water, except in the case of lead, where a strong KI solution was used, and the strips were given a final wash with alcohol before drying at 100° C., and reweighing.

In the second experiment the lower portion of an ordinary 8-inch desiccator was filled with resublimed iodine and two cc. of water added. A second set of clean, weighed metal strips were placed in the upper portion of the desiccator on a rack made of small glass rod. The cover was placed on the desiccator, which was then put in the steam closet at 65–70° C. for three days. The strips were then freed of iodine and iodides as in the first experiment and reweighed.

Following is a tabulation of the results:

TABLE I.—MOLTEN IODINE.

	Surface exposed, sq. cm.	Loss in Gm.	Loss in mg. per sq. cm.
Lead	35.1	6.1780	176.0
Carpenter steel	16.9	0.0078	0.46
Ascoloy	34.6	0.1650	4.77
Super ascoloy	26.6	0.0173	0.65
Allegheny metal	28.8	0.0429	1.49
Duriron	35.5	6.1651	173.7

TABLE II.—MOIST IODINE VAPOR.

	Surface exposed, sq. cm.	Loss in Gm.	Loss in mg. per sq. cm.
Lead	33.1	0.0224	0.68
Carpenter steel	17.0	0.2326	13.68
Ascoloy	33.9	0.4092	12.07
Super ascoloy	27.3	0.4385	16.07
Allegheny metal	32.4	0.5447	16.82
Resistol	220.3	2.687	12.20

RESEARCH DEPARTMENT OF THE CHEMICAL AND PHARMACEUTICAL LABORATORIES,
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A METHOD FOR THE SEPARATION AND DETERMINATION OF TOTAL ALKALOIDS AND PHENOLPHTHALEIN IN PILLS.

BY R. L. TAYLOR.*

Place a number of pills equivalent to one grain of alkaloids in a lipped-centrifuge tube. Add 15 cc. of 2% sulphuric acid, and shake until the pills are completely disintegrated. Place the tube in the centrifuge and rotate about five minutes. Remove the tube and decant the clear liquid through filter paper.

Add 5 cc. of the 2% sulphuric acid to the tube and again shake until the residue is thoroughly disintegrated. Rotate the tube in the centrifuge and decant the liquid through the filter as before. Repeat this extraction several times, using 5-cc. portions of the dilute acid, until 2 or 3 cc. of the last portion of dilute acid gives a negative test for alkaloids with Mayer's reagent.

After the alkaloids have been completely extracted from the mixture, add about two Gm. of purified sawdust to the tube and mix well. Filter the contents of the tube through the filter, and rinse the tube with successive small portions of water, filtering the rinsings through the filter containing the residue. Dry the filter paper and residue in the oven at 60° C.

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DETERMINATION OF ALKALOIDS.

Place the combined filtrates in a separatory funnel, and make alkaline with ammonia water. Completely extract the alkaloids from the aqueous solution by shaking with successive portions of chloroform, filtering each portion as drawn off. Completely extract the alkaloids from the combined chloroform portions by shaking out with successive portions of 2% sulphuric acid. Make the combined acid portions alkaline with ammonia water, and completely extract the alkaloids with successive portions of chloroform, filtering each portion as drawn off. Evaporate the chloroform solution to dryness on the steam-bath. Add a few cc. of neutral alcohol to the residue, and heat gently until the residue is in solution. Add 10 cc. of $N/10$ sulphuric acid, and titrate the excess acid with $N/50$ potassium hydroxide, using methyl red indicator. Multiply the number of cc. of acid consumed by the alkaloid, by the factor representing the alkaloids and their respective quantities present. The experimental samples contained strychnine sulphate and extract of belladonna and were calculated accordingly.

DETERMINATION OF PHENOLPHTHALEIN.

Fold the dried filter paper containing the residue from the pills and place in a paper extraction thimble. Place the thimble in a Soxhlet extractor and extract with alcohol for several hours, or until the phenolphthalein is completely extracted.

Disconnect the apparatus and transfer the alcohol solution of phenolphthalein to a 200-cc. volumetric flask. Dilute the solution with alcohol to the 200-cc. mark.

Pipette 10 cc. of this solution to a 100-cc. beaker and evaporate to dryness. Precipitate the phenolphthalein as tetraiodophenolphthalein, as recommended by Samuel Palkin.[1]

Filter the precipitate on a tared Gooch crucible. Wash the precipitate of tetraiodophenolphthalein, with successive portions of alcohol which has been saturated with tetraiodophenolphthalein, to remove the precipitated resins. Dry to constant weight at 100° C. and weigh. Multiply the weight of tetraiodophenolphthalein by 0.3871 to obtain the weight of phenolphthalein.

Pill masses containing the following ingredients were made and assayed and the following results obtained:

Ingredients.

Aloin	1.2906 Gm.
Strychnine sulphate	0.0648
Extract of belladonna	0.4315
Podophyllin	0.8640
Phenolphthalein	2.5920

EXPERIMENTAL DATA.

Sample.	Total Alkaloids. Present.	Recovery.	Error.	Phenolphthalein. Present.	Recovery.	Error.
1	0.0702 Gm.	0.0689 Gm.	1.8%	2.5920 Gm.	2.5725 Gm.	0.75%
2	0.0702 Gm.	0.0692 Gm.	1.4%	2.5920 Gm.	2.5645 Gm.	1.0%
3	0.0702 Gm.	0.0689 Gm.	1.8%	2.5920 Gm.	2.5625 Gm.	1.1%
4	0.0702 Gm.	0.0693 Gm.	1.2%	2.5920 Gm.	2.6000 Gm.	0.3%

[1] *J. Assocn. Official Agr. Chem.*, 8 (1924), 30–36.