removal of the oil may be transferred to a distilling flask, the hydrochloric acid neutralized and the alcoholic distillate from 25 cc. of the original spirit obtained in better condition, as regards freedom from oil, than usually results by following the official method of diluting the original spirit and filtering through magnesium carbonate, which always occasions a slight loss by evaporation which cannot take place to the same extent under the procedure given above.

PHOSPHORIC ANHYDRIDE CONTENT OF SIMPLE AND COMPOUND SYRUP OF HYPOPHOSPHITES.

H. E. BARNARD AND W. D. MCABEE.

The attention of this department was recently directed to the great difficulty of holding all the ingredients of Syrup of Hypophosphites, both simple and compound, in solution. The solution of the various salts in water is easily accomplished but the addition of the sugar precipitates a part and if the product is immediately strained, this precipitate is removed and the resulting solution is lower in strength than was originally intended. Upon inquiry, we found that several manufacturers had noted this fact and were somewhat in doubt as to the actual content of finished product, but had assumed the loss to be immaterial.

The first difficulty encountered in our investigation was the adoption or formation of a suitable method for the determination of the amount of phosphorus present. It was at once apparent that the great amount of organic matter must be destroyed by some means that would not interfere later in the phosphate precipitation and for this reason the simple process of ashing was not applicable because all hypophosphites decompose at low heat into pyrophosphates with the evolution of hydrogen phosphide. The reaction is quite variable and the remaining phosphorus cannot be used as an index of the amount originally present.

A search of the literature revealed the fact that while no method was recommended, the National Dispensatory states that the one of H. A. D. Jewett is "seemingly" accurate for the estimation of phosphorus in calcium hypophosphite. Briefly this method consists in the liberation and oxidation of the hypophosphorus acid to phosphoric acid by means of bromine and the determination of the phosphoric anhydride in this condition. Any phosphites that might be present as impurities are removed by precipitation with lead acetate. We experimented with this method as outlined and obtained very satisfactory results on calcium, sodium and potassium hypophosphites alone and in the presence of a large amount of sugar. In the course of our experiments we found that the lead acetate gave but a slight precipitate even in a concentrated solution of a hypophosphite and the percent of phosphites in the dilute syrup could be neglected. We also, for convenience, substituted concentrated nitric acid for the bromine and evolved the following method in detail:

Determine the specific gravity with a Westphal balance or pycnometer. Weigh accurately two grams of the sample into a one hundred cubic centimeter beaker, add ten cubic centimeters concentrated nitric acid and cautiously bring to boiling.

A violent reaction occurs at this point and care must be taken to prevent loss through bumping and boiling. When this violent reaction has ceased, add gradually forty cubic centimeters concentrated nitric acid and boil for five minutes. Nearly neutralize with ammonia, or, if not sufficient nitric acid remains to form the ammonium nitrate necessary for the molybdate precipitation, make alkaline with ammonia and again make acid with nitric acid. Ammonium molybdate is then added, the precipitate dissolved in ammonia and the phosphorus precipitated with magnesium in the usual way.

Having found a satisfactory method, the next step was to procure samples and we were somewhat surprised to learn that the two pharmacopœial preparations comprised but a small part of the hypophosphite syrups on the market. The majority found by our inspectors were made from special formulas and as the analysis of such samples would not aid materially in the determination of the point in question, we were compelled to investigate fewer samples than we should have wished to do. While only six of the samples examined were labelled so that they were presumed to be U. S. P. strength, two others, made from formulas that called for approximately the U. S. P. quantity, are comparable because of their nearly equivalent concentration. One sample examined claimed to be only ten percent of the pharmacopœial strength and three others contained no label statement and were so qualified as to prevent their being classed U. S. P. strength. The following table gives in detail the results of the analysis together with the label statement.

No.	Label.	% U. S. P. Strength. Label.	Found
1	Syrup Hypophosphites	100.	12.74
2	Comp. Syrup Hypophosphites	100.	15.72
3	Syrup Hypophosphites Comp. with Quinine	100.	28.89
4	Syrup Hypophosphites Comp., Churchill's Formula	111.	107.44
5	Comp. Syrup Hypophosphites	100.	43.04
6	Comp. Syrup Hypophosphites	100.	9.87
7	Syrup Hypophosphites	100.	96.59
8	Syrup Seven Hypophosphites	101.	91.59
9	Comp. Syrup Hypophosphites	10.	11.68
10	Syrup Hypophosphites, Lime and Soda	....	42.38
11	Syrup Hypophosphites, Hematic	....	23.49
12	Nutritive Hypophosphites	....	17.00

In this table the percent U. S. P. strength as indicated by the label was assumed to be one hundred when the formula was not given and the label was not qualified. When the formula was stated the U. S. P. strength was calculated from it directly, and where no formula was given and the label was qualified in some manner, this column has been left blank.

As may be readily seen from a glance at this table, only one sample, number four, contains the amount of phosphoric anhydride required by the Pharmacopœia. This sample was made from Churchill's formula which differs from that of the Pharmacopœia by increasing the quantity of the more soluble sodium salt and decreasing the less soluble calcium hypophosphite. While the excessive strength of this sample may be explained in this manner, the fact remains that if a correction is made for the two percent impurities in the ingredients, as allowed by the Pharmacopœia, sample number seven is very nearly legal and was made strictly according to the U. S. P. formula. The other samples clearly

demonstrate that the hypophosphite preparations now on the market are woefully below standard and more care must be used in their manufacture than has been done heretofore.

INFLUENCE OF SIZE AND SHAPE OF BOTTLES UPON THE ASSAY OF PEPSIN.

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In a previous paper, entitled, "Some Observations upon the Assay of Digestive Ferments," appearing in the *Journal of Engineering and Industrial Chemistry,* (Vol. 3—No. 12—December, 1911), I gave a resume of tests applied by me and also called attention to some of the peculiarities of these delicate enzymes shown in their standardization.

Under the subject of Pepsin, I was the first to call the attention of chemists to the influence of the age of the egg upon the apparent digestive strength of a pepsin when using coagulated egg albumen as the proteid to be digested. I showed that eggs from 5 to 10 days of age gave a maximum digestive strength to a sample of pepsin. I also called attention to the important part played by the strength of acid used for digestion and showed that the pepsin dissolved in an acid solution of more or less than 0.3%, by weight, absolute hydrochloric, would not digest as much proteid as when dissolved in a menstruum of exactly this official strength (0.3%).

Another factor, overlooked by chemists in the past and very important from the standpoint of accuracy and uniformity of results in standardization by various chemists, is the size and shape of the bottle used in the digestion experiments. The United States Pharmacopœia states, in reference to the digestion bottle used with pepsin standardization, to digest in a wide-mouth bottle of 100 cc. capacity; but the question arises, do you get the same relative amount of digestion in a short round bottle of 100 cc. capacity that you do in a taller square bottle of the same volume? From the results of my experiments, my answer is "No"; different bottles give different results. This is illustrated by the following experiments:

Pepsin Sample No.	Style of Bottle.	Strength Tested for	Residue
Standard 1:3000	(No. 1) 6 oz. French square wide mouth, capacity 175 cubic centimeters. 5¼″ Tall.	1:3000	1 cc.
1:3000	(No. 2) 4 oz. French square wide mouth, capacity 120 cc. 4¾″ Tall.	1:3000	1 cc.
1:3000	(No. 3) 3 oz. French square wide mouth, capacity 90 cc. 4½″ Tall.	1:3000	1 cc.
1:3000	(No. 4) 3 oz. Round, Prescription wide mouth, 100 cc. capacity. 4″ Tall.	1:3000	1¼ cc.
1:3000	4″ Tall.	1:2750	1 cc.

In the above experiments the eggs were but five days old and were too fresh,