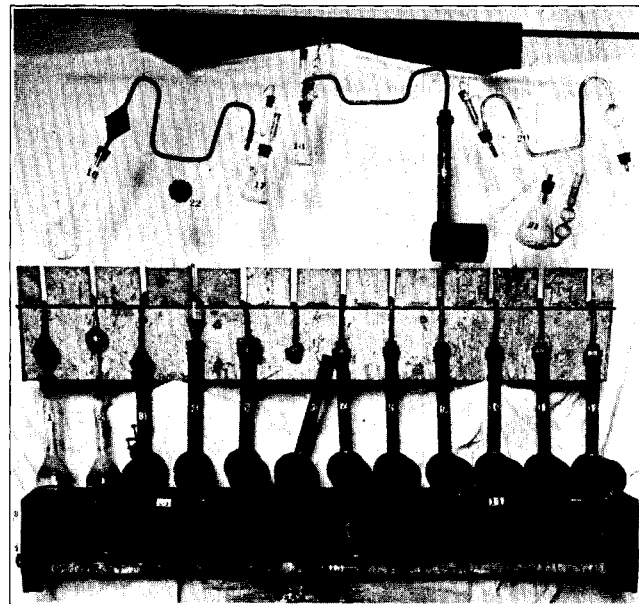


FIG. III—MODIFIED APPARATUS

hole in the bottom, through which the water can be drained into a trough running underneath and supporting all of the individual tanks. The writer has used experimentally and successfully for several years a similar little tank and condenser, interchangeably with the other condensers of this apparatus.

In Fig. I is shown at 6 and 7 an end and a side view of two of a set of twelve copper Kjeldahl stills. The parts are: a cylinder 13 cm. long and 10 cm. in diameter; on one end a tubulus; at the other end, and pro-

jecting into it, 2 or 3 cm., a tube about 2.9 cm. in diameter (inside) and 25 cm. long; projecting into the end of this tube is the connecting bulb, and into the connecting bulb the block-tin condensing tube. All parts are soldered together with ordinary tin solder, still and condensing tube forming one continuous piece. The connecting bulb, however, may be dispensed with, as it is of not much use. These stills are as easily charged, discharged and cleansed as a Kjeldahl distilling flask; these operations are performed, of course, through the tubulus. The tubulus may be stoppered with a cork; but a rubber stopper is preferred—either inserted or clamped on. The clamped-on stopper shown on still 6 was used two years, and could doubtless be used several years longer. One of the advantages of these stills is that they practically eliminate the trouble that stoppers cause in the case of Kjeldahl distilling flasks; their chief drawback is the time (about 15 minutes to a set of ten) consumed in transferring the digestage. Only the lower part of these stills is corroded; new bottoms and ends are then soldered on. The writer designed and used a still of this kind six or eight years ago. Five or so years ago he put into use a set of twelve. They were used with entire satisfaction for two years, undergoing repairs several times in the meanwhile. The use of them was discontinued, because they were thought to be no longer economical in view of the cheapness and durability of Jena glass, and in view of the time wasted in transferring.

Fig. III shows another method of connecting these copper stills with the condensing tube, namely, by means of an ordinary pipe coupling with rubber washer between. Despite the fact that this method renders the tubulus unnecessary, it was abandoned in favor of the tubulus. At 22 is shown a disc (copper or block-tin) that is used in some of our connecting bulbs. These discs are slightly less in diameter than the greatest inside diameter of the bulbs; are laid in loose, not soldered; they should have numerous notches, about 0.5 mm. deep, around the edge. The steam impinges against this disc, passes around it and out into the condensing tube.

Many thousands of nitrogen determinations have been made with this apparatus by the writer and by others (in the case of the copper stills, by the writer only). But you cannot see what is going on in the copper stills? You do not need to. But if there is frothing? There will be no frothing, except as the result of bad management; and bad management ought not to occur oftener than once or twice per thousand determinations. Moreover, when frothing does occur, it will manifest itself at the receiver end of the condenser, and the determination is lost. But the stills sometimes get dry and melt apart? Yes, through gross carelessness, once in perhaps, a thousand cases, for there is, to start with, about 250 to 300 cc. of liquid in the still; the distillation is stopped when about 100 cc. of distillate has accumulated in the receiver.

LABORATORY OF THE NORTH CAROLINA DEPARTMENT OF AGRICULTURE  
RALEIGH, N. C.

## ADDRESSES

### CHEMICAL PATENTS—I

By SEABURY C. MASTICK<sup>1</sup>

Received May 14, 1915

The subject of chemical patents and of patents for processes relating to industrial chemistry is one of the oldest, if not the oldest, in the realm of patents. As early as 1467 a patent was granted in Berne for the manufacture and sale of paper and in 1507 the Council of Ten in Venice granted an exclusive privilege for twenty years for the introduction of a secret process of mirror-making.

During the first ten years at the beginning of the patent policy in England (1561-70) twelve of the eighteen patents granted were for various chemical products and processes. The chemical patents related to such subjects as soap, saltpeter, alum, sulfur, oil, salt, glass, and cloth- and leather-dressing. In the course of the next decade three of the ten patents granted related to chemistry, *viz.*: those for earthenware, glass and sulfur.

The marked increase in the study and development of the arts relating to chemistry in the United States during the past decade and more has made it imperative for all having to do with the subject to give careful attention to patents, to the ascertaining of what is patentable, how to properly protect inventive ideas and how to enforce the protection so given, as well as how to avoid trespassing upon the rights of those holding patents with which the thing you are considering developing may conflict.

The importance and development of chemistry in the arts in the United States is shown by the following comparative figures relating exclusively to the manufacture of chemicals without

reference to allied arts such as fertilizers, petroleum products, rubber manufacture, etc., all of which involve chemical processes to a greater or less degree.

|                              | 1899         | 1909          |
|------------------------------|--------------|---------------|
| CAPITAL INVESTED.....        | \$89,069,000 | \$155,144,000 |
| MANUFACTURED PRODUCT.....    | 62,637,000   | 117,689,000   |
| SALARIES AND WAGES PAID..... | 12,316,000   | 20,222,000    |
|                              | 1901         | 1911          |
| EXPORTS.....                 | \$14,866,035 | \$ 23,077,414 |

Such figures and such growth as this in a decade in an art in which invention has played a conspicuous part indicates the necessity the chemist is under of having some knowledge of patents.

It is the purpose of these lectures to give those who have adopted the science of chemistry as their profession some guiding principles which will enable them to form an intelligent opinion concerning such questions of patentable invention, rights of inventors, infringement of patents and related subjects as may come to them for consideration.

As these lectures are primarily addressed to chemists, the illustrations and applications of the patent law have, as far as possible, been drawn from patents and cases relating to chemical compositions of matter and chemical processes, as it is thought that concrete examples, pointing out the principles involved, will not only prove more interesting but more instructive than a mere statement of abstract principles. Furthermore, in elaborating this latter idea, a single series of important cases, those relating to the industrial development of the production of aluminum, has been chosen for study in connection with the more detailed examination of certain phases of the subject: in this connection the basic patent of the late Charles M. Hall for the electrolytic production of aluminum has been followed from the time of the filing of the application in the Patent Office

<sup>1</sup> Special Lecturer on Chemical Patents, Department of Chemical Engineering, Columbia University, 1915.