

Ammonia fumes must be kept away from the acid solution of the alkaloids while evaporating to dryness.

The dry residue in the dish is taken up with a small amount of water, washed into a 100-cc. flask or cylinder, brought up to mark, mixed and 50 cc. of this transferred to a titrating vessel; a wide mouth bottle with a clear bottom is very good to see the color changes. A few drops of methyl red indicator are added and then the liquid is titrated to a pure yellow with $N/50$ sodium hydrate, and cc. used noted. A white surface placed under the bottle shows the color changes very well.

The other 50 cc. of liquid is transferred to another titrating vessel, then 50 cc. of 95% neutral alcohol added, then some phenolphthalein indicator, and titrated with the $N/50$ soda to a faint pink which remains so for at least one-fourth minute, and number of cc. noted. The error by measuring this way is practically negligible for this case.

Then make a blank test, using 50 cc. of same alcohol with 50 cc. of water and phenolphthalein indicator and note the number of cc. of $N/50$ required to obtain same slight pink.

From the number of cc. of second titration subtract the number of cc. for the blank test, then from this subtract twice the number of cc. used in the first titration, and this difference multiplied by two, should give the amount of $N/50$ for the strychnine.

To reiterate, it is important that the hot acid solution, wherein the tartrate is precipitated, is made sufficiently acid again while hot after the addition of the Rochelle salt, but not acid enough so as to dissolve too much of the precipitate. If not enough free acid present, some strychnine, or many other alkaloids, if present, will be carried down with the tartrate, about 0.020 Gm. more or less of strychnine when about 2 Gm. of quinine is precipitated, the amount of liquid not having such a large influence.

ASSAY METHOD FOR THE DETERMINATION OF AN ALKALOID OR TOTAL ALKALOIDS IN COATED OR UNCOATED TABLETS.*

BY LESTER C. DICK.

Coated tablets, except sugar-coated, white, are placed in a beaker and the interfering color removed by washing with a solvent.

The tablets are dried at 100° C. and fifty or more are weighed, the average weight noted and the tablets reduced to a fine powder.

Uncoated tablets are directly weighed and reduced to a fine powder. An aliquot part is taken—in either case representing about two grains of alkaloidal salt. The powder is transferred to a Florence flask of 300 cc. capacity, fitted with a tight stopper.

Ammonium hydroxide or sodium hydroxide T. S. is now added in quantity sufficient to liberate the alkaloid. A number of buckshot, depending upon the quantity of powder taken, are now added in amount necessary to subdivide the moist mass. Exactly 100 cc. of chloroform is added, the flask stoppered and rotated, thus coating the sides of flask with the moist powder.

* Scientific Section, A. Ph. A., Toronto meeting, 1932. No discussion.

Rotation of the flask at frequent intervals causes the buckshot to coat and recoat the sides of the flask with a thin layer of the moist powder, insuring complete extraction of the alkaloid or alkaloids by the chloroform. After carrying out the above procedure for several hours the chloroform is poured through a small filter paper and an aliquot part of 50 cc. collected.

The aliquot portion referred to is carefully transferred to a separatory funnel and extracted with dilute acid. The dilute acid is in turn made alkaline with ammonium or sodium hydroxide T. S. and extracted with chloroform. A test should be made in each case to insure complete extraction of the alkaloid. The final chloroform extractive is evaporated and the alkaloids determined either volumetrically or gravimetrically.

The weight of the alkaloid obtained multiplied by 2 gives the amount in the total sample used. The weight of the total sample used divided by the predetermined weight of one tablet gives the number of tablets containing the determined quantity of alkaloid or alkaloidal salt.

In many cases tablets containing no other chloroform-soluble material, the 50-cc. aliquot portion can be evaporated to dryness, the residue dissolved in 10 cc. of neutral dilute alcohol and directly titrated, using methyl red as the indicator, diluting the alcoholic solution to 100 cc. with distilled water after sufficient volumetric acid has been added to give a pink tint.

The following results were obtained in coated tablets by this method:

| Tablet. | Theoretical Strychnine Sulphate Content. | Results. | Percentage. |
|---------|---|---------------|-------------|
| A— | 0.00216 Gm. | 0.002088 Gm. | 96.71 |
| B— | 0.00162 Gm. | 0.001649 Gm. | 101.25 |
| C— | 0.00216 Gm. | 0.0021297 Gm. | 98.60 |
| D— | 0.00108 Gm. | 0.001025 Gm. | 95.82 |
| E— | 0.00108 Gm. | 0.001025 Gm. | 95.82 |
| F— | 0.00108 Gm. | 0.001022 Gm. | 94.59 |

The theoretical Strychnine Sulphate contents above were obtained from the labeled quantity and do not make allowance for possible errors in manufacture although this in each instance was done under rigid control.

Any other solvent where chloroform is not indicated may be used with equally consistent results.

CONTROL LABORATORY,
G. S. STODDARD & Co., INC.

SUGGESTED ASSAYS FOR SOME N. F. PREPARATIONS.*

STRONG RESORCINOL PASTE. MILD RESORCINOL PASTE.

BY WILLIAM B. BAKER.

The efficacy of the official resorcinol pastes as therapeutic agents is due mainly to the resorcinol and zinc oxide which they contain. It is, therefore, essential that standards be adopted fixing definitely the content limits of these ingredients, and that practical methods be developed for the quantitative determination of the

* From the laboratory of A. G. DuMez, Professor of Pharmacy, School of Pharmacy, University of Maryland.