Query No. 8. Amount of solvent.

Most practical analysts who are regularly making alkaloidal assays are agreed that insufficient solvents are specified for extraction of alkaloids in many of the U. S. P. processes. For example, in the assay of Nux Vomica after oxidation of the Brucine the quantity of chloroform specified will not leave the supernatant liquid clear, nor will twice the specified quantity, but by repeated extractions with chloroform the supernatant liquid will become clear. Is an assay made in accordance with U. S. P. process, when excessive quantities are used? If additional quantities of solvents are allowable, should each extraction be made until no precipitate is obtained with Mayer's reagent?

Query No. 9. Identification of alkaloids.

In the determination of alkaloids from crude drugs the U. S. P. makes no provision for the identification of alkaloids extracted. Would it be advisable to insert identification tests for the alkaloids after they have been extracted and estimated?

Query No. 10. Physiological tests.

After the alkaloids have been extracted and estimated, would it be advisable to insert physiological tests and determine the minimum lethal dose and note the characteristic action?

Conclusion.

In presenting the above queries I realize that I am presenting problems that can only be settled by extensive experimental work. The main practical question is to decide how great these various factors probably are and whether the necessary co-operative work is to be undertaken.

ANALYTICAL DEPARTMENT, SMITH, KLINE & FRENCH CO.

SWEET SPIRIT OF NITRE, A SUGGESTION FOR A CHANGE IN THE FORMULA.

LINWOOD A. BROWN.

Owing to the fact that a very large number of samples of Sweet Spirit Nitre collected by the drug inspector for the Kentucky Agricultural Experiment Station, were found to be badly deficient in Ethyl Nitrite, and the statement by the druggists that they are unable to keep it so that it will retain its strength, has prompted this department to make a study of the question, endeavoring to determine whether the trouble was due to the formula or to the conditions under which it was kept, or both.

The Ethyl Nitrite used in preparing the spirit used in the following experiments was prepared by the formula given in the United States Pharmacopoeia, and which gave a yield of 78.5 grams Ethyl Nitrite. Time consumed in process, two-

hours and twenty minutes, the work being carried along with the usual laboratory work.

Experiments Nos. 1 and 2. Thinking perhaps the small amount of water present in U. S. P. strength alcohol might exert a hydrolizing effect upon the unstable Ethyl Nitrite, we prepared 440 grams of the spirit by diluting the Ethyl Nitrite with absolute alcohol (U. S. P.) and assaying by the U. S. P. method, using mercury, however, in place of the saturated salt solution in the nitrometer, and shaking the apparatus after the reaction was over until the solution became colorless.

By shaking the nitrometer until all of the liberated iodine has combined with the mercury, it seems to disengage the NO gas from solution more completely and allows quicker and more accurate readings.

The volume of gas was reduced to standard temperature and pressure of 25° C. and 760 m.m.

After assay, the above preparation was cooled and divided equally and placed in well ground glass stoppered, flint glass bottles, and labeled "Experiments 1 and 2," respectively.

Experiment 1 was placed in ice chest and kept at a temperature of 9-10° C., as were the other experiments, which were subjected to storage in ice chest.

Experiment 2 was placed on shelf in laboratory in rather bright, diffused light and exposed to a temperature of from 20 to 30° C.

Experiments Nos. 3, 4, 5, 6, 7 and 8. The material for these experiments was prepared by dissolving a portion of the Ethyl Nitrite in ordinary U. S. P. alcohol (containing 93.82% ab. alc. by vol.) assayed as before and divided as follows:

Experiment 3 was kept in ice chest at 9-10° C., in glass stoppered, flint glass bottles.

Experiment 4, kept in clear, glass stoppered, flint glass bottles on shelf in laboratory alongside of Experiment 2.

Experiment 5, kept in one-ounce, amber-colored bottles, stoppered with corks soaked in hot paraffin, and then the necks of the bottles dipped in paraffin; bottles kept in ice chest.

Experiment 6, same as Experiment 5, but kept on shelf in laboratory, temperature 20-30° C.

Experiment 7, same as Experiment 5, except it contained 0.25 grams powdered potassium bicarbonate in each bottle, kept in ice chest.

Experiment 8, same as Experiment 7, kept on shelf in laboratory, at temperature of 20-30° C.

Experiment 9. This sample was prepared with 90% alcohol. Sample kept in ground glass stoppered bottle in ice chest.

Experiment 10. Same as Experiment 9, but kept on shelf in laboratory in clear, glass stoppered bottle, in strongly diffused sunlight, temperature 20 to 30° C.

Experiment 11. This sample was prepared with 80% alcohol, kept in glass stoppered bottle, in ice chest.

Experiment 12. Same as Experiment 11, but kept on shelf in laboratory at 20-30° C., in strongly diffused light.

The following table shows the results of our analyses, and we believe show it is possible to make and keep Spirit Ethyl Nitrite of good quality:

Sample Assayed	Mch. 29, '11	Apr. 12	Apr. 26	May 10	May 24	June 7	June 21
Experiment No. 1....	4.36%	4.21%	4.11%	4.10%	4.02%	4.05%	4.01%
Experiment No. 2....	4.36	4.10	3.98	4.00	3.77	3.60	3.49
Experiment No. 3....	4.46	4.28	4.22	4.14	4.03	3.83	3.61
Experiment No. 4....	4.46	4.21	3.88	3.88	3.69	3.63	3.50
Experiment No. 5....	4.46	3.96	3.82	3.76	3.67	3.96*	3.91
Experiment No. 6....	4.46	4.05	3.93	3.77	3.55	3.89*	3.67
Experiment No. 7....	4.46	4.22	4.02	4.03	3.80	4.13*	4.09
Experiment No. 8....	4.46	4.24	4.01	3.81	3.65	4.08*	4.02
Experiment No. 9....	4.46	4.15	4.09	4.09	3.98	3.97	3.81
Experiment No. 10....	4.46	4.02	2.51	2.27	2.12	1.88	1.37
Experiment No. 11....	4.35	4.14	4.07	4.00	3.93	3.79	3.50
Experiment No. 12....	4.35	3.95	3.53	3.28	2.90	1.93	1.17

Conclusion. The deterioration of Spirit Ethyl Nitrite appears to be due to a number of contributing causes, chief among which are: (1) Hydrolysis of Ethyl Nitrite by the water contained in the alcohol used. (2) This change appears to be accelerated by the acid produced during the change. (3) Loss of Ethyl Nitrite by volitilization. (4) Effect of actinic rays of light on the Ethyl Nitrite.

Therefore, in the author's opinion, in order to produce the best and most efficient preparation it is necessary to use absolute alcohol U. S. P., in place of that now in use; to keep the product at a temperature not greater than 10° C. (50° F.) and to keep the product in as small a container as possible, better in the size package called for by the trade, and to protect it from the light by use of amber-colored bottles.

DISCUSSION.

Philip Asher stated that his experience had lead him to believe that the disturbing factor was the slight acidity of the alcohol employed. He had been able to overcome the difficulty by neutralizing the acidity with potassium bicarbonate, and had obtained a spirit of full Ethyl Nitrite content that kept fairly well.

COMBRETUM SUNDAICUM.

I. V. STANLEY STANISLAUS, PHAR. D., PH. D., AND HORATIO C. WOOD, JR., M. D.

Combretum sundaicum Miquel, according to Holmes (P. J., 1907, p. 77), is a shrub indigenous to Sumatra and belonging to the family *Combretaceae;* its leaves are said to have been in use for a long time by the Chinese for curing the opium smoking habit.

An infusion made from the previously roasted stalks and leaves and drunk, is said to quickly give rise to an aversion to opium smoking and hence originates the name of "anti-opium plant."

Upon analysis only tannins and gums have been found, and hence the difficulty to understand the action of the drug. A thought was advanced in Merck's

* Original one-ounce bottle.