AN IMPROVISED NITROMETER FOR THE ASSAY OF SPIRIT OF ETHYL NITRITE.*

BY C. L. COX.¹

Due to the fact that Spirit of Ethyl Nitrite is such an unstable preparation, and so often found of sub-standard strength in the drug store it is almost imperative that it be assayed frequently to insure its meeting the official requirements. The official nitrometer is a satisfactory method of assaying this preparation, but the cost of the apparatus is too great for the average drug store to bear. For this reason it is not unusual to find many drug stores that do not have the facilities for assaying this important spirit.

With the purpose of developing a process for the assaying, that would require little or no expensive apparatus, I have been experimenting with the various methods suggested in the past. Titration of the liberated iodine requires a burette, as also does the saponification of the ester. The Squibb's ureometer method as applied to the assay of spirit of ethyl nitrite has several points of error, but there



Fig. 1.-Improvised Nitrometer.

are some principles in this process that form the basis of the nitrometer which I propose to use.

The apparatus I propose to use consists of about two feet of rubber tubing, a rubber stopper, a homeo vial, a graduate, a pinch cock, a wide mouth 8-ounce bottle, a tall 4-ounce bottle and about a foot of glass tubing.

The apparatus is set up as shown in the sketch. The 8-ounce bottle is filled with the regulation saturated salt solution and siphoned into the 4-ounce bottle until the 4-ounce bottle contains about 30 cc. of the saturated salt solution and all air has been excluded from the tubing. Now add about 10 cc. of dilute sulphuric acid and 15 cc. of potassium iodide test solution to the 8-ounce bottle. Tare the homeo vial on the prescription balance, fill with the spirit to be tested and again weigh. By means of a thread, lower the vial into the 8-ounce bottle until it just clears the stopper. Loosely insert the stopper and siphon the salt solution from the 4-ounce bottle until as much air as possible has been displaced from the 8-ounce bottle, taking care that the reagent does not enter the vial. Tighten the stopper and bringing the level of the liquids in both bottles to the same height, mark the level in the 4-ounce bottle. Now invert the 8-ounce bottle so as to mix the spirit in the vial with the reagent. Taking care that the gas generated does not pass through the tubing, agitate the bottle until the reaction is over. As soon as the temperature has become adjusted to that of the room, again bring the level of the liquids to the same height. Tighten the pinch cock on the rubber tubing, remove the stopper from the 8-ounce bottle and siphon the liquid from the 4-ounce bottle into a graduate until the mark of the first level has been reached. The liquid in

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the graduate represents the volume of gas liberated from the spirit. Calculate as directed in the U. S. P.

One source of error with this apparatus is that it is impossible to displace all the air from the 8-ounce bottle, about one cubic centimeter remaining. This reacting with the gas generated causes a contraction in volume. But as the air is only onefifth oxygen the error cannot be greater than one-fifth of a cubic centimeter. This is not greater than the error in the official nitrometer which is due to the fact that the saturated salt solution in the reservoir side of the nitrometer has a specific gravity of about 1.200 while twenty-five cubic centimeters of that in the calibrated side of the nitrometer has a specific gravity of about 1.000.

For practical purposes it is sufficient to ascertain that one cubic centimeter of the spirit evolves eleven cubic centimeters of gas. This is equivalent to about four per cent, or the mean of the pharmacopœial requirement and it would only be under abnormal conditions that barometric pressure would cause this to be above or below the official limit of toleration.

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A PHARMACEUTICAL STUDY OF MAGMÆ MAGNESIÆ-1900-1930.

BY A. J. LEHMAN, M.S.

Historical.—Magma Magnesiæ (Milk of Magnesia) made its official appearance in the N. F. III (1906). Previous to this little information can be found in the literature available, relative to this preparation. Aqueous suspensions of magnesium oxide were known and used at an earlier date, an example of which is the Magnesia usta in aqua in the 1871 Hungarian Pharmacopœia. In 1874, Wilder (1) offered a Calcined Magnesia Mixture consisting of 1 part of light calcined magnesia to 12 parts of water. The same year another writer (2) suggested the following formula for "milk of magnesia:"

Calcined magnesia	8 parts
Water	40 parts
Sugar	50 parts
Orange flower water	20 parts

The same article includes a formula for "Lac Magnesiæ" from the 1831 Schleswig-Holstein Pharmacopœia, as follows:

Calcined magnesia	2 parts
Rub uniform with distilled water	10 parts
Heat to boiling with constant stirring	
Remove from fire and add,	•
Powdered white sugar	12 parts
Orange flower water	4 parts

The Report on the Progress of Pharmacy (3) in 1881 includes the following statement under the heading of Milk of Magnesia: "Triturate 2 parts of calcined magnesia with well boiled or distilled water. Preserve in well-stoppered vials." Dietrich (4)