

Table III.—Preparations Made Using Bioassayed Natural Materials

Preparation Tested	No. of Samples Tested	Fluorophotometric Assay			Calculated Potency ^a
		Minimum	Maximum	Average	
A. Uni-vitamin products					
Yeast tablets	8	52.5	68.4	60.6	60 γ /tab.
Yeast tablets	33	47.4	79.2	58.5	63 γ /tab.
Malted wheat germ extract cpd	17	6.12	9.3	6.96	6.4 γ /Gm.
B. Multi-vitamin products					
Yeast-fish liver oil tablets	6	132	255	181.5	172.5 γ /tab.
Paste in capsules, Type I	18	93	119	105	114 γ /cap.
Paste in capsules, Type II	9	702	864	774	798 γ /cap.
Paste in capsules, Type III	4	798	933	879	900 γ /cap.
Paste in capsules, Type IV	10	95	130	111	104 γ /cap.

^a Calculated from the bioassay on the ingredients.

tures and finished products. For convenience of presentation the materials tested have been divided into three types and the data on each are shown in a separate table. (See Tables I, II and III.) These tabulated data require little comment. As would be expected there are variations in the results obtained by the fluorophotometric assay and there are differences between them and those obtained by bioassay or by calculation from potency data on the B₁ components of the products tested. However, taken as a whole findings are good, and it does appear that this fluorophotometric method is of practical value in testing for vitamin B₁.

One of the most important phases of this kind of work is the preparation of the sample for test. That is, of course, an easy matter when one is dealing with thiamin chloride itself or the simple forms of it such as solutions and tablets but becomes a very difficult matter when one reaches the complex multi-vitamin preparations such as the various pastes in capsules—obviously there are products representing practically the entire range from those easy to handle to the difficult ones. Inasmuch as each product or mixture is a problem in itself and the preparation for analysis has to be adjusted in accordance with the character and composition of the material to be tested and inasmuch as it is not the purpose of this paper to take up that phase of the work details on how to go about assaying, each type of sample will not be given. The point is that even though the complexity of the mixture does become high the assay can be run and results of the proper order are obtained.

Table III, Section B, relates to multi-vitamin preparations in which the fluorophotometric data were compared with figures calculated from the bioassay data as components of the mixtures instead of with the findings in bioassays on the mixture itself. For this reason and because of the difficulties associated with the assaying of these complex mixtures (see above), it is worth while to include a comparison of the fluorophotometric assay with the bioassay of the finished complex mixture. In a multivitamin paste in capsules the fluorophotometric assay showed 108 gamma of B₁ per capsule as compared with 99 by bioassay. In a syrupy emulsion of fish liver oil and other fats containing the B complex, carbohydrates, proteins,

minerals and flavoring materials, the two samples gave the following results:

	Fluorophotometric	Biological
A	423 γ /fl. oz.	450 γ /fl. oz.
B	540	465

Note that when the proper method of handling mixtures like these two have been satisfactorily worked out and skill in the technique has been developed close agreement with bioassay can be had.

A Method for the Determination of Mercury in Complex Ions*

By John T. Read and Roger F. Maize†

The determination of mercury in complex anions such as that occurring in potassium mercuric iodide presents a problem for which ordinary methods of analysis and available literature fail to give a satisfactory solution, the definite problem at hand being the estimation of mercury in germicidal tablets containing mercuric iodide, potassium iodide, sodium bicarbonate and an organic dye. The major difficulty consists of splitting the complex ion containing the mercury and converting the latter into a form in which it can be readily estimated.

METHOD DEVELOPED

The details of the method as finally developed are as follows: Weigh accurately an amount of the sample estimated to contain about 0.30 Gm. mercury and transfer to a 500-cc. Kjeldahl flask.

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Wash down into the flask with 25 cc. of hydrochloric acid and 50 cc. of water, then add 8 Gm. of zinc (C. P. 10 mesh). Shake the flask until the amalgam which adheres together has been broken up into a granular form. Add an additional 2 Gm. of zinc and heat on a steam bath, shaking frequently until the solution becomes clear and colorless. Remove about 2 cc. of the supernatant liquid and saturate it with hydrogen sulfide; it should remain colorless. If, however, a brownish tinge develops (indicating the presence of mercuric salts), continue the digestion until the solution shows no reaction with hydrogen sulfide.

When all the mercury is reduced, wash the amalgam by decantation until the washings no longer show a reaction with silver nitrate solution. In the event that fragments of the amalgam are floating on the surface of the liquid, add a few cc. of alcohol to break the surface tension.

The amalgam is now dissolved by treating with a mixture of 25 cc. concentrated nitric acid and 100 cc. of water. If the reaction becomes too violent, the flask should be cooled by immersing in cold water. When the zinc has been dissolved out of the amalgam and the reaction has moderated, add 20 cc. more of concentrated nitric acid and heat on the steam bath until all the mercury is in solution; then add 20 cc. more of concentrated nitric acid and heat for 30 minutes on a steam bath. The solution should at this time be clear and colorless.

Dilute the solution to 500 cc. in a volumetric flask and after thorough mixing, remove about 10 cc. and add 4-5 drops of dilute hydrochloric acid. The solution should remain clear; if, however, mercurous salts are present (indicated by the turbidity of the solution), a new assay must be started using more nitric acid and a more prolonged heating time to insure complete oxidation.

Titrate 100 cc. of the above solution with 0.1*N* Potassium Thiocyanate V.S. using 2 cc. of ferric ammonium sulfate T.S. as indicator.

Each cc. 0.1*N* Potassium Thiocyanate V.S. is the equivalent of:

Mercuric Chloride, HgCl ₂	0.013576 Gm.
Mercuric Iodide, HgI ₂	0.02272 Gm.
Mercury, Hg.....	0.01003 Gm.

Note: (1) If the original sample contains mercurous salts, it should be oxidized with bromine before proceeding with the assay. (2) If the original sample contains sodium bicarbonate as in certain germicidal tablets of mercuric iodide, diluted hydrochloric acid should be added until there is an excess equivalent to about 25 cc. (Using ten tablets, each containing mercuric iodide $\frac{3}{8}$ gr., potassium iodide $\frac{3}{4}$ gr. and sodium bicarbonate 14 gr.)

The method has been found useful in the assay of Ammoniated Mercury Ointment U. S. P. and in ointments containing ammoniated mercury and zinc oxide in combination. In this case place 15 Gm. of the ointment, accurately weighed, in a 200-cc.

beaker, warm slightly to soften the ointment and while stirring add 50 cc. ether and stir the mixture until the ointment base is dissolved. Transfer to a separatory funnel, washing the beaker with ether and diluted hydrochloric acid (10-cc. portions) until the ointment is completely transferred. Shake the mixture vigorously until all the inorganic compounds have been dissolved. Filter the aqueous layer into a 500-cc. Kjeldahl flask and wash the remaining ethereal solution with several portions of distilled water until the last washing produces no turbidity with silver nitrate T.S. Add the zinc to the acid solution in the Kjeldahl flask and proceed as outlined in the general method.

SUMMARY

In our hands this procedure has been found to yield accurate results and is more rapid than gravimetric methods. It has been successfully used in the assay of antiseptic tablets of mercuric chloride where the presence of organic dyes may lead to erroneous results if the sulfide precipitation method is employed. Other applications are evident and will be developed as opportunity arises.

Note on Philippine Turtle Oil*

By Pura Villarica and Patrocinio Valenzuela†

"A giant leathery sea turtle, known in science as *Dermochelys schlegeli* (Garman), was recorded in Philippine waters for the second time when Lt. Col. Zerbee of the U. S. Army shot the reptile in Tayabas Bay, off the coast of Lucena on March 26, 1939. The monster was brought to Manila by the S.S. *Masbate* on which the colonel was a passenger when he sighted the reptile, which had some pilot fish, or remora, attached to its back. The dorsal shield or carapace of the animal measured 194 centimeters from tip to tip and the whole animal weighed about 300 kilograms. Leathery turtles are known to attain a length of about 320 centimeters and a weight of about 500 kilograms. The turtle is known to be dis-

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