# PRELIMINARY REPORT
# ON A METHOD FOR THE DETERMINATION
# OF SMALL QUANTITIES OF THORIUM

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## ABSTRACT

A detailed procedure is presented for the separation and determination of thorium. The method is especially designed for the determination of very small quantities of thorium (as low as 0.10 mg) in rocks and ores of widely different composition, but can be used for the determination of any quantity of thorium. For quantities of thorium below one milligram, the thorium is determined colorimetrically as the iodine equivalent of thorium iodate; for quantities of thorium above one milligram, the thorium is determined gravimetrically as $ThO_2$. A discussion of the important features of the method and a summary of the more important experimental work that led to the adoption of the method are given.

## INTRODUCTION

The work of the Trace Elements Investigations of the Geological Survey necessitated the accurate determination of very small percentages of thorium in rocks and ores of widely different composition. As the methods described in the literature are not adapted to the determination of the small quantities of thorium sought or are not applicable to the determination of thorium in the presence of phosphate, a major constituent of many of the samples to be analyzed, it was necessary to develo methods especially designed for this work.

In the development of such a method adequate provision had to be made (1) for the separation of thorium from other elements, of which titanium, zirconium and rare earths are the most troublesome, (2) for the presence of phosphate, and (3) for the measurement of quantities of thorium less than one milligram.

In the procedure presented in this report separation of thorium as iodate is employed under conditions somewhat different from those described in the literature Particularly the acidity recommended was found to be too high for complete precipitation of very small quantities of thorium. For the separation of thorium from zirconium, advantage was taken of the insolubility in strong acid of the phosphate of zirconium.

Most of the titanium is also removed in this step. The rest of the titanium is removed by means of two iodate precipitations in the presence of hydrogen peroxide. The final determination of thorium is either colorimetric, as the iodine equivalent of $Th(IO_3)_4$, or gravimetric, as $ThO_2$, depending on the quantity of thorium present.

## OUTLINE OF PROCEDURE

The essential features of the method are:

1. Complete decomposition of the sample.
2. Removal of nearly all iron (and molybdenum) with ethyl acetate from a 1-1 HCl solution.
3. Precipitation of thorium with sodium carbonate. Any calcium, magnesium, titanium, zirconium, chromium, manganese, rare earths and some ~~of~~ of the aluminum and phosphate present are also ~~present~~ precipitated.
4. Solution of the carbonate precipitate in $HNO_3$ and precipitation of the thorium zirconium and titanium as iodates from 3% $HNO_3$ solution.
5. Digestion of the iodate precipitate with $HNO_3$ and addition of phosphate, separating the zirconium and part of the titanium.
6. Double precipitation of thorium as iodate in the presence of hydrogen peroxide separating the rest of the titanium.
7. Determination of thorium by ~~measure~~ measuring, spectrophotometrically, the iodine equivalent (dissolved in carbon tetrachloride) of thorium iodate or gravimetrically, as $ThO_2$, after reduction of iodate and precipitation with $NH_4OH$.

## PROCEDURE

Decomposition of sample.

1. Weigh out 5 g of each sample having a total radioactivity count equivalent to 0.015% U or less. Use proportionately smaller samples for those having higher counts. Prepare a hydrochloric acid solution of the sample as outlined in Geological Survey Trace Elements Project Report No. 2. Complete decomposition of the sample is essential as it is not considered safe to assume that no thorium is present in undecomposed material. Any hydrolytic precipitates of tantalum, columbium, tin, titanium, zirconium, and tungsten, especially in the presence of phosphate, may carry thorium and should be left in the solution.

Extraction of iron.

2. After obtaining a 1-1 HCl solution of the sample, evaporate down to a volume of about 25 ml and extract iron by shaking the solution with ethyl acetate in a separatory funnel. Shake the 1-1 HCl solution with 40 ml of ethyl acetate in a 250 ml separatory funnel and after the two layers have separated, draw off the acid layer into another separatory funnel of the same size, and repeat the shaking with 20 ml of ethyl acetate. The two extractions are sufficient as it is not necessary to remove iron completely. Draw off the acid layer into a 400 ml beaker. Combine the ethyl acetate layers and wash once by shaking with 5 ml of 1-1 HCl. Add the washings to the acid solution in the 400 ml beaker.

Separation of thorium.

3. Dilute the ~~acid~~ acid solution from the extraction of iron to a volume of 250 to 300 ml and heat to boiling. Remove from the burner and slowly add 50 percent NaOH solution until a slight permanent precipitate forms and then dry $Na_2CO_3$—very carefully—until effervescence no longer takes place, then 2 g of $Na_2CO_3$ in excess. Place the beaker in cold water bath and after 3/4 to 1 hour filter on a #40 Whatman 12.5 cm paper.

If uranium is to be determined on the same sample transfer the precipitate to the original beaker with a stream of distilled water and add 7 ml of HCl. Dilute the solution to 250 to 300 ml, heat to boiling and recipitate with 50% NaOH and dry $Na_2CO_3$ in the same way as before. Cool, filter, and wash the precipitate five times with a solution containing 10 g of $Na_2CO_3$ per liter. Combine the filtrates from the two carbonate precipitations for the ~~determinium~~ determination of uranium.

4. Transfer the carbonate precipitate to the original beaker with a stream of distilled water. Add $HNO_3$ dropwise, with constant stirring, until the mixture is red to methyl red, taking care to disperse lumps of the precipitate with a stirring rod. Add 5.0 ml of $HNO_3$ in excess and make the volume up to 100 to 125 ml with distilled water. Add 50 ml of a 7 percent water solution of $KIO_3$, stir and let stand covered over night.

Filter, without washing, on a #42 Whatman 9 cm paper. Transfer the precipitate to the original beaker with a stream of water. Replace the filter paper in the funnel and pass 30 ml of 2-1 $HNO_3$ through the paper into the beaker. By means of a stirring rod bring the solution into contact with any precipitate left on the walls of the beaker. Adjust the volume to about 100 ml with $H_2O$.

5. Boil the solution for about 1 minute, add 0.4 g of ~~any~~ anhydrous disodium acid phosphate ($Na_2HPO_4$), stir the solution well and digest on a steam bath until the volume is reduced to about 50 ml.

6. Filter on a #41 Whatman 9 cm paper, transfering most of the precipitate to the paper. If the filtrate is not clear, refilter on the same paper until a clear filtrate is obtained. Rinse out the beaker and wash the precipitate and ~~paper~~ paper with 40 ml of 1-9 $HNO_3$ containing 0.4 percent $Na_2HPO_4$ (anhydrous).

7. Neutralize the filtrate to the methyl red end point with 50 percent NaOH solution and add 10 ml of NaOH solution in excess. Digest on the steam bath for about 1/2shour.

Filter on a #40 Whatman 9 cm paper (or a smaller #40 Whatman paper if the size of the precipitate permits). If the filtrate comes through turbid, refilter on the same paper until a clear filtrate is obtained. Do not wash the precipitate.

8. Place the filter paper containing the precipitate in the original beaker. Add 6 ml of 1-1 $HNO_3$ and macerate the paper with the aid of a stirring rod. Add 25 ml of $H_2O$ and stir well.

Filter on a #40 Whatman 7 cm paper. Wash with 40 ml of $H_2O$, catching the filtrate and washings in a 150 ml beaker and allow to drain well.

9. To the filtrate, the volume of which is now 70 ml, add 10 ml of $H_2O_2$ (27-30 percent), then 20 ml of a 7 percent water solution of $KIO_3$. Stir and let stand overnight. The next morning add 5 ml of $H_2O_2$ (27 to 30 percent), stir and allow to settle for 30 minutes.

Filter on a #42 Whatman 5.5 cm paper. While filtering do not replace stirring rod in beaker as this stirs up the precipitate so that it may run through the filter. Do not wash. Place the paper containing the precipitate in the original beaker. Add 6 ml of 1-1 $HNO_3$ and macerate the paper with the aid of a stirring rod. Add 25 ml of $H_2O$ and stir.

10. Filter on a #42 Whatman 5.5 cm paper, catching the filtrate in a 150 ml beaker. If the filtrate is not clear refilter on the same paper until a clear filtrate is obtained. Wash with 40 ml of $H_2O$ and and drain well. To the filtrate, the volume of which is now 70 ml) add 10 ml of $H_2O_2$ (27 to 30 percent), then 20 ml of a 7 percent water solution of $KIO_3$. Stir.

At the same time prepare a solution containing 0.8 mg of $ThO_2$ in 67 ml of $H_2O$ and 3.0 ml of concentrated $HNO_3$. To this add 10 ml of $H_2O_2$ (27 to 30 percent), then 20 ml of a 7 percent ~~of a 7 percent~~ water solution of $KIO_3$. Compare the turbidity developed in this solution with that developed in the same solution to estimate the thorium content of the sample solution. Of the precipitate is compact and granular rather than fleecy, there is reason to suspect that the precipitate is not pure $Th(IO_3)_4$ and a reprecipitation should be made. If the $ThO_2$ content is estimated to be 0.8 mg or less, determine $ThO_2$ colorimetrically (Procedure, sections 11, 12, and 13). If the $ThO_2$ appears to be greater than 0.8 mg, determine $ThO_2$ gravimetrically (Procedure, section 14).

## Determination of thorium

### Colorimetric

11. After letting the thorium iodate precipitate settle over night, add 5 ml of $H_2O_2$, stir and allow to settle 30 minutes. Decant the supernatant liquid on a #42 Whatman 4.0 cm paper, taking care to select well packed, evenly laid papers. When about 3/4 of the supernatant liquid has been passed through the paper replace the original receiver with a 50 ml beaker. Resume filtration. After most of the

precipitate and the last drop of the liquid have been transferred to the paper, examine the filtrate. If it is not clear, refilter on the same paper until a clear filtrate is obtained. Replace the receiver with a 30 ml beaker. Rinse the walls and bottom of the precipitation beaker with a fine jet of 76 percent alcohol (80 ml of 95 percent alcohol diluted with distilled water to 100 ml). Transfer the rinsings to the filter paper, filling but not over running the paper. Scrub the beaker with a policeman, again rinse the beaker with a fine jet of ~~[illegible]~~ 76 percent alcohol and transfer the rinsings to the filter paper. Make a final rinse of the beaker with the alcohol solution and when this rinsing has drained through the paper, make 3 displacement washings of the paper with the alcohol solution. When the last of the alcohol has drained through the paper shake the liquid from the stem of the funnel. Total alcohol washings should not exceed 15 ml. Wash off the tip of the funnel with the ~~aich~~ alcohol solution.

12. Replace the receiver with a 100 mm (19 mm diameter) spectrophotometer cell. Treat the paper in the ~~g~~ funnel with 1 ml of 1-1 $H_2SO_4$, dropping the $H_2SO_4$ around the top of the paper, then tilt the funnel to insure contact of the $H_2SO_4$ with any precipitate that may have crept above the top of the paper. Let the $H_2SO_4$ stand in contact with the paper for about 5 minutes to permit complete solution of the precipitate. Wash the paper 6 times by running a jet of distilled water around the top of the paper. Total washings should not ~~exceed~~ exceed 10 ml.

13. Add 10 ml of carbon tetrachloride to the solution in the cell, add 0.2 ml of hypophosphorous acid, stopper the tube and shake for 2 minutes. Immediately measure the percent transmittancy of the colored solution in a spectrophotometer set at a wavelength of 520 millimicrons. Obtain the mg $ThO_2$ equivalent by reference to a standard curve. (See Experiments, II.) If a spectrophotometer is not available the mg $ThO_2$ equivalent of the colored solution may be estimated by comparison with a series of standards prepared from methyl red and thymol blue. (See Experiments, III.)

## Gravimetric

14. After letting the precipitate of thorium iodate (Procedure, section 10) settle over night, add 5 ml of 27-30 percent $H_2O_2$, stir and allow to settle for 30 minutes. Filter on a #42 Whatman 5.5 cm paper. Make no effort to ~~trans-fe-~~ transfer all the precipitate from the beaker to the filter paper. Do not wash. Allow the paper to drain and place paper and precipitate in the original beaker. Add 5 ml of HCl and macerate the paper with the aid of a stirring rod. Place the beaker on the steam bath for about 5 minutes, then add 75 ml of $H_2O$. While stirring, add $NH_4OH$ to the methyl red end point, then 2 ml of concentrated $NH_4OH$ in excess. Place on the steam bath for about 15 minutes.

Filter on a #42 Whatman 5.5 cm paper, transfering all the precipitate to the paper by rinsing the beaker with a few ml of the filtrate. Do not wash. Allow the filter paper to drain well. Dry the precipitate, wrapped in its moist paper, in a weighed platinum crucible, char the paper slowly without allowing it to ignite, and burn the carbon off with a gradually increased flame. Finally heat at the full heat of a Meker burner and blast for about 5 minutes. Weigh the residue as $ThO_2$.

## DISCUSSION OF PROCEDURE

The procedure, outlined in Geological Survey Trace Elements Project Report No. 2, which has been found to be suitable for the decomposition of most materials, results in a HCl solution of the sample. By the carbonate precipitation, thorium together with calcium, magnesium, titanium, zirconium, chromium, rare earths and some aluminum and phosphate, is removed from the HCl solution. The carbonate precipitation also separates thorium from uranium (the uranium going into the filtrate), thus making it possible to determine both elements on the same sample (see Geological Survey Trace Elements Project Report No. 2).

The iodate precipitation made on the $HNO_3$ solution of the carbonate precipitate effects a preliminary separation of thorium, titanium, and zirconium from most metals including the rare earths (except quadrivalent cerium). However, if the sample contains much titanium and zirconium there may be some occlusion of these elements. If the sample contains phosphate, that also may be retained combined with titanium and zirconium.

Preliminary tests showed that when the quantity of thorium present is very small, complete recovery is not obtained in the very strong nitric acid concentration commonly used for the precipitation of thorium iodate. Experimental work showed that complete recovery of very small amounts of thorium is effected only when the acidity as nitric acid is less than 5 percent. In the present work an acidity of 3 percent is used.

Thorium phosphate, even in the presence of excess phosphate, is readily soluble in 15 percent $HNO_3$. On the other hand, zirconium ~~phosphate~~ phosphate is very insoluble, even at much higher acidity. Tests showed that when zirconium phosphate is boiled in 1-1 $HNO_3$ and in concentrated $HNO_3$, the amount of zirconium dissolved is ~~sufficient~~ insufficient to give a precipitate with $KIO_3$ in 3 percent $HNO_3$ solution. Titanium phosphate is considerably more insoluble than thorium phosphate, but not as insoluble as zirconium phosphate. The acid treatment of the phosphates, therefore, results in a separation of zirconium and part of the titanium from thorium.

The low acidity (3 percent) used in the iodate precipitation of thorium requires a reduction of the acidity of the filtrate from the acid treatment of the phosphates. This is accomplished by precipitating the thorium, and any titanium present, in the filtrate from the acid treatment, with sodium hydroxide, then dissolving this precipitate with 6 ml. of 1-1 $HNO_3$. The volume in which the sodium hydroxide precipitate is dissolved plus the volume of wash solution used is adjusted to provide a final volume of 100 ml after the addition of $H_2O_2$ (27 to 30 percent) and $KIO_3$ in the subsequent iodate precipitation.

Precipitation of thorium as the iodate from the solution of the sodium hydroxide precipitate in the presence of an excess of $H_2O_2$ effects a separation of thorium from any titanium and rare earths carried through to this point. After the solution has settled over night, $H_2O_2$ is again added before filtering to dissolve any titanium iodate precipitated by escape of $H_2O_2$. A second iodate precipitation, again in the presence of excess $H_2O_2$, is desirable, however, to remove any traces of titanium and cerium that may have been occluded by the paper or the precipitate. If the sample is very high in cerium it may be necessary to make one or more additional iodate-$H_2O_2$ precipitations.

In washing the thorium iodate precipitate prior to the colorimetric determination, it is necessary to use a solution which will remove $KIO_3$ completely from the filter paper and in which thorium iodate is insoluble. A number of wash solutions were tested but none was found to fulfill the conditions as effectively as 76 percent alcohol. The size filter paper used and procedure for washing outlined was found to be most satisfactory and it is strongly suggested that it be followed carefully.

When thorium iodate is ~~reduced~~ reduced with hypophosphorous acid in the presence of $H_2SO_4$, iodine is liberated. $Th(IO_3)_4 + 2H_2SO_4 + 5H_3PO_2 = Th(SO_4)_2 + 5H_3PO_4 + 2H_2O + 2I_2$. By causing the reaction to take ~~p~~ place in the presence of carbon tetrachloride the iodine liberated is immediately dissolved, imparting a bluish-red color to the carbon tetrachloride. As the iodine liberated is directly ~~equivalent~~ equivalent to the thorium iodate reduced, a color measurement of the carbon tetrachloride-iodine solution may be used as an indirect measurement of the thorium iodate. Because the measurement is indirect, it is, of course, essential that the thorium iodate be free of any other iodate.

In the gravimetric determination, it was found that complete solution of the thorium and reduction of the iodate in the thorium iodate precipitate was brought about by treatment with concentrated hydrochloric acid, as proved by repeated determinations on samples of known thorium content. The thorium hydroxide precipitate, made with an/excess of ammonia, must not be washed after being filtered, because a minute quantity of the thorium hydroxide invariably goes through the paper. Weak ammonia and solutions of varying strengths of ammonium nitrate were tried as wash solutions and found to be ~~less~~ ineffective in preventing small quantities of the thorium hydroxide from running through the ~~paper, th~~ paper. However, the amount of KCl (the only contaminant at this stage of the analysis) remaining in the well drained precipitate is of the order of 0.1 mg and most if not all of this is volatilized by the blast lamp.

## EXPERIMENTS

### I Analysis of synthetic mixtures

The procedure described herein was tested by determining thorium in solutions whose thorium content was not known to the analyst. The test solutions were prepared by adding different amounts of a standard thorium nitrate solution to 50 ml of either o two synthetic mixtures. The standard thorium solution was made from reagent grade thorium nitrate that was found, spectrographically, to contain less than 0.1 percent of any other metal. The solution was standardized by precipitating the thorium as ~~iode~~ iodate from a 10 ml aliquot, filtering and reducing with concentrated HCl and finally weighing as $ThO_2$ after precipitating with ammonia. As a further check on the standardization eight samples containing 0.2 ml to 1.6 ml inclusive of $ThO_2$ solution were precipitated as the iodate and the iodine equivalent, of $Th(IO_3)_4$ determined spectrophotometrically. The readings were then plotted against a theoretical $KIO_3$-$ThO_2$ curve (see Experiments, II). The two methods of standardization checked very closely.

One of the synthetic mixtures, Solution S, was made to represent the average composition of a solution obtained on decomposition of a shale (as outlined in Geological Survey Trace Elements Project Report No. 2), the other mixture, Solution P, was made to represent the solution obtained on decomposition of a phosphate rock. The solutions contained 10 percent HCl. Fifty milliliters of each solution contain respectively:

| | Solution S | Solution P | Compound used |
|---|---|---|---|
| $Al_2O_3$ | 0.75 g | 0.20 g | $AlCl_3.6H_2O$ |
| $Fe_2O_3$ | .50 | .20 | $FeCl_3.6H_2O$ |
| MgO | .05 | .05 | $MgCl_2.6H_2O$ |
| CaO | .50 | 1.50 | $CaCl_2$ |
| $K_2O$ | .30 | .20 | KCl |
| $TiO_2$ | .05 | ... | $TiCl_4$ |
| $ZrO_2$ | .005 | ... | $ZrO(NO_3)_2.2H_2O$ |
| $V_2O_5$ | .02 | .02 | $(NH_4)_2V_2O_5$ |
| $Cr_2O_3$ | .02 | .02 | $K_2Cr_2O_7$ |
| $MoO_3$ | .005 | .005 | $(NH_4)_6Mo_7O_{24}.4H_2O$ |
| MnO | .005 | .005 | $MnCl_2.4H_2O$ |
| BaO | .005 | .005 | $BaCl_2.2H_2O$ |
| PbO | .005 | .005 | $PbCO_3$ |
| $P_2O_5$ | .010 | 1.25 | $(NH_4)_2HPO_4$ |
| $Y_2O_3$ | .005 | ... | $Y(NO_3)_3.4H_2O$ |
| $CeO_2$ | .005 | ... | $Ce(NO_3)_4$ |
| NiO | None | .005 | $NiCl_2.6H_2O$ |
| $As_2O_5$ | None | .005 | $Na_2HAsO_4.12H_2O$ |

$TiO_2$, $ZrO_2$, $CeO_2$ and $Y_2O_3$ were not added to stock solution P but a separate solution, Solution C, containing these constituents was made up. Each 10 ml of Solution C solution contained

| | | | |
|---|---|---|---|
| $TiO_2$ | 0.05 g | added as | $TiCl_4$ |
| $ZrO_2$ | .005 | " " | $ZrO(NO_3)_2.2H_2O$ |
| CeO | .010 | " " | $Ce(NO_3)_4$ |
| $Y_2O_3$ | .010 | " " | $Y(NO_3)_3.4H_2O$ |

In preparing the test solutions 50 ml of Solution S respectively were measured out. To each 50 ml of solution P was added 10 ml of solution C. Definite quantities unknown to the analysts of standard thorium nitrate solution was added to each test solution, and thorium was separated and determined by the procedure herein outlined. The results obtained are given in Table I.

Table I. Determination of thorium (in milligrams) in synthetic mixtures

| Sample No. | Type of test sample | $ThO_2$ added | $ThO_2$ found colorimetrically | $ThO_2$ found gravimetrically |
|---|---|---|---|---|
| T4 | S | .05 | .11 | ... |
| T1 | P | .30 | .42 | ... |
| T5 | S | .40 | .42 | ... |
| T6 | P | .70 | .66 | ... |
| T3 | P | .90 | .75 | ... |
| T8 | S | .80 | .83 | ... |
| Y1 | P | 1.2 | ... | 1.2 |
| T7 | S | 1.4 | ... | 1.3 |
| Y4 | P | 1.6 | ... | 1.6 |
| T2 | P | 1.85 | ... | 1.5 |
| Y2 | P | 2.4 | ... | 2.2 |
| Y3 | P | 4.7 | ... | 4.6 |

## II Standard reference curve for the spectrophotometric determination of thorium

Figure 1 shows the spectral transmittancy curve, using a Coleman spectrophotometer, of a solution of iodine in carbon tetrachloride. There is a definite minimum transmittancy at 520 millimicrons. This curve was obtained against carbon tetrachloride as reference.

The transmittance-concentration curve of carbon tetrachloride solutions of iodine equivalent to different amounts of thorium is given in Figure 2. To obtain this curve a solution of $KIO_3$ was used of which each millimeter was equivalent in $IO_3$ content to 0.2 mg of $ThO_2$ as $Th(IO_3)_4$. Definite amounts of the $KIO_3$ solution were measured out into the cells, the volumes were made up to 9 ml, and in the following order, 1 ml of 1-1 $H_2SO_4$ and 10 ml of carbon tetrachloride were added. The color was developed in one sample at a time by adding 0.2 ml of hypophosphorous acid, stoppering and shaking for 2 minutes and the reading made on the spectrophotometer before developing the color in the next sample. The curve is based, therefore, on the theoretical iodate-iodine equivalent of thorium.

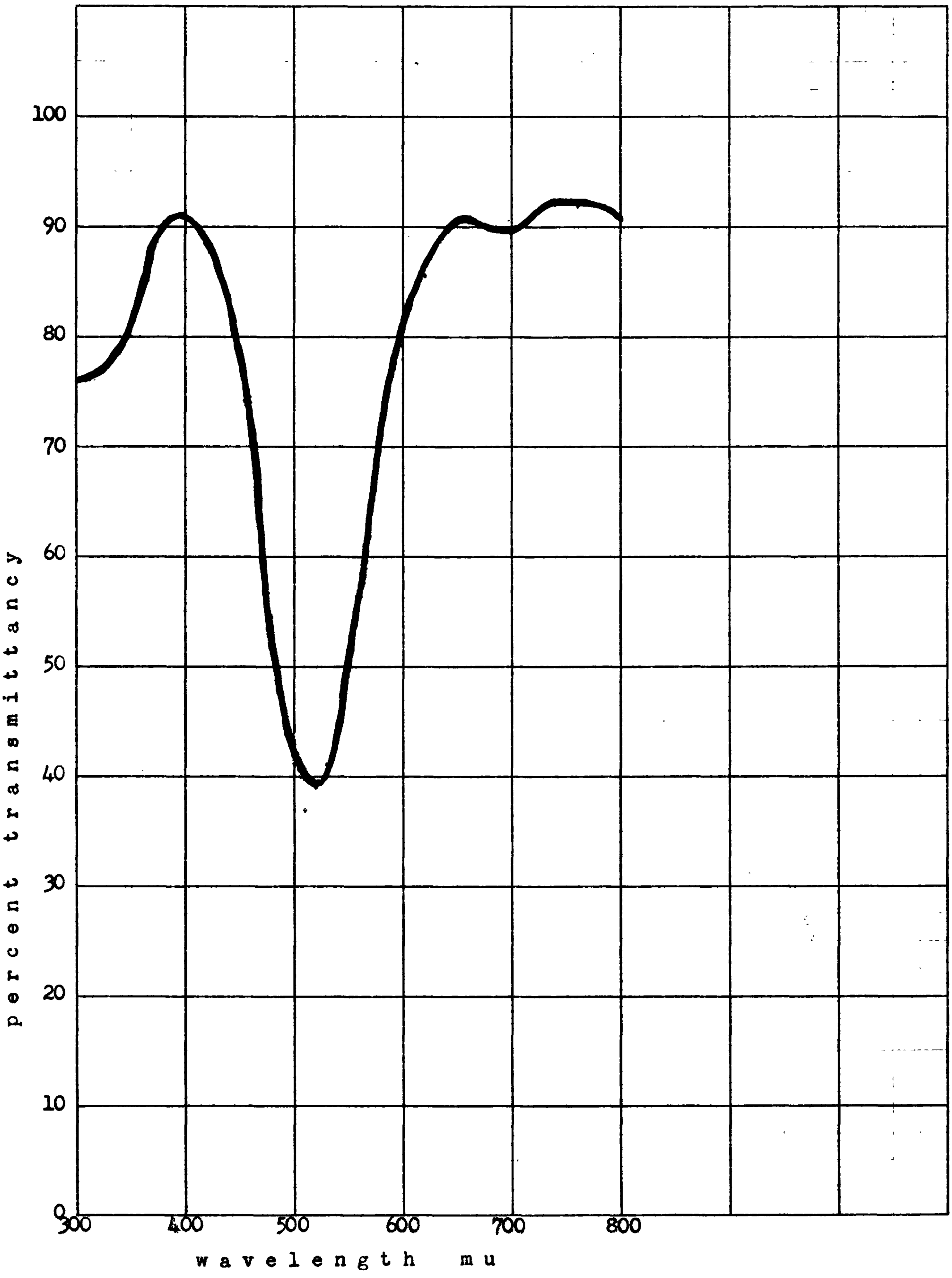
To check the validity of this theoretical curve and to check the completeness of recovery of thorium as $Th(IO_3)_4$ when precipitated from a 3% $HNO_3$ solution with 20 ml of a 7 percent water solution of $KIO_3$, definite amounts of the standard thorium solution were measured out and the thorium precipitated as iodate, filtered, dissolved, and the color developed and read as described in the colorimetric procedure (see Procedure, Sections 11, 12 and 13). The readings indicate that the ~~theorit~~ theoretical transmittance-concentration curve is valid and that the recovery of $Th(IO_3)_4$ from a 3% $HNO_3$ solution is complete within the limits of experimental error.

## III Synthetic colorimetric standards

The spectral transmittance curves of methyl red and of thymol blue in acid solution are very similar to the spectral transmittance curve of iodine in carbon tetrachloride—with the minimum transmittance at 520 and 540 millimicrons respectively compared with 520 millimicrons for iodine in carbon tetrachloride. From the curves it would appear that methyl red alone in the proper concentration should closely match the color of iodine in carbon tetrachloride, but actually the color is not quite blue enough and it is necessary to add some thymol blue (in acid solution) to exactly match the color.

The standards may be prepared by visually matching different concentrations of an acid methyl red-thymol blue solution with iodine-carbon tetrachloride solutions prepared from known amounts of $KIO_3$ or, if a spectrometer is available, by adjusting such mixtures to give the proper reading on the spectrophotometer for iodine equivalen to definite amounts of thorium. Such synthetic standards have been found to be stable for about a month.

Figure I.
Spectral transmittance curve of iodine in carbon tetrachloride.



Analyst ______ Date ______ File No. ______ Method ______

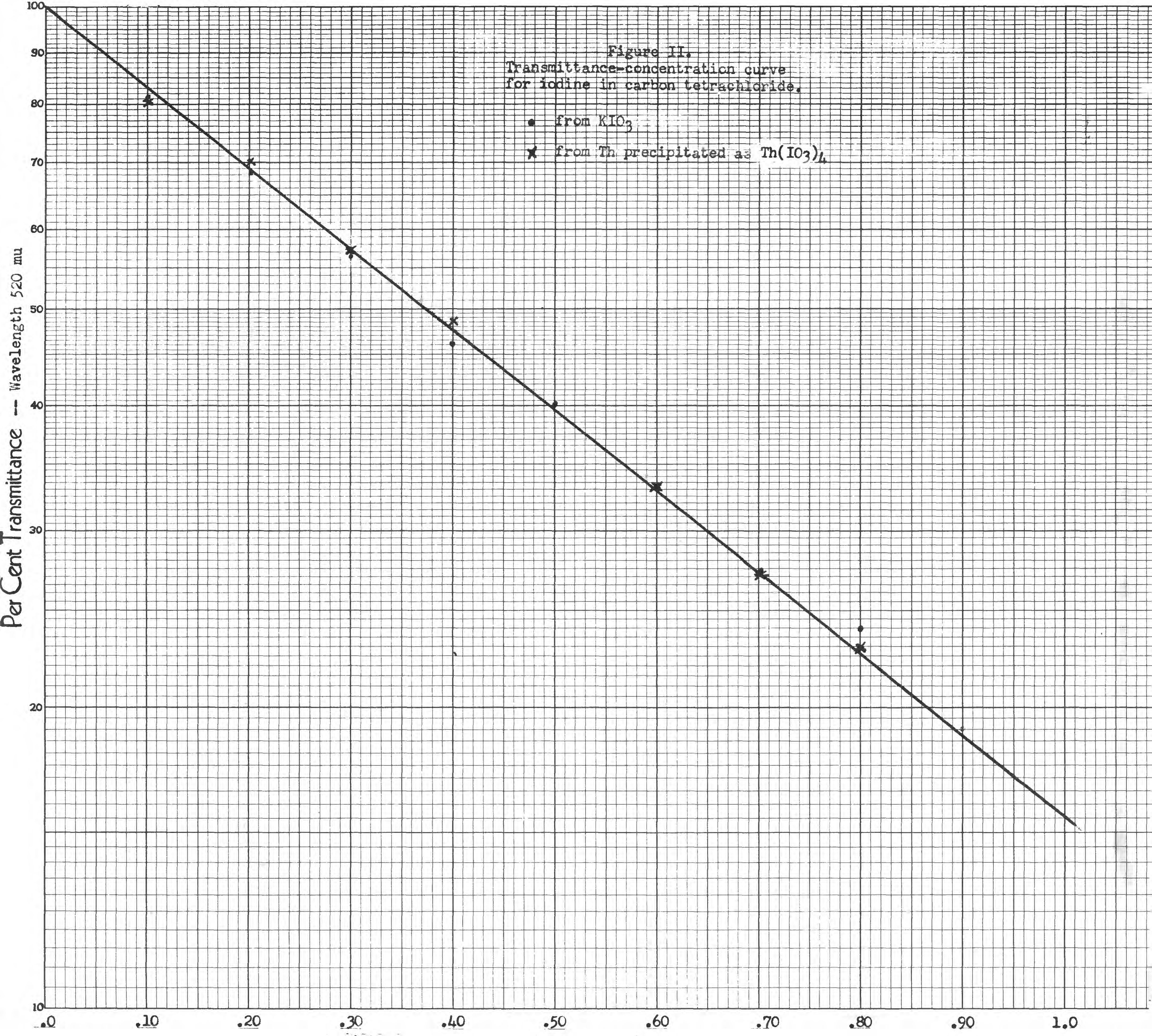


Figure II.
Transmittance-concentration curve
for iodine in carbon tetrachloride.

Figure III.

Spectral transmittance curves of iodine in carbon tetrachloride, methyl red in acid solution, thymol blue in acid solution.