

Section on Scientific Papers

Papers Presented at the Fifty-Ninth Convention

A NEW METHOD OF ASSAY FOR ALKALOIDAL FLUIDEXTRACTS.

CHARLES H. LA WALL.

While recently experimenting in the effort to devise a method of assay for fluidextract of colchicum seed I thought of trying the sodium chloride method of salting out objectionable constituents as previously used by me in the method of determining benzoic acid in catsup. (A. J. P., April, 1908, p. 171.)

A number of plans were outlined and tried and a method was soon found, which, when applied to a number of fluidextracts that had been previously assayed by the standard methods, was found to give results practically identical with those previously obtained in each case, with a reduction of about 50% in time, labor and solvents required. The method is as follows:

Dissolve 25 Gm. of sodium chloride in a 100 Cc. graduated, stoppered cylinder, in enough water to make 85 Cc. Add 10 Cc. of the fluidextract to be assayed and then make up the volume to 100 Cc. Agitate well for about one minute. Let stand for five minutes, agitate again and pour on a dry filter. Collect 50 Cc. of filtrate, representing 5 Cc. of fluidextract, and shake out with the proper amounts of the appropriate solvents, as directed for the final extraction of the alkaloid.

It is sometimes, although not always, necessary to return the first portion of the filtrate which comes through cloudy, collecting only clear filtrate for the final extraction.

The sodium chloride, in approximately saturated solution, as in this case, throws out fat, resins, chlorophyll and particularly all substances that are commonly extracted with immiscible solvents such as chloroform and ether.

I have been making alkaloidal assays for a number of years but I have never obtained cleaner final residues than are obtained by this method. The gravimetric and volumetric results check up very closely and the alkaloid is frequently beautifully crystalline.

In such drugs as colchicum seed, physostigma and nux vomica, where there is a large amount of fat present, I have found it necessary and advisable to add 10 Cc. of a 10 per cent. solution of alum in place of part of the water used to dissolve the salt, but I prefer not to use the alum unless necessary, as there is a greater tendency to emulsification when the immiscible solvent is shaken up with the liquid.

The filtrate in most cases is a straw yellow or brownish yellow color, even when very green fluidextracts are used.

The simplicity of the method and its ease of application should recommend it for adoption wherever possible. The only instance among the numerous

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. I. 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