

practice, but the National Formulary makes no distinction between allopaths, homeopaths, osteopaths, chiropractors or any other school of practice. Its mission is to perfect or select the pharmaceutical qualities of the formulas, and to allow the physicians as a whole to adjust or judge the therapeutics. Hence there is not likely to develop any rivalry or antagonism between the two books. Since both Parts I and II contain assays and tests which require test solutions and some special test directions, it seemed wise to embody the more special tests in Part III. This is taken directly from the U.S.P. IX, by permission of the Board of Trustees, and is designed to make the Formulary more complete as a working book. Volumetric solutions and the usual test solutions are not included, although employed in N.F. tests, because these are made by standard and well-known methods. Only the special tests are included. This part will doubtless be used more by manufacturers than by retail pharmacists, but the latter should not overlook the fact that the standards of the National Formulary IV apply after September 1, 1916, to all stock sold under the titles therein. Pharmacists should go over their stock, and set aside for readjustment all preparations not conforming to the new standards.

ALCOHOL DETERMINATION BY DISTILLATION.

BY A. B. LYONS.

An official method for the determination of alcohol in pharmaceutical preparations appears in U. S. P. IX and also in N. F. IV. The principle is the familiar one of distilling from an accurately measured volume of the alcoholic liquid, diluted with sufficient water, an equal volume of distillate, the specific gravity of which indicates, by reference to an alcoholometric table, the percent by volume of anhydrous alcohol. It is essential that the measurements before and after distillation shall be made with exactness, and it goes without saying that in all exact measurements of liquids temperature is an important factor.

It has been the common practice to make these measurements at 60° F., the standard temperature for alcohol determinations. The air temperature in most laboratories is many degrees higher than this, the year around, and where the pycnometer is used for determination of the specific gravity, condensation of atmospheric moisture* makes accurate weighing difficult. Hence it has become customary to make the weighing at room temperature, although the measurement has been made at 60° F., the necessary correction being taken from an empirical table, such as is given in U. S. P. IX.

It is simpler to make the measurement also at room temperature, taking care only that this temperature is the same for both the measurements. The principle is sound, and *so long as the volume of the distillate is the same as that of the sample* taken for distillation, the results will be correct, provided the whole of the alcohol passes over by the time this volume is obtained—*i.e.*, in case of a liquid containing not more than 25 percent alcohol.

If this liquid contains more than 25 percent of spirit, it is necessary to carry this distillation further and the common practice has been to bring the volume of the distillate to just double that of the sample distilled. Now, however, we may no longer make our measurements at any but the standard temperature, *viz.*: 60° F. The reason for this is that the coefficient of expansion for mixtures of alcohol and water varies greatly with the proportion of alcohol present, being, for example, four times as great for 95 percent alcohol as for 8 percent. Consequently the error in the first measurement will be materially greater than that of the second if both are made at the same temperature.

The measurements may, however, both be made at 60° F., for the reason that volume percentages of alcohol are true only at 60° F. A 50 percent alcohol is one which contains at 60° F., one-half its volume of anhydrous alcohol, measured also at 60° F. If the temperature is raised to 75° F., the dilute spirit will contain more than one-half its volume of anhydrous alcohol measured at 75° F.

In determining alcohol in a liquid containing more than 50 percent, some advise to distil off four volumes for one, determining the percent of alcohol in the distillate and multiplying this figure by four. Obviously if this is done the error arising from making the measurements at room temperature will be considerably greater than in case the distillate is double the volume of the sample.

The official directions are to make the measurements at any convenient temperature, being careful only that both are made at the same temperature. The error involved where the temperature is not higher than 25° C. is negligible in case the percentage is not above 30. In case of stronger spirit in warm summer weather, the error may easily exceed 0.5 percent.

The official process should therefore be amended to require that both measurements be made at 60° F. The weighing can then be made at any convenient temperature, the percent being deduced from the official alcohol tables.

ACETYSALICYLIC ACID.

While the loosely worded test of the British Pharmacopœia for this article is quite unsatisfactory, there can be no doubt that the melting point has not been, as was recently alleged, fixed too high by that authority. The intensity of the ferric-chloride color reaction is undoubtedly in more or less direct relationship with the depression of the melting point, and, of course, with the amount of impurities present. An interesting series of experiments has recently been carried out by Tsakalotos and Horsch (*Bull. Chem. Soc.*, 1915, 17, 401), who have attempted to study the rate of decomposition of acetylsalicylic acid in aqueous solution at 23°, 50°, and 60°, the rate of decomposition being measured by removing 25 Cc. of the solution at given intervals and titrating it with one fiftieth-normal barium hydroxide. At 50° they also carried out a series of electrical conductivity measurements. In both cases the curves showed two minima, the times at which these occur being found to be dependent on the temperature. It appears to be generally assumed that the impurities and decomposition-products of acetylsalicylic acid are traces of salicylic acid and of acetic acid. But, although this may be, and probably is, to a great extent true, there is little doubt that the matter is not quite so simple as that generally assumed. There is no doubt that in the course of the reaction by which some manufacturers prepare acetylsalicylic acid a secondary reaction goes on. Whether it be while in the nascent state only or not cannot be definitely decided, but the acetylsalicylic acid first formed in the reaction itself reacts upon unaltered salicylic acid forming a further substitution-product, acetylsalicyl-salicylic acid, a product which probably decomposes in contact with water into aceto-salicylic and salicylic acids, the former in turn decomposing into acetic and salicylic acid. Hence the quantitative experiments of Tsakalotos and Horsch may well be considered indefinite, unless the acid experimented upon was free from acetylsalicyl-salicylic acid. There is reason to believe that the true melting point of acetylsalicylic acid is nearer 136°–137° than to any other figure, and that unless prepared by certain methods the impurities above indicated are exceedingly difficult, if not impossible, to eliminate.—E. J. PARRY in *Chem. and Drug.*, June 3, 1916.
