It can be used for handling poisonous or infectious material with great satisfaction, and can, furthermore, be readily used to calibrate pipets.

MADISON, WIS.

[CONTRIBUTION FROM THE WOLCOTT-GIBBS MEMORIAL LABORATORY OF HARVARD UNIVERSITY.]

AN ADVANTAGEOUS FORM OF STILL FOR THE EXACT MEASUREMENT OF BOILING POINT DURING FRACTIONAL DISTILLATION.

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In the course of a recent investigation on the heats of combustion of liquid hydrocarbons, it was needful to prepare certain of these substances in a very high degree of purity, and at the same time in considerable quantity. For this work it was necessary to determine exact boiling points, and an advantageous form of still was devised, which proved itself, especially in this respect, more efficient than any of the apparatus now in common use. This still, because of its simplicity and usefulness, and because it does not appear to have been proposed before, seems to the authors to merit separate brief description.

In the accompanying drawing, two forms of the instrument are shown. Fig. 1 represents the type first constructed: of this the other is a simple modification. A glance will show the immediate purpose of the device. The still was designed to hold a sensitive Beckmann thermometer, so that very small fluctuations in the boiling point of the liquid under examination could be accurately observed, and the boiling point of the purest distillate precisely determined by a comparison with a standard vapor of neighboring known boiling point. The still not only served this purpose admirably, but proved itself (as had been expected) a very effective instrument for fractionation, without being further modified.

It consists essentially of a flask, of any desired capacity within practicable limits, to which a side tube is attached, the latter being held parallel to the neck of the flask, and connected with it, by two smaller tubes at the top and bottom. The upper of these (which serves to carry the outgoing vapor) communicates directly with the upper end of the flask-neck; while the lower tube, which serves to return the prematurely condensed liquid to the flask, is a constricted continuation of the side tube itself, and is bent into a shallow U, fused into the flask-neck a few centimeters above the bulb. The relative positions and sizes of these tubes are shown in the drawing (Fig. 1). The lower connecting U-tube should be very narrow in diameter, and bent in such a way that the orifice at the flask-neck is not much more than the tube's diameter above its lowest point, so as to minimize the amount of dead space which can hold and thereby waste the liquid being distilled.

The vertical side tube is left open at the top, where it is constricted to such a diameter that it will receive and hold closely the shell of the Beckmann thermometer at a point near the zero mark. No cork should be used; the thermometer may easily be made to fit without grinding

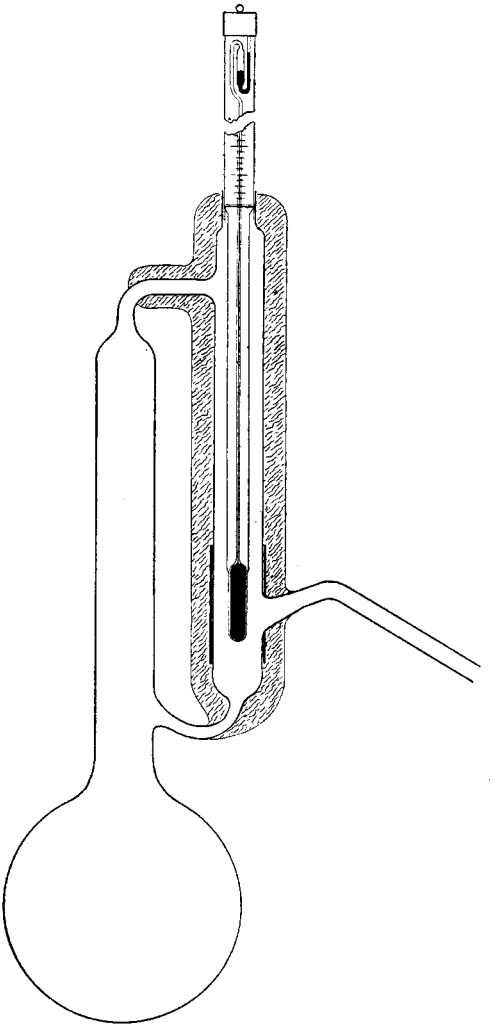

Fig. 1.

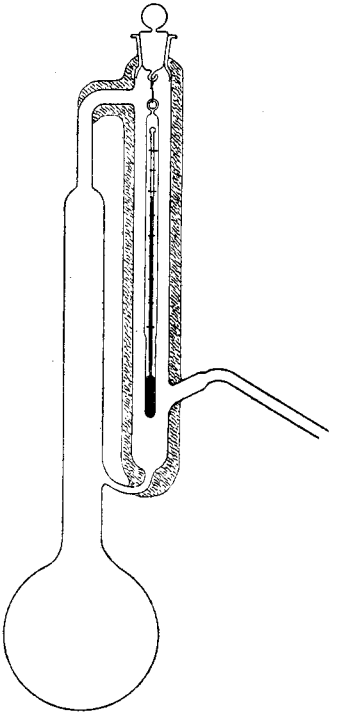

Fig. 2.

closely enough to prevent the escape of vapor, for a liquid seal will collect in the annular space at this point. If necessary, a packing of clean asbestos fiber will make the closure secure. With hygroscopic liquids, of course, the thermometer must be fitted into place with a ground joint. The delivery tube should be wide, and should be bent upward at the

point where it leaves the side tube, so as to cause condensed liquid from the bulb of the thermometer, or from the walls, to flow back into the flask, and not into the condenser. A mirror of tinfoil to prevent radiation may advantageously be bound around the side tube so as to surround the thermometer bulb; and the whole of this tube as well as the upper connecting tube should be encased in a thick shell of asbestos.

When liquid is boiled in this apparatus, there is, of course, an almost complete reflux condensation until the tubes reach the temperature of ebullition. In full operation, the distillation should be not rapid, but very steady. All that condenses before the delivery tube is reached returns to the flask through the narrow lower connecting tube, and only that part which still remains as vapor after the long journey passes out into the condenser. No stream of superheated vapor can rise around the thermometer, nor direct spatterings from the liquid impinge upon it. When the tubes are well insulated thermally, the irregular motions imparted to the vapor-stream by the shape of the tubes seem to be sufficient to render the vapor thermally homogeneous when it reaches the thermometer bulb. Here the presence of both vapor and liquid in approximate equilibrium enables the temperature at the moment of egress into the condenser to be accurately determined.

The essential feature of the apparatus is the fact that in it the whole uncertain stem of the thermometer, practically up to the zero of the Beckmann scale, is kept really at the temperature of the vapor. The importance of this precaution in determining any exact temperature with the Beckmann thermometer can hardly be overestimated. The degree of uncertainty introduced by a merely partial heating of the lower tube of this instrument may be very great. Moreover, the bulb of the thermometer is exactly opposite the exit tube into the condenser, so that the thermometer indicates the true temperature of the vapor which is actually being condensed, as nearly as possible. In these respects it is very different from the Claisen form.

Another modification of the same type of still is seen in Fig. 2. This is intended for use with small Anschütz thermometers; it is like the first modification except for the addition of a hooked glass stopper to the side tube, and the inserting of mica windows into the asbestos shell around this tube. When liquids are to be distilled which leave much insoluble residue, another stopper on the neck of the flask itself facilitates cleaning, but this is not necessary. Electric heating by an immersed platinum coil[1] is practicable, and with non-conducting liquids advantageous. The use of platinum scrap, fragments of porcelain, or the like to introduce the gaseous phase into the body of the liquid is, of course, advisable; but

[1] See Richards and Mathews, *Proc. Am. Acad.*, 43, 21 (1908); also Beckmann, *Z. physik. Chem.*, 64, 506 (1908).

except in order to prevent violent bumping, such precautions are less necessary than with the simple boiling flask. In our work the flask was usually heated with the naked flame, which gives less trouble from superheating than a more evenly distributed source of heat of lower temperature.

To prevent irregular cooling from the outside, especially when high boiling liquids were being distilled, the whole still, together with the burner beneath, was enclosed in a cylindrical asbestos box, or air-bath, through which only the outlet tube and the stem of the thermometer protruded. By proper manipulation of the flame, or with the help of a second lamp, the whole system could be kept at a sufficient constant temperature, while through a mica window in the protecting jacket the boiling liquid could be watched.

An example of the progress of a typical case will show the efficiency of the apparatus. Five hundred cubic centimeters of a good commercial toluene were fractionated, six fractions being collected in each distillation and each of these fractions distributed into six others in the following. After six such complete distillations, the final product, 130 cc. in volume, redistilled within an interval of 0.02°, although the original range of boiling point was at least fifty times as great. Two more fractionations of a half of this material yielded 40 cc. of a product which distilled within a range of less than 0.01° under barometric pressure constant during the time of distillation; and very nearly all of this purer material redistilled over the same range. By immediate immersion of the warm,[1] dry bulb of the standardized Beckmann thermometer (which had a range of 20°) into a carefully regulated steam bath, the difference between the two boiling points of toluene and water was thus measured, and the boiling point of the toluene determined to within 0.01°.

Although no claim is made that this sort of still is more efficient than some of the other forms, it is seen to possess advantages with regard to exact thermometry not possessed by any other usual form. Moreover, stills of this type will be found to be not only sufficiently effective in operation (as is evidenced by the data just quoted), but also convenient in use. They are uncomplicated in design, and are, therefore, inexpensive. They are not cumbersome, and are easy to handle. If properly made they are by no means fragile, and will not crack at the joints if they are gradually heated with ordinary precaution, and if no cold liquid is put into them while they are hot. Made of Jena glass, they are very resistant to fracture by irregular heating; made of silica, they can be used at high temperatures still more safely; and they may be used with resistance thermometers if desired. They may easily be cleaned and are much more practicable

[1] The thermometer was allowed to fall just below 100° so that the boiling point of water should be read on a rising thread.

than some of the more complicated still heads. Distillations from this apparatus are also accompanied by little loss, because the surface of condensation is small, and the liquid retained by the U-tube may be reduced to a very slight amount by constricting its diameter. But the most important object of the apparatus, as already stated, is the exact determination of the boiling point of the actual distillate.

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NEW BOOKS.

Laboratory Experiments in General Chemistry. Designed especially for use with Stoddard's Introduction to General Chemistry. 22 pp. Northampton, Mass.: Gazette Printing Co., 1913. Price, 30 cents.

This collection includes 159 experiments, about two-thirds being devoted to the nonmetallic elements. These are followed by a brief treatment of the metals, including simple directions for their identification. The last eight experiments are quantitative. All of the experiments are well selected and the directions to the student are brief, even meagre. Some modification of the order is desirable, but this is easily accomplished since the loose-leaf plan is employed. The collection is well suited for use in the smaller laboratories. B. S. HOPKINS.

Handbuch der Mineralchemie, Vol. II, No. 5 (Bogen 41–53) with Titelbogen. DOELTER, *et al.* Dresden and Leipzig: Theodor Steinkopff. Price, M. 9.10.

Mineralochemie in the modern sense is only just emerging from mineralogy, and that only at certain points. The mass of our knowledge pertaining to minerals is still practically unchanged in form since a much earlier day. The present volume is peculiarly arid in that it deals with matter which has been practically untouched by modern ideas. There are a few hydrous silicates, the role of water in which has been experimentally studied, and there are some synthetic data on the silicates of copper, lead, and zinc, but they are mostly of a fragmentary sort, made without much regard for physical conditions or chemical relations. A great many pages are devoted to jade and nephrite; there are long tables of analyses and many physical constants, the latter probably determined not infrequently, as was the habit of the earlier physicists, on material of unknown composition; but of mineral chemistry there is practically none. However, the editors should not be blamed for omitting what does not as yet exist. E. T. ALLEN.

Praktikum der Wasseruntersuchung. By PROF. DR. O. EMMERLING. Gebrüder Borntraeger, Berlin, 1914. Price, 7 mk., 20 pfg.

This little book of approximately 200 pages covers the entire field of chemical, biological, microscopical, and bacteriological water examination, in a very clear and concise manner. The "chemical examination" in-