

mine its density at 25°, and at once compute its hydrochloric acid content with a precision sufficient for any ordinary volumetric work. Any reference to the pressure of the distillation is thus avoided, it being only necessary to see that the barometric pressure is not changing rapidly during the preparation of the constant boiling acid.

Foulk and Hollingsworth, in the work already cited, found that the composition of the constant boiling acid was influenced markedly by the rate of the distillation. We have been unable to corroborate this. We have made, at various pressures, series of distillations in which the rate was varied from 1.7 cc. to 4.9 cc. per minute. These distillates were compared by determining the densities, and in no case did we find a difference in density as great as 1 part in 10,000. For example, in a set of ten such distillates, the mean density was 1.09709, with an average deviation of 0.00004 and a maximum deviation of 0.00009. We would conclude that the rate of distillation has a negligible effect on the composition. However, for the sake of convenience, we carried out all of our distillations at the uniform rate of 4.5 cc. per minute.

### Résumé

We have determined the composition and density of constant boiling hydrochloric acid between pressures of 50 and 1220 mm.

We have given an equation by the aid of which the composition of any constant boiling hydrochloric acid may be computed from its density, at 25°. The use of this acid as a primary standard in acidimetry is thus simplified.

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### NOTES

**An Inorganic Stopcock Lubricant.**—Up to the present time there has been no inorganic material discovered which compares favorably in all respects with stopcock grease, although there are many types of research in which such a material would be extremely valuable. The two main makeshift substitutes which have been used are moist phosphorus pentoxide and moist metaphosphoric acid, both of which leave much to be desired as lubricants for high vacuum work. The former tends to leak and to "freeze," while the latter "freezes" unless the stopcocks are turned very frequently.

During attempts to develop a more satisfactory lubricant for use in work at present in progress, the writer discovered that a thick, sirupy liquid with practically no tendency to crystallize on standing could be obtained by preparing a solution of the three phosphoric acids in one another. This mixture may be prepared with almost any desired viscosity by simply varying the proportions of the components. Due to

the hygroscopic nature of the material, it must, of course, be protected from moisture but this can easily be done by using mercury seal stopcocks. Absorption of water causes crystallization to take place, with a simultaneous thinning of the remaining liquid phase.

It must be admitted that this mixture is not as satisfactory a lubricant as grease, and stopcocks on standing under vacuum tend to "freeze" to a certain extent. However, they can be loosened very readily by warming gently with a flame. This latter operation should be performed, if possible, when the system is not evacuated but can be performed under vacuum, if necessary. In the latter case the tendency to "freeze" is increased but in the former it is not.

Due to this slight tendency to "freeze" the stopcocks, it is advisable to re-grind the latter with fine emery powder before using with the phosphoric acid lubricant. If one takes the additional precaution of finishing with rouge, "freezing" will be reduced to a minimum.

For high vacuum work the following directions will yield a mixture that has been found by the writer to be very satisfactory. On heating the mixture of ortho- and metaphosphoric acids above the decomposition point of the former, there will also be formed some pyro acid. This increases the complexity of the mixture and probably hinders crystallization.

**Directions.**—Add 18 g. of metaphosphoric acid to 35 g. of 85% orthophosphoric acid, containing a drop or two of concentrated nitric acid, to destroy reducing substances. Heat to 300° in a platinum crucible, over a period of fifteen minutes, then hold at 300° for fifteen minutes. Cool to about 100° and transfer equal portions to three or four small bottles equipped with rubber stoppers. (Division into three or four portions is recommended because the lubricant is usually spoiled by the admission of moist air into the bottle before it is nearly all used.)

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**A New Regulator Allowing Rapid Changes in the Temperature Setting of the Thermostat.**—In the course of an investigation involving the use of a thermostat at widely different temperatures, the author has designed a vapor-pressure regulator that made possible rapid changes from one temperature setting to another. Although many forms of vapor-pressure regulators have been used before, it is believed that certain new features in the construction and operation of the device justify a detailed description of it.

The filling of the apparatus is carried out by a suitable application of vacuum and tilting. When ready for operation, it has both ends sealed. The bulb (Fig. 1) contains a vapor phase devoid of air, a suitable liquid and a layer of mercury. The space in the inclined tube above the mercury is evacuated. The whole rests on an iron bar (not shown in the figure) bent to fit the tube and capable of rotation around an axis at O.

As the thermostat is heated, the mercury level in the tube rises. When the desired temperature is reached, the upper wire from the relay is attached to the nearest electrode (tungsten wire sealed into the pyrex glass), and the apparatus is tilted to just make or break contact. After this the regulator will work for weeks without requiring any attention.

Using aniline as the source of vapor pressure, the author was able to obtain any temperature within the range 110 to 160° by using the proper electrode. The temperature was kept constant to within 0.1°.

It will be noticed that the tube is bent through an angle of about 45°. With the apparatus fixed, to every electrode there corresponds a certain temperature. Intermediate values are obtained by

tilting, at which time all points move along inclined arcs CO. Were the tube made vertical, the points of contact would move along nearly horizontal arcs AB, necessitating much larger angles of rotation for the same temperature shift. Incidentally, the sensitiveness of the regulator is increased by a factor of about 1.4.

The chief advantage of this apparatus lies in the ease with which the thermostat temperature setting can be changed. Furthermore, contact is made in a sealed tube, with no air present, thus eliminating any possibility of contamination.

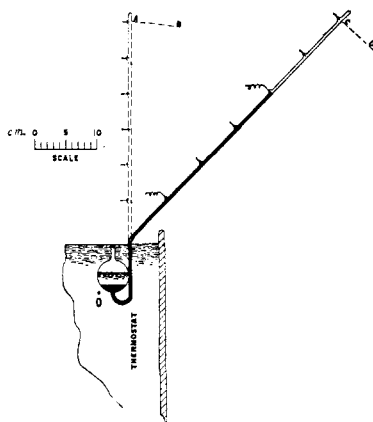


Fig. 1.

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