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THE ANALYSIS OF NITROGLYCERIN TABLETS.

BY MEYER SAMSON.*

The following method for the analysis of nitroglycerin tablets is a modification of the Hay nitrite method as given in the "Methods of Analysis" of the Association of Official Agricultural Chemists. The purpose of these modifications is to avoid the following errors or inconveniences of the old method:

(*a*) Inconvenience of extraction:

The old method involves in the case of machine-made tablets the passing of a "mess" of starch granules from one separatory funnel to another. Refusal to pass through the stopcock and sticking to the sides of the funnel with resultant incomplete extraction are of frequent occurrence. This is now avoided by the use of chloroform in the extraction of the nitroglycerin.

(*b*) Incorrect standard:

The preparation of a standard nitrite solution from silver nitrite involves a nitrite loss of 1% to 2.5% as shown by Laird & Simpson (*J. A. C. S.*, Vol. XLI, page 531). This gives correspondingly high results in the analysis of nitroglycerin tablets. This error is avoided by standardizing the nitrite solution against permanganate as suggested by these authors (*Ibid.*, page 528).

PREPARATION OF SAMPLE.

Introduce 20 tablets directly into a 60-cc. glass-stoppered separatory funnel. Moisten with 5 cc. water and extract with 10 cc. chloroform. Pass the chloroform through a pledget of

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purified cotton moistened with chloroform into a 50-cc. volumetric flask. Repeat the extraction with 10-cc. portions of chloroform three times, then rinse the stopper and stem of the funnel through the cotton pledget with chloroform. Fill to the mark and mix thoroughly. Place a 5-cc. aliquot in a 100-cc. volumetric flask, fill to the mark with ether and mix well.

This procedure should take about ten or fifteen minutes. The chloroform settles readily, and the time-consuming "messes" of the old method are avoided.

REAGENTS.

(*a*) Sulphanilic acid solution:

Sulphanilic acid 5 Gm.
Concentrated hydrochloric acid 50 cc.
Water to make 1000 cc.

This is identical with Ehrlich's Diazo Reagent, used in urinalysis.

(*b*) Alpha-naphthylamine hydrochloride solution:

Boil 2.5 Gm. alpha-naphthylamine hydrochloride with 25 cc. conc. HCl and 500 cc. water till dissolved. Filter into a liter volumetric flask. Cool and make up to volume with water.

The addition of acid to the reagent avoids in large part the precipitation of the base by the alkalinity of the glass and renders it useful for several years.

(*c*) Standard nitrite solution:

Dissolve about 3.5 Gm. sodium nitrite stick in 500 cc. water. Determine the nitrite content according to a slight modification of the method of Laird & Simpson (*J. A. C. S.*, Vol. XLI, page 524) as follows: Place an excess $N/10$ potassium permanganate in a flask (about 40 cc.), add 10–20 cc. dilute sulphuric acid (1–6) and run in slowly from a burette a measured amount of the nitrate solution (about 25 cc.) stirring constantly. Now add a measured excess of $N/10$ oxalic acid (about 15–20 cc.) and proceed as usual in titrating an oxalate with permanganate.

Dilute a measured amount of this known nitrite solution with nitrite-free water so that one cc. shall contain 0.16 mgm. nitrite nitrogen. This is the strong standard solution. To prepare the standard solution used in the analysis dilute 10 cc. of the strong standard to a liter with nitrite-free water. One cc. of this solution contains 0.0016 mgm. nitrogen, equivalent to 0.0128 mgm. nitroglycerin.

The standard solutions should be protected from atmospheric oxidation and bacterial decomposition by overlaying with toluene.

DETERMINATION.

Place 10 cc. of the ether solution in a 50-cc. beaker, dilute with 10 cc. alcohol and add one cc. $N/2$ alcoholic potassium hydroxide. Cover with a watch glass and allow to stand ten minutes. Place on a steam-bath and when the ether boils remove the watch glass. When most of the liquid has evaporated add 10 cc. nitrite-free water and evaporate until the odor of alcohol can no longer be detected. This solution represents 0.2 of a tablet. Introduce it directly into a 100-cc. volumetric flask. Dilute with sufficient nitrite-free water to make the volume about 90 cc., add one cc. dilute hydrochloric acid, then 2 cc. of the sulphanilic acid and 2 cc. of the naphthylamine solution, complete the volume with nitrite-free water and mix thoroughly.

At the same time the standard solution, which is also a blank on the reagents, should be prepared. Place 9.5 cc. ether and 0.5 cc. chloroform in a 50-cc. beaker, add the alcohol and $N/2$ potassium hydroxide and proceed parallel with the determination up to the point of diluting to 90 cc. Here add first 10 cc. of the standard nitrite solution equivalent to 0.128 mgm., or 1/500 grain nitroglycerin, and then go ahead as in the determination.

Allow to stand 30 minutes (avoiding direct sunlight) and then compare the two solutions in a good colorimeter. If the Duboseq biological type colorimeter is used, setting the standard at 30 will give a good depth of color for comparison. Daylight is best for the reading but a colorimeter lamp with a "Daylight" glass window may be used.

All the details given above are worked out for 1/100 grain compressed tablets which are by far the most common nitroglycerin tablets used. For tablets of other grainages a proportionate aliquot must be taken for the final analysis. Results checking within 0.5% of each other should be easily obtained.

The use of the nitrite method in routine work in preference to the nitrate method is advocated for the following reasons:

(*a*) No evaporation in a vacuum desiccator is necessary.

(*b*) The pink color resulting from the nitrite method can be more accurately read on a colorimeter by most people than the yellow color resulting from the nitrate method.

With careful work, however, the nitrate method gives accurate results and it may be used as a check if desired, or routinely by those who prefer the yellow for colorimetric work.

NOTES ON THE VARIABILITY OF COMPOSITION OF U. S. P. SYRUP OF WILD CHERRY.*

BY L. F. KEBLER AND W. F. KUNKE.[1]

During the examination of a sample of syrup of wild cherry, purchased as of U. S. P. quality, it was found that the sucrose content was materially lower than that calculated from the formula. In fact, a large portion of the saccharine material was in the form of reducing sugar. Another brand of wild cherry syrup was purchased and it likewise proved to be low in actual sucrose content.

Examinations of certain galenicals, marketed under trade names, have also been found to contain considerable quantities of invert sugar compared with the sucrose claimed to be present. Manufacturers generally contend that they do not use glucose but sucrose only in preparing their goods. No reasons are usually offered to explain the presence of the invert sugar. In one instance a chemist, with the formula available to him but not to the Government, made the point that owing to the fact that a considerable quantity of the sucrose had been inverted, some combination of drugs possessing unknown or unrecognized medicinal properties might be present.

The literature contains some observations on the inversion of sucrose in medicinal syrups. F. W. Haussmann,[2] who reports some of the earliest results, found that the sucrose in the regular acid syrups was materially inverted in from one day to four months. In the case of the syrups prepared with plant drugs the inversion was comparatively small. He tested Althæa, Senega and Lactuacarium syrups. Among other observers may be cited Joseph L. Mayer[3] and G. W. Lloyd Plette.[4]

It was then decided to prepare a U. S. P. wild cherry syrup, according to the present Pharmacopœial formula and ascertain the degree and rapidity of inversion of this syrup under several conditions. One sample was kept in a bottle at room temperature and another in an ordinary ice-cooled refrigerator. In a little over four months practically all of the sucrose in each sample was inverted. The in-

* Presented at the Asheville meeting, 1923, Scientific Section.

[1] Bureau of Chemistry, U. S. Department of Agriculture.

[2] *Am. J. Pharm.*, 70, 585, 1898.

[3] JOUR. A. PH. A., 4, 945, 1915.

[4] *Ibid.*, 7, 609, 1918.