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APPARATUS FOR DISTILLING ALCOHOL FROM BIOLOGICAL FLUIDS AND A CALCULATOR FOR HARGER TITRATIONS

T. U. MARRON AND C. S. BANWARTH

For the determination of alcohol in body fluids the Harger (1935) titration is extensively used with distillates obtained in various ways; e. g., by steam distillation, desiccation, distillation from picric acid, picrate-tartrate or tungstic acid. Gibson (1939) has combined it with the simple distillation from picric acid solution (Nieloux, et al. 1934) to produce an essentially satisfactory and rapid determination. Clinical simplicity with research accuracy has been achieved by the use of a specially designed distillation apparatus with the method (Johnston and Gibson, 1940).

After using this method for a long period the authors have been able to revise it for still more convenience in use, and to design a calculator for obtaining the alcohol concentration in a body fluid directly from the burette readings of Harger titrations. This paper presents the revised design of the apparatus, with some modifications in use, and the calculator.

DISTILLATION APPARATUS

The distillation apparatus, Fig. 1, of Pyrex glass is particularly adapted to recovery of alcohol from body fluids after the addition of picric acid. Nevertheless, it is desirable for a variety of distillations of similar nature.

An ordinary ring stand with conventional clamps serves to hold the assembled apparatus. A micro burner is used for heating the sample flask. When the distillation has been completed the Vigreux tube and accompanying flask are simply separated from the water condenser, which is left attached to the receiving flask. The sulfuric acid is then introduced into the distillate through the condenser, carrying all condensation in the inner tube with it. The stand and apparatus are shaken to mix the contents of the flask. After about a minute a little distilled water is used to wash the substances from the condenser into the flask. The flask can then be removed and the condenser is ready for another determination. By the introduction of acid and water through the tube that is used for condensation, the time the apparatus is in use per determination has been reduced appreciably.

The number of flasks to be supplied with the set is governed by the number of determinations to be made at one time.

The design has a number of advantages over the Johnston and Gibson apparatus. Flasks of identical volume and standard taper are more conveniently obtained and used. The standard taper joint between the Vigreux tube and condenser facilitates the replacement of either one in case of breakage. The vapor tube in the condenser has been centered to reduce strain and awkwardness in apparatus alignment during assembly. Since addition of the acid through the vapor channel increases accuracy and precision, an air vent replaces the second condenser tube, formerly used for acid introduction.

CALCULATOR

Fig. 2 illustrates the calculator design. The materials and construction of the one in use in this laboratory are as follows:

S, U, and C scales are inked on white Bristol board, 28" x 40", glued to $\frac{1}{4}$ " Masonite. The S line is 21 $\frac{7}{8}$ " long, 2 $\frac{1}{2}$ " in from the left edge and begins 3 $\frac{3}{8}$ " from the top. The U line is 21 $\frac{7}{8}$ ", erected perpendicular to S. The scales on both S and U are equal and divided into 35 sections, $\frac{10}{16}$ " each, representing 35 ml. Each subsection is subdivided into 10 units, $\frac{1}{16}$ " in length, for 0.1 ml. values. The zero is placed at the junction of the two lines.

The mechanical device is made of 4 bars cut from steel strap, $\frac{3}{8}$ " wide by $\frac{1}{16}$ " thick. Bar A is 32 $\frac{1}{16}$ " long; B is 34" long and tapered to a sharp point at one end. The two smaller bars are 14 $\frac{1}{16}$ " in length with rivet holes drilled $\frac{3}{8}$ " from each of the ends. Rivet holes in A are $\frac{7}{16}$ " and 18 $\frac{1}{2}$ " from one end; in B they are drilled $\frac{3}{8}$ " and 30 $\frac{3}{8}$ " from the square end. The two short bars are riveted to the upper surface of A at the $\frac{7}{16}$ " and 18 $\frac{1}{2}$ " drillings, and to the corresponding surface of B at the 12 $\frac{3}{8}$ " and the 30 $\frac{3}{8}$ " drillings, respectively. Thus, a parallel bar arrangement is formed. The rivets are just tight enough to hold any parallelogram made by hand movement of the bars. Felt pads are cemented to the under surface of the rivets to prevent scratching. Through the third hole in B the apparatus is attached to the board by riveting $\frac{1}{4}$ " above the 1 $\frac{13}{16}$ " to the right of the 35 ml. mark on line S. This rivet is loose enough to allow free motion of the bars in one plane.

Scale C is an arc of a circle marked off by the point of B as it

Distillation Apparatus.

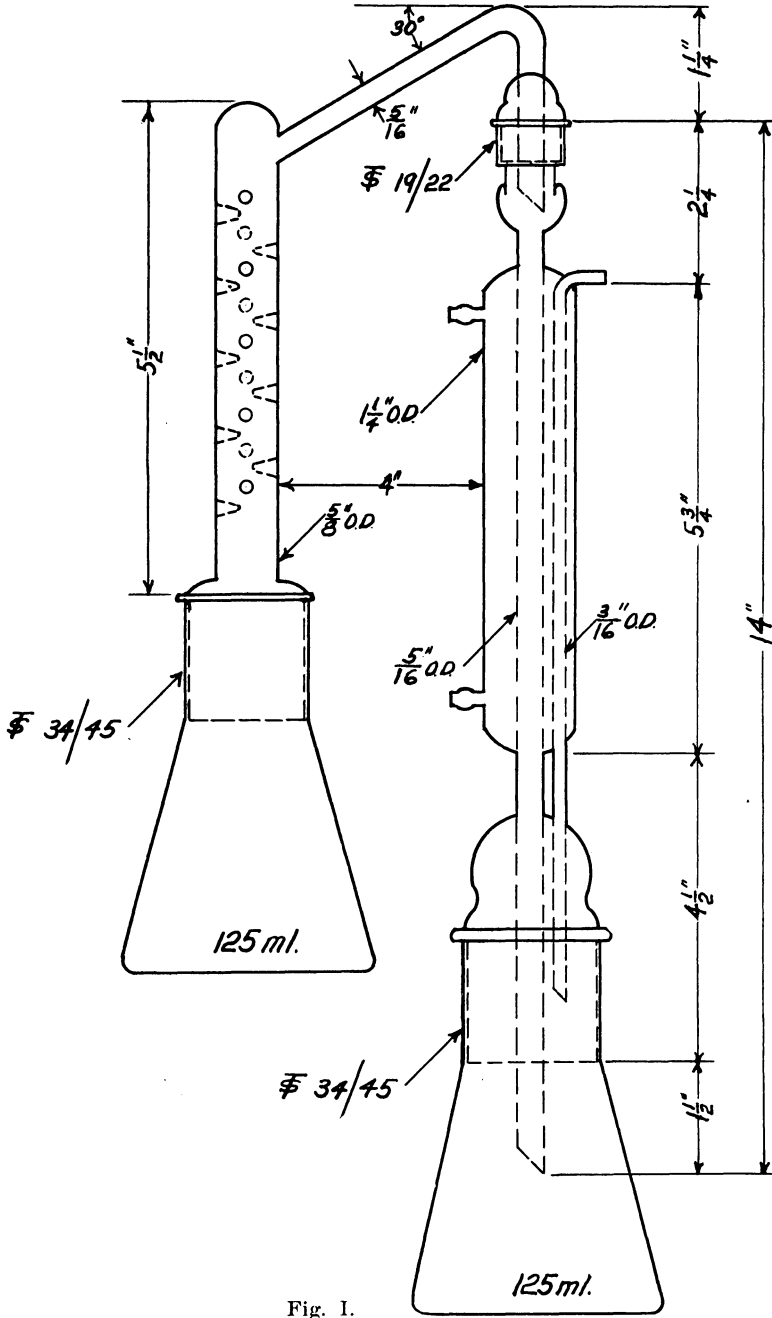


Fig. 1.

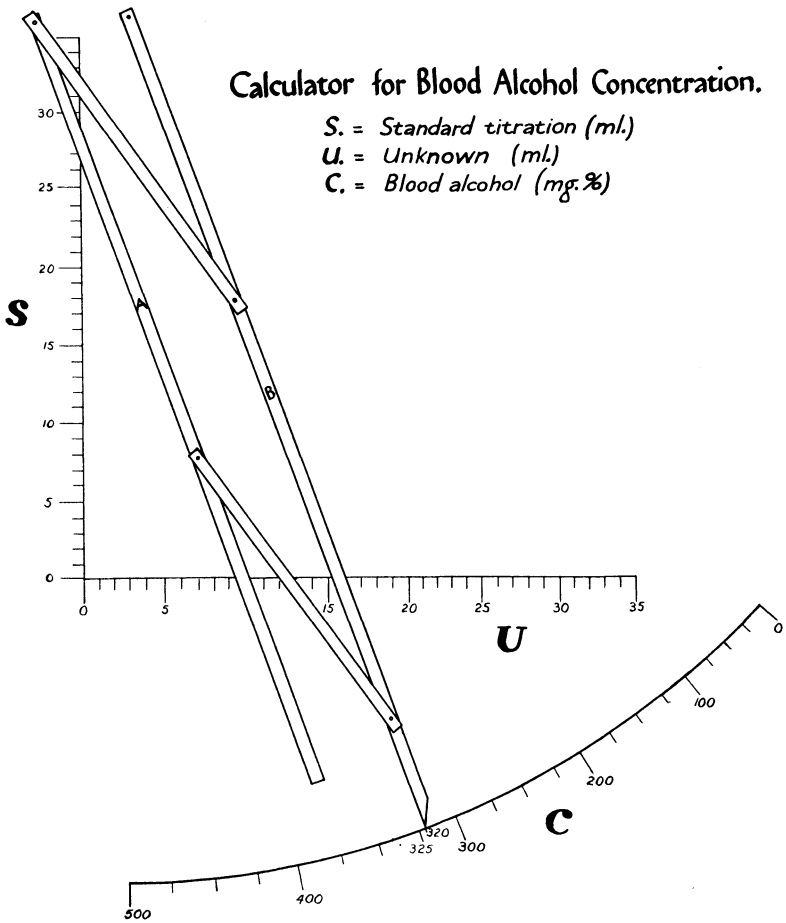


Fig. 2

is passed over the board. The scale goes from 0, where the arc runs off the board at the right, to 500, where the point of B rests when this bar is parallel with line S. There are 100 divisions in the arc, numbered at every fifth division. Each division corresponds to 5 mg. %. For various simultaneous values of U and S the values on the C scale were calculated from the formula,

$$10(1-U/S) \times 0.5 \times 100 = \text{mg. \% alcohol} = C$$

(Where S=ml. of red reducing fluid used to reduce 10 ml. of the standard dichromate; and U=ml. of the same fluid used to back titrate the portion of 10 ml. of dichromate not reduced by the alcohol in a distillate representing 1 ml. of sample.)

To use the calculator, bar A is placed so that its outer edge connects the standard titration value on S with the unknown titration value on U. Bar B will have followed the adjustment so that its point will rest on C at the alcohol concentration of the fluid tested.

The board is so constructed that B points to 250 when $2U = S$. This arrangement is for calculations when the distillate from a 1 ml. sample of blood, urine, saliva, spinal fluid or any other fluid has been allowed to react with 10 ml. of potassium dichromate having an ethyl alcohol titre of 0.5 mg. Other aliquots used merely necessitate a division of the result by the fraction of one ml. represented by the sample. However, if the dichromate and sulfuric acid are reduced in the same proportion as the sample, as in the micro method of Marron (1940) the scale reading still gives the alcohol value directly.

The calculator will take care of Harger titrations when the ferrous sulfate-methyl orange solution used does not exceed 35 ml. for the standard. Greatest accuracy occurs in the region where 20 ml. of the reducing fluid are equivalent to 10 ml. of potassium dichromate solution (2.1288 gm./L.). Such a balance can be achieved by adding a little more ferrous sulfate solution than Harger (1935) recommends in making up the red fluid. Since the titration value of ferrous sulfate solution varies with age, it is usually added in greater or less quantity anyhow.

Readings to within 1 mg.% of the mathematical value can be obtained on the calculator. The instrument could be reduced in size by one-half or more without loss of accuracy if the mechanical part were to be cut from a plastic strip of smaller dimensions than the metal, with an etched line for accurate settings, and if the riveting were somewhat more refined.

Use of the calculator is far more rapid than figuring values from the formula, and has a time advantage over slide rule calculations. Both calculator and slide rule are used in this laboratory to check results. If either is to be used alone, the calculator is preferred because the greater simplicity of its numerical system reduces the possibilities for error in the hands of technical assistants.

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