DETERMINATION OF ARSENIC IN BISMUTH SALTS.*

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Results obtained in the testing of bismuth salts for arsenic by the U. S. P. XI procedure suggested that this modification of the Gutzeit method, at least in our hands, tends to give low results on these salts or fails to disclose their full arsenic content. For a study of this question bismuth subcarbonate was chosen and a number of manufactured batches were tested "as is" and with added known amounts of arsenious oxide. The sample used was, of course, the specified 0.2 Gm. and the amount of arsenious oxide added was 0.002 mg. (10 p. p. m.) so as to find out whether when a quantity of arsenic approximating what the U. S. P. permits is added the test will disclose the full amount. The reagents used in the work were shown by test to be arsenic free and in estimating the amounts of arsenic the stains produced on the mercuric bromide test papers were compared with those obtained with known amounts of arsenious oxide in the absence of bismuth using the U. S. P XI method.

Eight bismuth subcarbonates were used. Seven of them when tested alone failed to give any stain; the other one gave a stain corresponding to less than 0.001 mg. of arsenious oxide—less than 5 p. p. m. in the 0.2-Gm. sample. Samples of these same subcarbonates were then tested with 0.002 mg. of added arsenious oxide—10 p. p. m. in the 0.2-Gm. sample. Of the seven giving no stain when tested alone one still failed to give any stain, indicating that the added arsenic was not being detected and the other six gave stains corresponding to 0.001 mg. arsenious oxide—half the amount added. The eighth sample—the one which when tested alone showed some arsenic but less than 0.001 mg. of arsenious oxide per 0.2 Gm. sample—now gave a stain corresponding to 0.002 mg. of arsenious oxide (10 p. p. m.) whereas a higher result (more than 0.002 and less than 0.003 mg. or 10–15 p. p. m.) should have been obtained. The tests on two of the eight samples—both alone and with added arsenic—were repeated and the results were the same. These data show that the test, as we have done it, does not disclose the full arsenic content of bismuth subcarbonate. Of course the amounts being worked with are small but the U. S. P. XI directs that a small (0.2 Gm.) sample be used and permits only a small amount (10 p. p. m.) of arsenic so a test, in order to be satisfactory must be sensitive enough to detect and differentiate between amounts which are less than 10 p. p. m. The fault does not reside in the size of the sample used for the test; larger amounts of bismuth subcarbonate and reagents were used but the results were not improved.

The U. S. P. X method, also a modification of the Getzeit test, gave somewhat better results. It differs from the U. S. P. XI method in using a larger amount of zinc; in using dilute sulfuric acid instead of the stannous chloride acid test solution of the U. S. P. XI which is essentially hydrochloric acid, and in not using potassium iodide in the reaction in the generator bottle. Here the standard stains were of course made using the U. S. P. X method. These tests were done using those seven of the eight bismuth subcarbonates which when tested alone using the U. S. P. XI

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method gave no stain. In order that the findings may be compared with those obtained in the U. S. P. XI test, they are given in tabular form, together with those reported above—Table I.

TABLE I.

	U. S. P. XI.		U. S. P. X.	
Sample	"As is"	Plus 0.002 mg. As_2O_3 per 0.2 Gm. sample. (10 p. p. m.)	"As is"	Plus 0.002 mg. As_2O_3 per 0.2 Gm. sample. (10 p. p. m.)
681	No stain	No stain	No stain	Stain equivalent to 0.002 mg. As_2O_3
682	No stain	Stain equivalent to 0.001 mg. As_2O_3	No stain	Poor stain.
	No stain	Stain equivalent to 0.001 mg. As_2O_3	No stain	Stain equivalent to about 0.002 mg. As_2O_3
381	No stain	Stain equivalent to 0.001 mg. As_2O_3	No stain	Poor stain.
	No stain	Stain equivalent to 0.001 mg. As_2O_3	No stain	Stain equivalent to about 0.002 mg. As_2O_3
	No stain	Stain equivalent to 0.001 mg. As_2O_3	No stain*	Poor stain.*
	No stain	Stain equivalent to 0.001 mg. As_2O_3	No stain*	Stain equivalent to about 0.002 mg. As_2O_3*
991	No stain	Stain equivalent to 0.001 mg. As_2O_3	No stain	Stain equivalent to 0.002 mg. As_2O_3*
			No stain*	Stain equivalent to 0.002 mg. As_2O_3*
300	No stain	Stain equivalent to 0.001 mg. As_2O_3	No stain*	Stain equivalent to about 0.002 mg. As_2O_3*
			No stain*	Stain equivalent to about 0.002 mg. As_2O_3
359	No stain	Stain equivalent to 0.001 mg. As_2O_3	No stain*	Stain equivalent to 0.002 mg. As_2O_3*
			No stain*	Stain equivalent to 0.001 mg. As_2O_3*
158	No stain	Stain equivalent to 0.001 mg. As_2O_3	No stain*	Stain equivalent to 0.001 mg. As_2O_3*
			No stain*	Stain equivalent to 0.002 mg. As_2O_3*

* Because of the too vigorous reaction, the stannous chloride test solution of the U. S. P. X was omitted.

Although the U. S. P. X modification of the Gutzeit test did give better results than the U. S. P. XI modification, it is not entirely satisfactory. Thus, even after omission of the stannous chloride test solution the reaction was probably still too vigorous, and in some tests the full amount of arsenic known to be present was not found and the stains produced were not as concentrated and definite as they should be. The subject is, therefore, in need of further study, and the development of a new test or a new modification of the Gutzeit test which will regularly give accurate results regarding the arsenic content of bismuth salts should be undertaken.