

warm to room temperature, and weighed. With the 100 gram size it is not necessary to weigh closer than 1 milligram. Subtract the tare, and the specific gravity is obtained at once to five figures without calculation. When only small quantities are available, these flasks can be obtained to hold only 25 or 50 grams, but the necks are smaller and more care is required in filling. For complete directions on the use of this form of pycnometer see "Der Wein und seine Chemie," by Paul Arauner, p. 25-31.

In determining alcohol in any product, a wine for example, the specific gravity bottle is filled as above indicated, then contents transferred to an appropriate distilling flask, the specific gravity bottle rinsed out, and the rinsings added to the main portion. A small quantity of calcium carbonate is added to neutralize any acidity, a piece of porcelain (broken crucible cover) to prevent bumping, and water added to make a total volume about 50 per cent. greater than sample taken, and the liquid is then distilled, catching the distillate in the specific gravity bottle. Distillation is continued until the liquid is almost up to mark, then bottle and contents put in constant temperature bath at 15.6° as before. After about one-half hour the flask is filled exactly to mark with distilled water at 15.6, neck wiped out, etc., as before, and weighed, and tare being subtracted, gives the specific gravity direct when using 100-gram bottle.

These bottles when stoppered do not change in weight, except on long standing, such as overnight, and by following the method given above very accurate results can be obtained. The weights of contents on 100 gram bottles can be checked easily by different operators to less than 5 milligrams, which means a variation of only about 0.05 alcohol at the most, and the agreement is often closer than this amount. Agreement as close as this is not obtainable by the use of ordinary forms of apparatus. Flasks No. 1 and No. 2 in the attached photograph show two forms of this apparatus, form No. 2 being most satisfactory, if mark is low on neck in order to allow for expansion, as it is more stable than form No. 1. The long slender funnel tube is used to fill both forms of bottles.

Reischauer's bottles as shown in photograph form No. 1 in 25, 50 and 100-grams capacity, are listed and kept in stock by Eimer & Amend, New York City.

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#### DEWAR VESSELS AS APPLIED TO THE DETERMINATION OF SPECIFIC GRAVITY OF LIQUIDS.

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The now familiar principle of the Dewar flask, originally designed by Professor Dewar for the heat insulation of liquid air, and at present so extensively used for maintaining liquids at constant temperature, is most advantageously employed in maintaining liquids at constant temperature during specific gravity determinations. Two types of such apparatus are here described: one type to be employed by the method of the hydrostatic balance, and the other as a pycnometer.

*Hydrostatic Balance Type of Dewar Vessels* This type is shown in the accompanying etching as No. 4. It consists of a double walled glass cylinder having a

vacuous space between the walls. It is designed to be employed on the specific gravity bench of the usual analytic balance. Dimensions in millimeters are as follows: height, 100; external diameter, 53; internal diameter, 33; internal depth, 90. The weight when empty is 85 grams and the capacity of the internal chamber is 75 cc. These dimensions may, of course, be somewhat varied. In addition to the cylinder just described, one needs a suitable plummet and suspensory wire. My own practice is to use a small pear-shaped lump of glass, having a hooked apex, as the plummet. This plummet is conveniently made in the blast lamp flame by the analyst himself from a piece of heavy glass rod. For sustaining the plummet I employ fine phosphor-bronze wire, except where special circumstances demand the use of platinum wire. The use of phosphor-bronze is dictated by the fact that it is easy to procure very fine, thoroughly annealed wire of this character, say nothing exceeding .14 mm. diameter. The cost is negligible, and one spool will last indefinitely. I have not found other cheap wires so readily obtainable of suitable fineness, well annealed and so reasonably resistant to chemical action. When platinum wire must be employed I use a wire not exceeding .05 mm. in diameter.

In making a specific gravity determination, a suitable length of the wire is prepared by twisting a loop at each end—one of the loops being intended to hang upon the hook of the balance, and the other being intended for the suspension of the plummet by its hook. Two such wires are prepared—substantially exact duplicates of each other. The analyst, having ascertained the depth to which the lower end of the suspensory wire is to dip beneath the surface of the liquid, cuts off that much of one of the two prepared suspensory wires and then weighs the two parts separately—so as to ascertain what fraction of the weight of the other wire will be immersed in the liquid during the actual specific gravity determination—the pieces being then discarded. The other or actual, suspensory wire and the plummet are now weighed separately, and to the weight of the plummet is added that fraction of the weight of the suspensory wire which is immersed. We will call this combined weight of the plummet and the immersed portion of wire the *weight of the plummet*. The liquid is now brought into the Dewar cylinder at a definite temperature. This is accomplished by immersing a thermometer in the liquid contained in another vessel, bringing the liquid by usual methods of manipulation to the desired temperature and then pouring it backward and forward from the auxiliary vessel to the Dewar cylinder, etc., a sufficient number of times to bring the inner glass walls of the cylinder to the same temperature as the liquid—after which the cylinder is filled to a suitable depth and the liquid will now maintain this temperature practically unchanged throughout the experiment. There is, of course, some opportunity for change of temperature at the surface of the liquid by its contact with the air; but even this local alteration may be made negligible during the time of the experiment by the use of a perforated cover. The weight of the plummet whilst suspended in the liquid is now determined and the usual calculation made: that is to say, from the weight of the plummet in air is subtracted the weight of the plummet in the liquid, and this difference in weight is divided by the difference similarly obtained when the observation is made upon pure distilled water. Of course, temperature plays a

part; and in alcohol determinations the observations should be made at such temperatures as may eventually be officially directed. In making weighings with the plummet immersed, equilibrium must be established with the needle of the balance stationary at zero and the wire immersed to the predetermined depth.

It is believed that the method just described is the most accurate method which could be recommended in the premises. It requires no special apparatus except the special Dewar cylinder.\*

*The Pycnometer Type of Dewar Vessel:* This type is shown in the accompanying etching as No. 3. The pycnometer is best made from Jena thermometer glass, because in this case its capacity at stated temperature once determined, remains fixed—which is not the case when ordinary glass is employed. The apparatus consists of a double-walled glass flask having a vacuous space between the walls and having an accurately ground glass stopper which is one piece with an inserted thermometer graduated from minus 4° to plus 35° C. by 1/5°. Capacity of inner vessel with stopper inserted, 25 cc. Total weight of apparatus empty, 40 grams. Dimensions in millimeters are: total height over all with thermometer inserted, 210; total height of flask, 100; greatest diameter, 44. In ordering such an apparatus care should be made to specify very accurate grinding of the stopper and the absence of any annular channel exteriorly between the stopper and the neck of the flask. Unless this specification is made the maker is apt to be a little careless and to leave an annular space which is somewhat troublesome to wipe dry before weighing.†

The advantage which the apparatus under discussion has over the usual pycnometer arises from the fact that temperature alterations of the investigated liquid, which are the bane of ordinary pycnometer measurements, are here eliminated because of the thermal insulating property of the vacuous envelope.

The apparatus is used like an ordinary pycnometer, but it is probably desirable to state that the liquid is brought to the specified temperature exactly as in the case of the hydrostatic balance method described above.

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\*This has been furnished from Germany to the Medico-Chirurgical College of Philadelphia at 56c. duty-free. It may further be interesting to note that the writer uses this Dewar cylinder also for pyrometric measurements by the principle of the water calorimeter pyrometer. It is only necessary to have a small lump of copper and an ordinary mercurial thermometer in addition to the Dewar cylinder in order to be enabled to make convenient, rapid and reasonably accurate measurements of temperatures up to 1000° C.

†Such an apparatus made in Germany, costs \$1.12, duty-free.

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## GENERAL METHOD PROPOSED FOR THE ESTIMATION OF ALCOHOL IN PHARMACEUTICAL PREPARATIONS.

W. A. PEARSON.

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It is with much timidity that I propose a general method for the estimation of alcohol in pharmaceutical preparations, because so much has been written on this subject and no two authors have agreed on any technic for the determina-