

added, an amorphous yellow precipitate is obtained. If an equal portion of alcohol now be added, the precipitate immediately dissolves.

V. Tincture of iodine, when added to the solution of the sulphate, gives a brown precipitate.

W. If to the solution of the sulphate, some alcoholic potassium hydroxide solution be added, the mixture slowly takes on a pink color, gradually changing into cherry, then red, and, finally, purple.

This test is characteristic of this alkaloid only, and therefore many modifications were tried, until the following was finally determined as being the sharpest and most definite:—

Y. If to the saturated alcoholic solution of the sulphate (1-100) an equal part of alcoholic solution of potassium hydroxide 10% be added, within one minute, the mixture will take on a cherry-red color, which turns darker upon standing. If to this colored mixture, four drops of alcoholic solution of resorcinol (1-100) be added, the color immediately changes to intense dark green. If alcoholic solutions of either hydrochinone, or anthra-quinone (1-100) be used in place of the resorcinol, the same green color will be obtained.

These experiments, reactions and descriptions prove conclusively that there is a third alkaloid in nux vomica, unlike any described in literature under the name igasurine. While studying the alkaloid, much more research work suggested itself to the author, and the results of some of it will be published in a later paper. Experiments will also be made to obtain the physiological reaction of *struxine*.

*Laboratory N. Y. Quinine and Chem. Works.*

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## LABORATORY NOTES.

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In every laboratory where a large variety of work is being done, many observations are made from time to time, which are not of enough importance in themselves to be dignified by making them the subjects for scientific papers; yet they may be of interest and importance to workers in other laboratories. Under the title of "Laboratory Notes," therefore, we have collected a number of such observations of miscellaneous character, hoping that they may prove to be of interest to members of the Scientific Section, and that they may prove to be a step in the direction of encouraging, at our meetings, the mutual exchange of laboratory ideas.

*Criticism of U. S. P. Directions for Making Decinormal Iodine Solution:—*  
The U. S. Pharmacopœia directs, in making N/10 Iodine solution, to

"Dissolve 12.59 gm. pure iodine in a solution of 18 gm. of potassium iodide in 300 cc. water."

This procedure of making the solution, requires from one to three hours. A great saving in time may be effected by cutting down the amount of water to about 40 cc. By this modified method, the solution may be prepared in about 10

minutes, and will be found to be quite as accurate as a solution made by the longer process, as is shown by the following experiments:—

Method	Time Required	Factor of the N/10 Solution
U. S. P. procedure.....	3 hours	.....1.000
Short method, diluted after 10 minutes.....	15 minutes	.....1.001
Short method, allowing to stand 1 hour after iodine is dissolved.....	1¼ hours	.....1.001

*Note on U. S. P. Method of Assaying Solution of Lead Subacetate.*—In this assay, the U. S. Pharmacopœia directs that an aliquot part of the solution be taken for titration of excess of oxalic acid, after allowing the precipitate to settle. To insure freedom from the lead precipitate and to effect a saving of time, filtration of the mixture is advisable. The mixture may be filtered without waiting for the precipitate to settle.

*Note on the U. S. P. Assay of Nux Vomica for Strychnine.*—In the assay for strychnine, the U. S. Pharmacopœia directs the filtration of the final chloroformic extractions containing strychnine, in order to prevent the carrying over of some of the fixed alkali used to liberate the alkaloid. This filtration of the chloroform is not always productive of perfect results, since the filtered solutions sometimes still contain traces of fixed alkali. In four experiments, the extra precaution of washing the united chloroformic extractions with 10 cc. water resulted in reductions of 1.9%, 1.1%, 1.8%, and 2.3% of the total strychnine apparently present.

*Assay of Tincture Ferric Citro-Chloride N. F.*—The National Formulary provides no methods of assay or control for N. F. preparations, each laboratory being obliged to provide its own methods for their control. We have found the U. S. P. method for the assay of Tincture of Ferric Chloride is applicable to this tincture with fairly accurate results. The presence of the citric acid does not materially interfere with the oxidation of the iron to the ferric state nor the subsequent reduction to ferrous iron by the hydriodic acid. Experiments on samples which contained known amounts of iron returned on the average, 98.5% of the iron present, by this method.

One sample of Tincture Ferric Citro-Chloride came under our observation, which contained a large precipitate consisting of a complex double salt of iron and sodium citrate. The precipitate contained about 50% of the iron present.

*Determination of Chromium in Presence of Acacia.*—Organic substances generally interfere with the precipitation by ammonia, of members of the iron group, to which chromium belongs. Chromium sulphate occurs in the market in the form of scales, which, with the assistance of a binder such as acacia, may be readily compressed into tablets. In attempting to assay chromium sulphate tablets containing 10% of acacia, it was found that the chromium could be precipitated quantitatively as hydroxide by ammonia, without interference from the acacia. On the other hand, acacia prevents the precipitation of iron. The possibility of a new method for the separation of iron and chromium, is also suggested by this observation.

*Detection of Nitric Acid in Lime Juice.*—Although nitric acid is a very infrequent adulterant in lime juice, its detection, when present, presents difficulty, since most of the tests for nitric acid are color-reactions, in which concentrated sul-

phuric acid is used, and the latter carbonizes the organic matter present, thus obscuring the color-reaction. The following method, which has no such objection, will readily detect 0.1% of nitric acid in lime juice.

Place 5 cc. of lime juice, 5 cc. of 20% of potassium hydroxide solution, and about 0.5 gm. of granulated aluminum in a test-tube loosely stoppered with cotton. Heat on steam-bath for 10 minutes. No odor of ammonia should be noticeable during that time, indicating absence of nitric acid.

*Interference of Aniline Colors in Alkaloidal Assays.*—We have occasionally found on the market alkaloidal preparations, such as Powdered Extract Stramonium, containing a green aniline dye to simulate the natural color of the leaf. In attempting to assay such extracts we have found that the green color appears in the alkaloidal residue which is to be titrated. Besides obscuring the end-reaction, some of these colors are basic in character and increase the apparent amount of alkaloid present. In one specimen which we examined, the color present increased the apparent amount of alkaloid present to the extent of 24%. The interfering color can usually be eliminated by extracting the acid solution of the alkaloids, with chloroform or other suitable solvent, before making alkaline for the extraction of the alkaloids.

*Occurrence of Guaiac Resin in Scammony Resin.*—Alcoholic solutions of scammony resin, occasionally give a blue coloration with ferric chloride, indicating the presence of guaiac resin. This test, which is given in the U. S. Pharmacopœia under Scammony, should be repeated under Resin of Scammony.

*Physical Instability of Mercurial Ointment.*—Because of the high specific gravity of metallic mercury, mercurial ointments are subject to a settling of the mercury to the bottom of the container. In one instance, a 50% ointment was examined which assayed 75% mercury when sampled from the bottom. It would be well to include in the U. S. Pharmacopœia, directions to "mix well before dispensing."

*Influence of Method of Manufacture on Composition of Compound Solution of Cresol, U. S. P.*—When prepared by the cold process, exactly as prescribed by the U. S. Pharmacopœia, a lot of Liquor Cresolis Comp. assayed 8.5% water and 48.5% phenols by the methods of assay given in Bulletin 107, Bureau of Animal Industry, U. S. Department of Agriculture. When prepared by the more satisfactory method of heating to saponify the linseed oil and dissolving the resultant soap while hot in the cresol, many lots manufactured ranged from 5 to 8.6% of water and from 48 to 50% of phenols.

*Volatility of Caffeine and of Acetanilid in a Current of Steam.*—Having occasion to separate monobromated camphor from a tablet-mixture containing among other ingredients caffeine, the mixture was distilled with steam. Caffeine was found along with the monobromated camphor in the distillate. To determine to what extent the caffeine was volatilized under the conditions employed in the separation of the monobromated camphor from the tablet-mixture, 1 gm. of caffeine was distilled with steam until a 500 cc. distillate was obtained. 0.8% of the caffeine was recovered from the distillate.

Acetanilid is also slightly volatile in a current of steam, and as was expected, the volatility is somewhat greater than that of caffeine. An experiment resulted

in 3% of the acetanilid being found in the distillate, when 2 gm. was distilled with steam until a distillate of 500 cc. was obtained. In a second experiment, using 1 gm. of acetanilid, 2.4% was found in the distillate.

*Determination of Free Phosphorus in Rat Pastes:*—The following method works well with phosphorus rat pastes:—Take a sample of about 1 gm., place in a distilling flask connected with a CO<sub>2</sub> generator and a condenser, and connect the condenser with a 300 cc. Erlenmeyer flask containing 50 cc. of 3% silver nitrate solution. Connect the flask with a series of two U tubes containing 3% silver nitrate solution. All connections exposed to the phosphorus must be of glass or of cork covered with plaster of paris. Pass CO<sub>2</sub> through the apparatus for 20 minutes, testing for leaks with flexible collodion, which will bubble at a leak. Place 125 cc. of cold, freshly boiled, distilled water containing 2 cc. of sulphuric acid in the flask containing the sample by means of the tube which leads to the CO<sub>2</sub> generator. Continue to pass in CO<sub>2</sub>, and heat the flask gently until, after about three hours, practically all of the liquid in the flask has been distilled into the silver nitrate solution.

Finally, allow the condenser to become hot from the distillation, and disconnect between generator and distillation flask before removing flame. Collect all the silver nitrate solutions in the Erlenmeyer flask, using nitric acid to dissolve the black precipitate in the U tubes. Add 15 cc. of nitric acid to the mixture, boil for five minutes, and add hydrochloric acid, in moderate excess, to precipitate the silver. Boil for 20 minutes, let cool, filter, and concentrate to 150 cc. Cool to 40° C., add 100 cc. ammonium molybdate solution, stir well, and let stand in warm place over night. Filter off clear liquid, wash precipitate by decantation, using 25 cc. water for each washing, transfer to filter, and wash until two fillings of the funnel are rendered pink by one drop of N/2 potassium hydroxide solution, using phenolphthalein as indicator. Place the filter and precipitate, in a glass-stoppered flask, add an excess of N/2 potassium hydroxide solution, shake for a few minutes, add phenolphthalein indicator, and titrate back with N/2 sulphuric acid. Each cc. of N/2 potassium hydroxide solution, is equivalent to 0.0007071 gm. phosphorus.

This method is also applicable to the assay of phosphoretted resin and phosphorus tablets, for free phosphorus. Three samples of commercial phosphorus rat-pastes, were found to contain respectively 57.9%, 41%, and 82%, of the claimed amounts of phosphorus in free condition.

*Note on Charging Melting-Point Tubes:*—The usual method of taking melting-points involves the use of a capillary melting-point tube, sealed at one end. This type of tube presents the difficulty of requiring the shaking down of the sample, into the closed end of the tube. A capillary tube open at both ends is far more convenient. It may be charged with the sample, to the depth of about one-fourth inch, merely by pressing the end of the tube into the powdered substance in the manner of filling capsules. The tube may then be attached to the thermometer in the usual manner, and used the same as a tube closed at one end. In all our experience with this open capillary tube, the melted sample has always been retained within the tube by capillary attraction. This type of tube permits of the employment, if desired, of a finer capillary tube, as the sample does not

have to be shaken down. It is unnecessary to say, of course, that these tubes can only be used in a melting-point apparatus provided with an air chamber, such for example, as Roth's, they could not be submerged in a water or acid bath.

*Lead Storage-Tanks for Pharmaceutical Preparations:*—In investigating the suitability of lead-lined storage-tanks for pharmaceutical preparations, the following observations were made:—Tincture of Opium, and Syrup of White Pine Compound, dissolve lead; Tincture of Aconite and Fluid Extract of Ergot, do not permanently dissolve lead, but form precipitates containing lead; Tincture of Vanilla and Aromatic Fluid Extract of Cascara Sagrada, dissolve lead and also form precipitates containing lead.

*Note on Soft Gambir:*—The U. S. P. requires that gambir be friable, that it contain not less than 70% alcohol-soluble matter and not more than 5% ash. Gambir, when soft, contains excessive water, which is included as alcohol-soluble matter. If the amount of soft gambir used in a formula, is corrected for its excessive water, soft gambir may be used, in place of the friable gambir required by the U. S. P. The point to be considered is the maximum amount of water which gambir may contain and retain its friable condition.

In order to determine this point, a flat cake of soft gambir was alternately heated at 100° C. for fifteen minutes, and then cooled, and the condition of the gambir ascertained when cool. Under these conditions, the gambir became friable when it contained 5.3% of water. This figure, however, was not consistent with the moisture-content of spontaneously-dried, commercial, friable samples, which contained as much as 12% moisture. It is evident, therefore, that the maximum content of water consistent with a friable condition, is variable according to the method of drying. The commercial method of drying gambir, is the spontaneous one, so that the determination of the maximum content of water consistent with a friable condition, should be based on spontaneously-dried commercial samples. The maximum moisture in the samples of friable gambir, which we have examined up to the present, is 12%. The minimum moisture in the samples of soft gambir which we have examined up to the present, is 21%. The proper maximum-content of water in gambir, consistent with a friable condition, to adopt as a standard, should be that which we have found in practice and which is 12%. In valuing soft gambir, the moisture, alcohol-solubility, and ash should be determined and only 12% of the moisture should be calculated as alcohol-soluble matter.

As an illustration, we will consider a soft gambir which assays 25.4% moisture and 74.2% alcohol-soluble matter. The moisture in excess of the 12%, consistent with a friable condition, is 13.4% which should be subtracted from 74.2% alcohol-soluble matter, leaving 60.8% alcohol-soluble matter, exclusive of excessive water for the gambir, instead of the 70% which the U. S. P. requires, thus indicating that 116 parts are equivalent to 100 parts of U. S. P. gambir.

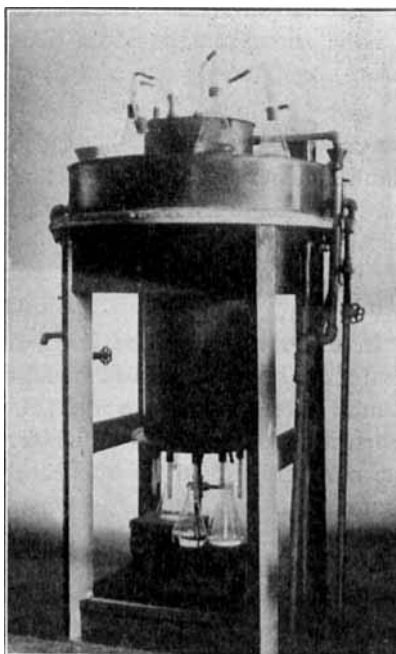
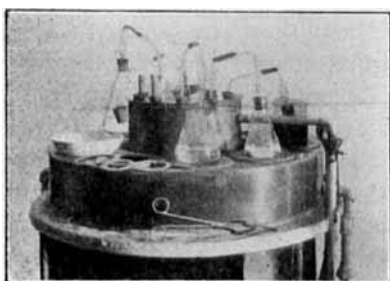
Gambir labeled as catechu, is always friable and for catechu and friable gambir, this method of calculating is not called for. These should assay not less than 70% alcohol-soluble matter and not more than 5% ash. There may be some slight objection to the use of soft gambir on the score of its diluting the menstruum and therefore changing the character of the extractive a trifle. But this

is not likely, as the chief medicinal ingredient is tannic acid and tannic acid is quite soluble in both water and alcohol.

*An Improved Form of Steam Bath:*—A circular steam bath containing the usual steam coil, constant water-level, openings with accommodating-rings for various-sized apparatus, etc., and with the additional arrangement of having an upright condenser placed in the center of the bath, has many advantages over the usual rectangular-shaped steam-bath without condenser.

The circular steam-bath requires no special explanation, but the upright condenser may be described as follows :

The condenser consists of two cylinders, one within the other, with glass tubes for condensing purposes placed in the space between the two cylinders. These cylinders contain the water used for condensing purposes,—the water entering the inner cylinder at the top, passing through holes in the bottom of the cylinder in to space between the two cylinders,



where it comes in contact with the condensing tubes, and then, passing up through the outer cylinder to the overflow near the top. The inner cylinder is designed for use with ice, when weather conditions require its use.

Flasks containing light solvents, are connected, for the process of distillation, with small spray traps, by means of corks which have been extracted with the kind of solvent undergoing distillation. The spray traps, in turn, are connected with the condensing tubes by means of short pieces of rubber tubing. To prevent loss of substances due to sudden boiling, it is desirable that the inner bottoms of the flasks be scratched with a diamond, taking care not to cut deeply into the glass.

This type of steam-bath has the advantages that it is always ready for the recovery of small quantities of volatile solvents, without the necessity for setting up distillation-apparatus; cold condensing can be conveniently carried out; the apparatus is compact; breakage, such as is common with glass condensers of the ordinary unprotected type, is prevented; the recovery of light solvents, which can be repeatedly employed for the same class of work is encouraged; and finally it may be used as an ordinary steam-bath, when the condensing arrangements are not required.

Cuts of the apparatus accompany this description.

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