

D	In distilled water	Plain quartz tube	Same as Test No. 5	Same as Test No. 5
D-1	In isotonic solution	Plain quartz tube	Same as Test No. 6	Same as Test No. 6
E	In distilled water	Red quartz tube	Same as Test No. 5	Same as Test No. 5
E-1	In isotonic solution	Red quartz tube	Same as Test No. 6	Same as Test No. 6
F	In distilled water	Green quartz tube	Same as Test No. 5	Same as Test No. 5
F-1	In isotonic solution	Green quartz tube	Same as Test No. 6	Same as Test No. 6
G	In distilled water	Purple quartz tube	Same as Test No. 5	Same as Test No. 5
G-1	In isotonic solution	Purple quartz tube	Same as Test No. 6	Same as Test No. 6
H	In distilled water	Pyrex tube	Same as Test No. 5	Same as Test No. 5
H-1	In isotonic solution	Pyrex tube	Same as Test No. 6	Same as Test No. 6

These results indicate that exposure to ultraviolet radiation in the various types of containers employed has no deleterious influence upon the mydriatic action of solution of homatropine hydrobromide.

CONCLUSIONS.

(1) Certain physical measurements of solution of homatropine hydrobromide have been made.

(2) Sterilization or exposure to ultraviolet radiation has no apparent deleterious effect upon the mydriatic action of homatropine hydrobromide in either distilled water in sodium chloride solution isotonic with the tear.

(3) Homatropine hydrobromide in distilled water or isotonic solution preserved in ampuls shows no apparent loss in mydriatic action during a period of nine months.

RESEARCH LABORATORIES,
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MERCURIC IODIDE DETERMINATION IN TABLETS.*

BY H. O. MORAW.

Mercuric iodide as a medicinal agent is available in the U. S. P. form, in tablet triturates of one-eighth or one-fourth grain, in potassium iodide oil suspension in ampuls and in ointments. From the standpoint of pharmaceutical manufacturing as well as the Government regulatory work, tablets and ampuls are probably the more important forms of dispensing this drug. Since, in the case of tablets the usual builders, disintegrators and lubricants, such as sugar, starch and talc, might interfere in the electrolytic determination which is specified for the assay of the U. S. P. product, it is necessary to adapt a method to the assay in the presence of interfering substances. This has been done by Bender¹ who effected solution of interfering substances other than talc by free chlorine treatment and subsequent estimation as mercuric sulphide. Morgan² has adapted this method to calomel tablets and suggests it for other mercurials. Since the electrolytic apparatus is not available in the average laboratory, consideration was given to the practicability of other methods employing the usual laboratory equipment. The time

* Work performed while employed in the U. S. Food & Drug Laboratory, 1625 Transportation Bldg., Chicago, Ill.

¹ *Ind. & Eng. Chem.*, 6, No. 9 (1914), 753.

² *J. Assocn. Official Agr. Chem.*, 10 (1927), 367-9; cf. Spencer, *C. A.*, 20, 3210.

required by the sulphide method as well as the toxicity of hydrogen sulphide gas, are other reasons for using other methods if available. Sandilands'¹ method based on dissolving the mercuric iodide in sodium thiosulphate and completing the estimation either as mercury sulphide or after reduction with zinc, applying the Volhard method to the estimation of the iodine, has not been applied to tablets but should be considered as an alternative method if its adaptation can be effected.

The method reported herein is sound in principle, sufficiently accurate for routine regulatory work, and rapid. Common laboratory apparatus and reagents are required.

EFFECTING COMPLETE REDUCTION OF MERCURIC IODIDE.

The chief difficulty in assaying mercuric iodide arises from its insolubility in suitable solvents. It is slightly soluble in fixed alkalies, undergoing partial decomposition with the formation of a soluble iodide and mercury and a complex containing $\text{HgI} \cdot 3\text{HgO}$ and HgO . When treated with both a fixed alkali and reducing agent, such as lactose or formaldehyde, there is formed metallic mercury in addition to the above.² This incomplete reduction was found by the author to be due to occlusion and incomplete peptization. A coating of mercuric oxide formed around the occluded particles.

This suggested mixing the lactose intimately with the mercuric iodide, then treating the mixture with just sufficient of the alkali solution to make a smooth paste by stirring with a rod. The remainder of the alkali is then added. Thus by proper manipulation, complete reduction was found to take place and a soluble iodide obtained which could be separated from insoluble material and converted to silver iodide.

Results are given in Table I on pure mercuric iodide by this procedure alone and on tablet triturates by this procedure in comparison with the mercuric sulphide method.

TABLE I.—RESULTS ON MERCURIC IODIDE AND ON TABLETS OF UNKNOWN COMPOSITION.

Pure mercuric iodide.		Manufacturer.	Tablet triturates.		Per cent of declared HgI_2 found.	Method used.
Grams.	Per cent.		No. of tablets used.	Grainage.		
0.2	99.7	A	18.39	$\frac{1}{8}$	85.2	AgI
		A	18.39	$\frac{1}{8}$	87.1	AgI
0.2	99.4	A	24.525	$\frac{1}{8}$	87.8	AgI
		A	24.525	$\frac{1}{8}$	86.9	AgI
0.2	100.6	B	24	$\frac{1}{8}$	90.99	AgI
		B	20	$\frac{1}{8}$	90.0	HgS
0.3	100.1	C	10	$\frac{1}{4}$	94.07	AgI
		C	10	$\frac{1}{4}$	94.8	AgI
		C	20	$\frac{1}{4}$	97.10	HgS

Determination.

Transfer to a 250-cc. beaker an accurately weighed quantity of the finely powdered sample equivalent to 0.25 to 0.4 Gm. of mercuric iodide. Add about 0.2 Gm. of lactose, mix well with a glass rod. Make a smooth paste of this mixture by treating it with 2 to 5 cc. of 10% sodium hydroxide solution. Add an additional quantity of the sodium hydroxide solution, suffi-

¹ "Note on Preliminary Estimation of HgI_2 ," by James Sandilands, *C. A.*, 21, 1076; *Pharm. J.*, 116, 357-8, 368.

² National Standard Dispensatory.

cient to make a total of 30 cc. Dilute with water to about 75 cc. Digest gently over a low flame for 30 to 50 minutes. Filter the hot solution into a 400-cc. beaker. Wash the filter free from iodides. Add a slight excess of silver nitrate solution (5 cc. of 10%) and stir. Add 25 cc. of concentrated nitric acid, stir, cover the beaker with a watch glass and boil gently till the liquid becomes clear. Filter on a tared gooch crucible, wash with water. If chlorides are present wash with ammonia water. Dry at 120° to 130° C. and weigh. Silver iodide $\times 0.9677$ equals mercuric iodide.

CONCLUSIONS.

1. The incomplete reduction of mercuric iodide in alkaline solution with lactose as a reducing agent is due to incomplete peptization and occlusion.
2. Thorough mixing of the lactose in a dry condition together with proper manipulation in moistening it with the alkali solution prepares the mercury compound for complete reduction.
3. The iodine is completely changed to a soluble iodide which can be separated and determined as silver iodide.
4. A method is submitted, the procedure of which is simple and rapid, and the principles are sound. Apparatus common to every laboratory may be used.
5. The limits of accuracy are within one per cent which is satisfactory for control and regulatory purposes.

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THE TOXICITY OF THALLIUM SULPHATE.*

BY JAMES C. MUNCH.¹

The introduction in 1920 of proprietary rat poisons containing thallium compounds as the toxic ingredients directed attention to the toxicity of these products. The literature upon thallium is meager, so this investigation was undertaken to obtain definite knowledge of the toxicity of thallium compounds.

Thallium occurs in various ores which usually also contain sulphur. The chemical symbol is Tl; the atomic weight is 204.0. It forms two series of white, water-soluble salts: thallos, in which the metal is monovalent; and thallic, in which it is trivalent. The monovalent compounds are considered to be more stable and are used in pharmaceutical practice. The acetate ($\text{CH}_3\text{COO-Tl}$) has been used as a depilatory, and to check the night sweats in phthisis: the sulphate, Tl_2SO_4 , is most commonly employed in the preparation of rat poisons.

Thallium was discovered by Crookes in 1863 by the characteristic green line in the spectrum (19, 20). Olmer and Tian (40) claim that the limit of detection with a spectroscope is 1:500,000,000 (2 micrograms per liter). It may be determined quantitatively by fusion with sodium carbonate, acidification with hydrochloric acid, adding an excess of potassium chlorate and boiling to remove excess

* Scientific Section, A. P. H. A., Portland meeting, 1928.

¹ Pharmacologist, Division of Economic Investigations, Bureau of Biological Survey, United States Department of Agriculture, Washington, D. C. Feeding tests were conducted in the Pharmacology Laboratory, F. D. I. Administration, U. S. Department of Agriculture.