

Among these twelve articles are a number that are distinctly toxic, and all are indisputably active therapeutically.

Crocus, sold in 41%+, Fenugreek in 37%, Logwood in 35% and Rape Seed in 36%, may be objected to as not being specifically therapeutical in their use.

The following are sold in 25% or more, but in less than 35%:

Cassia Buds	34%	Citrullus or Watermelon Seed	30%
Cataria or Catnip	30%	Populus Buds	33%
Cinchona Yellow (specifically sold as such)	25%	Resina Draconis	31%

Here we have in all thirty-one drugs sold in 25% or more of our pharmacies, all therapeutically active, but not in the Pharmacopœia, except the Cinchonas and Saigon Cinnamon. It remains to be noted that many drugs not sold in so many as 25% of the pharmacies are already in the Pharmacopœia for other reasons, principally because they are used for preparations that are recognized. Of these, I name Cantharis 24%, Ipecac and Guaiac 3%, Lobelia 17%, Kino, Stramonium, Podophyllum, Calumba and Styrax 15%, Senega, Spearmint and Cimicifuga 13%, Opium and Granatum 12%, Rhus and Gambir 10%, Herberis and Ergot 9%, Stavesacre 6% and Krameria 5%. A number of other drugs sold in less than 25% of the stores, should be included because of the demand for their preparations.

Whether the pharmacist does or does not manufacture his own preparations, his professional status assumes that this is within his province. Should he do so, it is quite as important that he use a standard drug as that a drug which he sells entire should be of standard quality. Of such drugs, I mention the following: Taraxacum and Triticum 19%, Salvia 15%, Caulophyllum 13%, Hamamelis 10%, Leptandra, Apocynum and Viburnum Prunifolium 6%.

DETERMINATION OF TOTAL CITRIC ACID IN SOLUTION OF MAGNESIUM CITRATE.

BY JOSEPH L. MAYER.

From the nature of the preparation and experience gained in its analysis it appeared to me that the U. S. P. IX did not go far enough in simply establishing a standard for magnesium in **Solution of Magnesium Citrate**; I, therefore, suggested that the U. S. P. X should include in addition standards for *free and total citric acid* and supplied methods for their determination. Being an auxiliary member of sub-committee 7, "Inorganic Chemicals," of the revision of the United States Pharmacopœia X, I saw the methods suggested by me go through the various *Bulletins*, but in an effort to bring them into accord with the procedure in the U. S. P. X under the "Assay for Alkali Salts of Organic Acids," they have been weakened; the method for total citric acid now reads as follows:

"Boil down exactly 10 cc. of the solution to about 5 cc. to expel carbon dioxide, dilute with 15 cc. of distilled water and titrate with half-normal sodium hydroxide, using phenolphthalein T. S. as indicator: it requires not less than 9.5 cc. of the alkali for neutralization (minimum of acidity). Concentrate the liquid and transfer it completely with the aid of a little distilled water to a platinum or porcelain crucible, evaporate to dryness, and proceed as directed under the assay for alkali salts of organic acids," page 431.

The method on page 431 directs that:

"After allowing the carbonized mass to cool, moisten with distilled water, ignite again and repeat the moistening and igniting until a white residue is obtained. Then disintegrate the ignited mass with the aid of a stout glass rod and transfer the mass and crucible to a beaker. Add 50 cc. of distilled water and titrate with half-normal sulphuric acid, using methyl orange T. S. as indicator."

This procedure applied to the determination of total citric acid in **Solution Magnesium Citrate** is not only time-consuming but is apt to lead to erroneous results due to the directions to "concentrate the liquid and transfer it to a platinum or porcelain crucible." The danger of loss can be avoided by following the method directed below in which the initial titration, evaporation, ignition and final titration are made in a platinum dish or a sufficiently large porcelain evaporating dish, adding an excess of half-normal sulphuric acid to the white residue on ignition, heating on a steam-bath and by means of half-normal sodium hydroxide determining the free acid in the presence of one or two drops of methyl orange. The U. S. P. should adopt the following method which leaves little to be desired with reference to ease of manipulation and accuracy:

Into a platinum or porcelain evaporating dish of about 100-cc. capacity accurately measure 10 cc. of solution of magnesium citrate which has been freed from gas by pouring from one container to another, and 10 cc. of distilled water, after which evaporate on the steam-bath until the volume is reduced to about 10 cc. then titrate the free acidity with half-normal sodium hydroxide, employing phenolphthalein as indicator. Now evaporate the neutralized liquid on the steam-bath until practically all the liquid is gone and then carefully ignite, allow to cool, moisten with distilled water and evaporate to dryness on the steam-bath and ignite again. Repeat the moistening with distilled water, evaporating and igniting until a white ash is obtained and then add to it 30 cc. of half-normal sulphuric acid and heat on the steam-bath for about 15 minutes; allow to cool, and titrate with half-normal sodium hydroxide in the presence of one or two drops of methyl orange.

If magnesium is to be determined on the ignited material, carefully transfer this neutralized liquid which results from the determination of the total citric acid to a 250-cc. beaker, add distilled water to make about 100 cc., 20 cc. of diluted hydrochloric acid and complete the assay as directed in the U. S. P. In an effort to determine whether the results obtained on magnesium by this procedure agree with those obtained by the U. S. P. method and whether the results differ when the determination is made in platinum or porcelain a sample of solution of magnesium citrate was analyzed with the following results:

A.—Original solution ignited in platinum, the residue repeatedly moistened with distilled water, evaporated and ignited until a white ash was obtained, diluted hydrochloric acid and water added and the analysis completed by the U. S. P. method showed the presence of 1.5476 Gm. MgO per 100 cc.

B.—Neutralized liquid resulting from determining total citric acid in platinum dish, diluted hydrochloric acid and water added and the assay completed by the U. S. P. method showed the presence of 1.5660 Gm. MgO per 100 cc.

C.—Neutralized liquid resulting from determining total citric acid in porcelain dish treated as under *B* showed the presence of 1.5868 Gm. MgO per 100 cc.

The U. S. P. states that after the evaporation of the solution of magnesium citrate in determining the magnesium, the diluted hydrochloric acid be added and "filter if necessary." Since the above manipulation yields a white ash in one case and a liquid with everything in solution in the other it is not necessary to filter before proceeding with the assay.

In the JOURNAL AMERICAN PHARMACEUTICAL ASSOCIATION, 9, 253 (1920), I published some results which showed that it mattered very little whether the magnesium was determined after ignition of the Solution Magnesium Citrate or directly in the original sample. The revision committee disregarded these results and again directs that the assay be made by first igniting the sample. I again tested out this phase of the subject by assaying the sample for magnesium by accurately measuring 10 cc. of the sample solution of magnesium citrate into a 250-cc. beaker, adding 20 cc. diluted hydrochloric acid, about 100 cc. distilled water, 5 drops of methyl red indicator, sodium phosphate test solution, ammonium hydroxide and completing the assay as directed by the U. S. P. The analysis showed the presence of 1.5570 Gm. MgO per 100 cc. This same sample, following *Method A*, which is the official one showed the presence of 1.5476 Gm. MgO per 100 cc. These results confirm the former ones and prove that it is unnecessary to ignite the material before determining magnesium in solution magnesium citrate. In determining magnesium it is directed to "gradually add ammonia T. S. with constant stirring until slightly alkaline." In the above analyses this point was ascertained by means of methyl red indicator. The magnesium precipitate was filtered on asbestos in Gooch crucibles.

The total citric acid present in solution magnesium citrate can quickly and accurately be calculated as follows, if the quantity of magnesium, free citric acid and citric acid combined with bicarbonate are known: Multiply the grams of Magnesium as MgO per 100 cc. by the factor 3.4743, to the result so obtained add the free citric acid per 100 cc. found upon analysis and to this result add the acid (0.4997 Gm. per 100 cc.) corresponding to alkali in the solution of magnesium citrate as bicarbonate. Since practically all manufacturers and pharmacists who make Solution of Magnesium Citrate employ bicarbonate slugs of exact weight and purity there should be little variation in the factor for citric acid combined with bicarbonate. (0.4997 Gm. per 100 cc.)

The following case illustrates the accuracy of this method:

The above referred to Solution of Magnesium Citrate contained 1.5476 Gm. of Magnesium as MgO per 100 cc., therefore 3.4743 multiplied by 1.5476 equals

	5.3768 Gm. citric acid combined with magnesium
Add	3.8872 Gm. free citric acid found upon analysis
Add	0.4997 Gm. citric acid combined with bicarbonate

Calculated Result.—9.7637 Gm. total citric acid per 100 cc. The amount of total citric acid found upon analysis was 9.7853 Gm. per 100 cc. These results check very closely.

SUMMARY.

1. The U. S. P. method for the determination of total citric acid in **Solution Magnesium Citrate** being unsatisfactory a better one is suggested.

2. Additional results confirm the findings published in 1920, that magnesium in **Solution Magnesium Citrate** can be determined much more rapidly and with as great accuracy with the original solution, ignition of the material being unnecessary.

3. The quantity of magnesium as MgO, free citric acid and citric acid combined with bicarbonate being known it is easy by means of a factory to calculate the quantity of total acid per 100 cc. of **Solution of Magnesium Citrate**.

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THE NEED OF GREATER ACTIVITY IN THE MAKING OF ANALYSES OF MEDICINAL PREPARATIONS FOUND IN THE OPEN MARKET AND OF A WIDER PUBLICITY OF THE ANALYSES.*

BY FREDERICK J. WULLING.

For many years Professor G. Bachman has been Chairman of the Committee on Drug Adulteration of the Minnesota State Pharmaceutical Association and, in that capacity has presented annually a comprehensive report setting forth the results of the analyses of many drugs and preparations found in the open market. These annual reports have been regarded as the most valuable, from a practical viewpoint, of all reports presented. They are actually used and consulted by members who constantly recognize their value. These reports are useful not only because of their value to the conscientious and careful pharmacist, but also because they, no doubt, discourage many of those who might otherwise make or use or sell drugs and preparations of inferior quality or strength. The work involved in the making of the many analyses reported upon annually, is very great and could not be done in volume except through the coöperation and assistance of many workers. Years ago, therefore, Dr. Bachman hit upon the idea of enlarging his course in the analysis and assay of drugs to include the practical application of his instruction to a larger number of U. S. P. and N. F. preparations and thus affording students increased practice in the application of the official tests for identity, purity and strength. This course has therefore not only increased the value of his instruction to students in the practical aspects involved, but has afforded opportunity, through the annual reports of the voluminous work done by himself, his faculty associates and students, to the pharmacists of the Northwest (for the reports are read and used in adjoining States) to learn the quality of medicines by the average pharmacists. Not only do pharmacists of the Northwest use and appreciate these reports but physicians and medical societies have recognized them and commented upon them most favorably.

To convey an idea of the scope of the analyses reported, let me say that the 1926 report (page 88 of the 1926 Proceedings of the M. S. P. A.) covered in all a total of 528 pharmaceutical preparations and chemicals. Samples were purchased from drug stores in various cities and sections of Minnesota. Dr. Bachman has made it a practice to purchase samples of drugs, chemicals and preparations recognized in the U. S. P. and N. F. whenever they are advertised at cut prices

* Scientific Section, A. PH. A., Philadelphia meeting, 1926.