samples tried, a list of which is appended, when no satisfactory results could be obtained, is with the fluidextract of cinchona, and in this case the difficulty is not in obtaining a clear filtrate but in the fact that the alkaloids seems to be held back and there may be found the means of overcoming the difficulty in this one case.

In the case of the fluidextract of guarana it was found necessary to cut down the amount of fluidextract used to 5 Cc. and to slightly acidulate the sodium chloride solution, probably to break up the tannin-alkaloid combination.

The use of this process permits assays to be made with surprising speed, as indeed can be understood when one appreciates the fact that only a single and final extraction is required after the precipitation and filtration, which takes not over 10 or 15 minutes from the beginning of the process and that the alkaloid is then ready for gravimetric or volumetric determination or both, as may be desired. The following results have been obtained since the first trial of the process, which was only a few days ago:

	U. S. P Method	Sodium Chloride Method
Fluidextract of Aconite Leaves	0.25	0.24
Fluidextract of Aconite Root	0.44	0.44
Fluidextract of Belladonna Leaves	0.38	0.38
Fluidextract of Belladonna Root	0.52	0.54
Fluidextract of Calabar Bean	0.12	0.13
Fluidextract of Guarana	3.68	3.74
Fluidextract of Ipecac	1.76	1.82
Fluidextract of Cola	0.82	0.83
Fluidextract of nux vomica (total alkaloids)	1.70	1.75

These are sufficiently varied in type to indicate that the process has a wide range of application and its publication without further experiment at this time is deemed advisable in order that it may be tried out by others and thus be made available for pharmacopoeial recognition, if merited.

A method of precipitation and filtration of an aliquot portion for shaking out is already employed with success in the official method of assay of fluidextract of hydrastis where potassium iodide is used as the precipitating agent.

SOME QUERIES ON ALKALOIDAL ASSAY.

W. A. PEARSON.

Much good work has been recently presented on alkaloidal assay, and it is reasonable to expect that much more satisfactory and accurate methods will be inserted in the next Pharmacopoeia of the United States.

There are a few differences of opinion in regard to technique, however, that should be agreed upon before uniformity is to be expected

Query No. 1. Amount of Moisture in Drug.

Crude drugs are not, as a rule, assayed in the exact condition in which they are received. Frequently they must be dried before they can be ground and this loss of water may amount to as much as 30 per cent. Is it advisable to

compute the results obtained to correspond to the original condition of the drug or to the moisture free basis?

Query No. 2. Fineness of Powder.

It is well known that when a powder is ground, all of the particles are not of equal size and that if all the drug is ground and only the particles of a certain size are taken, the sample will not be a representative one.

Would it, therefore, be advisable, instead of stating that the powder should be of a certain fineness, to state that it should be at least of a certain finesness or between certain limits of fineness?

Query No. 3. Temperature.

In certain alkaloidal determinations the temperature plays an important part, in the results obtained. For example, in the assay of opium, the crystalization flask is directed to be set aside in a *moderately cool place.* No limits are given in U. S. P. for "moderately cool" and this temperature has been variously interpreted by different analysts. It is certain that much larger crystals are obtained near 0° C than at slightly higher temperatures; it therefore seems important to ask what influence does temperature have upon the results of an alkaloidal assay?

Query No. 4. Fumes.

Free alkaloids very readily combine with acids, and the analytical laboratory contains fumes of hydrochloric or nitric acids. Before the delicate titration of an alkaloidal residue is made there seems to be danger of these fumes combining with the alkaloid and lowering the results. To what extent does the fumes ordinarily present in the laboratory influence the results of an alkaloidal assay?

Query No. 5. Indicators.

It has been claimed by the analysts in one laboratory that cochineal is the best indicator for all alkaloidal titrations, the men in another laboratory prefer the general use of iodeosin. Does the indiscriminate use of these indicators give concordant results and would the assay be considered as being made according to the U. S. P. if an indicator not specified in the particular assay were used in the titration?

Query No. 6. Color of end point.

In all the alkaloidal titrations, the U. S. P. specifies that the standard solution should be added until a certain color is obtained. Owing to differences in judging the end point and the absence of a definite color standard a considerable variation is to be expected.

Ought not the end point of an alkaloidal titration be determined by matching a certain color of a standard chart under definite conditions?

Query No. 7. Blank determinations.

To avoid the difficulty of judging the color of the end point and to provide a check on the solutions being used a blank determination is usually made by most analysts. Even this method is faulty where the alkaloidal residue still retains some color. Would it be advisable to specify that a blank test be made with every alkaloidal titration?

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.

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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-