

formula, therefore, which now consists of 30 percent Linseed Oil, a large saving of both oil and money could be introduced by the Pharmacopoeial Committee announcing permission for a substitution as is suggested above.

This possibility of conservation of Linseed Oil and the attending monetary saving occurred to the writer, particularly in its application to Government contracts for Liquor Cresolis Compositus.

It might be well then to call the attention of the reader to a few figures. The Army Medical Department in the last six months has let contracts for about 250,000 gallons, or at this rate there appears to be at least an annual consumption of 500,000 gallons of Liquor Cresolis Compositus. Thirty percent of this or 150,000 gallons represents the quantity of Linseed Oil necessary to produce these annual requirements. When this subject was under investigation in September, 1918, the *New York Commercial* was quoting Linseed Oil at \$1.88 per gallon, making the cost of 150,000 gallons equal \$282,000. At the same time other oils were much lower, such as Soya Bean Oil at about \$1.20 per gallon, 150,000 gallons of which had a value of about \$180,000. Because of the closeness in the saponification value of the two oils mentioned they can be interchanged without altering the proportion of the oil used, hence the replacing of Linseed Oil with Soya Bean Oil at these figures would mean an annual saving of \$100,000 and conservation of 150,000 gallons of Linseed Oil, and the product obtained with either oil would be comparable in every respect, physically and germicidally.

It has been found that Corn Oil or mixtures of Corn Oil and Soya Bean Oil may be substituted for either the Linseed Oil or Soya Bean Oil in this preparation.

It appears that the U. S. P. specifications should be appended by a statement giving the names and quantities of other oils than Linseed, which might be used in the manufacture of Liquor Cresolis Compositus.

THE ASSAY OF HYPOPHOSPHITES.*

BY J. L. DICKERSON AND J. P. SNYDER.

For the assay of official hypophosphites, the U. S. P. and N. F. direct that weighed samples of the salts which have been previously dried to constant weight in a desiccator over sulphuric acid be treated with two portions of nitric acid, evaporating to dryness after each addition. The residue is dissolved in water, neutralized with alkali, made to a definite volume and an aliquot portion titrated with potassium sulphocyanate, ferric ammonium sulphate used as indicator. Our experience with the above method has been uniformly unsatisfactory. The results we obtain are low, caused no doubt by the failure of the nitric acid to completely oxidize the hypophosphite to phosphate, and through possibilities of danger of mechanical loss in handling the process. Fairly good results may be obtained by oxidizing the solution of hypophosphite with nitric acid and potassium permanganate and using the double precipitation method with ammonium molybdate and magnesia mixture, finally weighing as magnesium pyro-phosphate. This method is long and tedious, requiring considerable manipulation, and unless great care is used in washing the precipitate of ammonium magnesium phosphate high results will be obtained.

By taking advantage of the fact that a solution of a hypophosphite, when

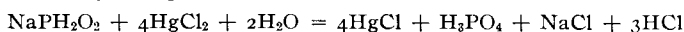
* Read before Scientific Section, A. Ph. A., Chicago meeting, 1918.

added to a solution of mercuric chloride, reduces an equivalent amount of the latter to insoluble mercurous chloride and collecting the precipitated calomel, treating this with an excess of iodine and back-titrating with sodium thiosulphate, we have been able to devise a method which works in a very satisfactory manner, gives good check results and takes a minimum amount of time and manipulation. The following is descriptive of the assay:

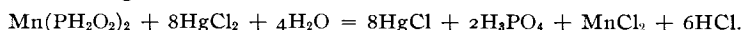
One gramme of the hypophosphite, which has been previously dried to constant weight in a desiccator over sulphuric acid, is accurately weighed and transferred to a 200 Cc. volumetric flask and made up to the mark with distilled water. When the salt has completely dissolved and the solution has been thoroughly mixed, 20 Cc. are pipetted into an Erlenmeyer flask of about 300 Cc. capacity, 20 Cc. of hydrochloric acid added, followed by 40 Cc. of mercuric chloride solution. The flask is placed upon the steam bath and allowed to remain there with occasional shaking for one hour or until the precipitated mercurous chloride is settled and the supernatant liquid is clear. The solution is filtered, the calomel collected upon a 11 cm. filter paper and washed well with distilled water. The filter paper and contents are transferred to the flask in which the precipitation was made, 3 to 4 grammes of potassium iodide added and 50 Cc. of *N/10* iodine (75 Cc. in the case of ammonium hypophosphite). Stopper the flask and let stand for about one-half hour, with occasional shaking. The excess of iodine is now treated with *N/10* sodium thiosulphate, starch solution as the indicator. 1 Cc. of *N/10* iodine is equivalent to:

Manganese Hypophosphite.....	0.002538 Gm.
Ammonium Hypophosphite.....	0.002077 Gm.
Calcium Hypophosphite.....	0.002127 Gm.
Sodium Hypophosphite.....	0.0026517 Gm.
Potassium Hypophosphite.....	0.002604 Gm.

The reaction may be expressed as follows:



and in the case of manganese which is divalent



It is essential that the solution after the addition of the mercuric chloride be heated, or otherwise the calomel will not be completely precipitated. At steam bath temperature, it was our experience that there was no danger of reducing the mercurous chloride to metallic mercury. It is advisable in order to be sure that the reaction has been complete, to return the filtrate to the steam bath and after standing a short while, examine for a further precipitation of the calomel. If this should occur, it is filtered and the precipitate treated as described above.

We have found that it makes no perceptible difference whether the precipitate of mercurous chloride was washed with dilute hydrochloric acid or distilled water. The mercuric chloride solution is prepared by shaking 5 Gm. of U. S. P. mercuric chloride with cold distilled water and filtering. Excellent results were also obtained electrolytically, by determining in this manner the amount of the mercury remaining in the filtrate, a definite amount of a standard mercuric chloride solution being added to the sample of hypophosphite. The mercury is deposited by a current strength of one ampere upon a revolving cathode and required about 20 minutes for a complete deposition. Undoubtedly, although we have not tried it, the residual mercuric chloride could also be estimated by one of the volumetric methods for this substance.