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PRELIMINARY REPORT
ON METHODS OF ANALYSIS
FOR VERY SMALL PERCENTAGES OF URANIUM

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Abstract

Procedures are presented in detail of two methods for the determination of very small percentages of uranium (down to 0.002%). The methods are applicable to a wide variety of materials. Notes on the methods and confirmatory experiments are also given.

Introduction

The Chemical Laboratory of the Geological Survey was requested to develop methods by which accurate determinations of very small percentages of uranium could be made on rocks and ores of widely varying types. This report presents the procedures developed and now in use, and is preliminary; further work is in progress.

Samples for analysis are selected by geologists in the field on the basis of field counts of total radioactivity by means of a portable Geiger-Müller counter (see Trace Elements Project Report No. 1, by A. L. Slaughter and S. H. Clabaugh), and similar more accurate laboratory counts of total radioactivity.

The diversity of the samples to be analyzed, which include shales, phosphate rock, pegmatites, rhyolites, ores of various metals, fluorites, and other types, has made necessary a rather long process for the decomposition of the sample. It is considered unsafe to assume that no uranium is present in undecomposed material; the procedure therefore provides for the complete decomposition of the sample.

The elapsed time from the beginning to the completion of an analysis by either of the methods described in this report is about four days, and each analyst can complete approximately three analyses per day, provided the samples do not require special treatment. For the accuracy obtainable, see paragraphs 9 and 18 below.

Outline of procedure

The essential features of the methods are:

a) Complete decomposition of the sample.

b) Removal of nearly all Fe by extraction with ethyl acetate from a 1-1 HCl solution.

c) Collection of the uranium by:

Method A (carbonate) consisting of:

Removal of Ca, Th, Zr, Cr, Ti, rare earths, and part of the Al by precipitation with sodium carbonate.

Separation from V and coprecipitation of U and Al as phosphates.

Method B (cupferron) consisting of:

Precipitation by cupferron after reduction of U, Ti, V, Zr, etc. (separation from Al, Ca, P, etc.).

Ignition, fusion, and solution of the cupferron precipitate.

Separation of uranyl ion by extracting the cupferron compounds of other elements with ethyl acetate.

d) Colorimetric determination of uranium by peroxide in alkaline solution.

Decomposition of sample

1. Weigh out 5.00 g. of sample, ground to pass 60-80 mesh, for samples with total radioactivity counts equivalent to 0.015% U or less; for samples with higher counts, weigh out proportionally less.

2. Ignite at top heat of a Meker or Fischer burner if the sample contains organic matter or sulfides.

3. Digest in a covered platinum dish on the steam bath for 30 minutes with 40 ml. 1-1 HNO_3. Remove the cover, add 10-15 ml. of HF, and take to dryness on a slow steam bath. Repeat if there is much unattacked material. Evaporate to dryness

twice with HNO_3. Transfer the dry residue in the platinum dish to porcelain or glass, rinse with 1-1 HCl, add 10 ml. of HCl and evaporate to dryness. Follow by two more evaporations to dryness with HCl. Digest the residue, covered, with 20 ml. of 1-1 HCl and filter. Wash with hot 1-1 HCl and finally with a little hot water. Ignite the residue in platinum, add a little HF and one drop of H_2SO_4 and take to dryness. Sinter the remaining residue with as little Na_2CO_3 as possible and dissolve in 1-1 HCl. If there is an unattacked residue, filter, adding the filtrate to the main sample and again sinter the residue with Na_2CO_3 and dissolve in 1-1 HCl.

If much SiO_2 separates when the fusion is treated with 1-1 HCl, evaporate to dryness in platinum, treat with HF and a few drops of H_2SO_4, and fume. Take up in 1-1 HCl and add to main sample. If little or no SiO_2 separates when the fusion is treated with 1-1 HCl, add the solution to the previous filtrates. Evaporate the combined filtrates to about 25 ml. If there is any residue, filter. Dissolve the residue in hot water and add this solution back to the main portion after Step 4 (next paragraph).

(See Notes, paragraphs 19 and 20, and Confirmatory Experiments, paragraph 32.)

Extraction of Iron

4. Extract Fe by shaking the 1-1 HCl solution with ethyl acetate in a separatory funnel. Two extractions are sufficient as it is not necessary to remove the Fe completely. Use 40 ml. of ethyl acetate for the first extraction, and 20 ml. for the second. Combine the ethyl acetate layers and wash twice with 1-1 HCl (5 ml. each), and add the washings to the acid water layer. Finally discard the ethyl acetate layer containing the iron. Proceed to the determination of uranium by method A (carbonate) or method B (cupferron).

Procedure

5. Evaporate the combined acid water layers to 25 ml. (This step is important as it limits the acid concentration and subsequent salt concentration of the solution.

6. Dilute—in a 400 ml. beaker—to 275 to 300 ml. with distilled water and heat to boiling. Add 50% NaOH solution until a slight permanent cloud forms (about 15 ml. of 50% NaOH solution is required) 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 a cold water bath and filter after 3/4 to 1 hour through #40 Whatman 12.5 cm. paper. Wash the precipitate 3 times with a wash solution containing 10 g. of NaCl and 10 g. of Na_2CO_3 per liter. Transfer the precipitate from the paper to the beaker with a stream of distilled water, add 10 ml. HCl. After the precipitate has dissolved dilute the solution to 275 to 300 ml., heat to boiling and reprecipitate with 50% NaOH and dry Na_2CO_3. Cool and filter as before—adding the filtrate to the filtrate from the precipitation. Make the filtrate acid with HCl and add 2 ml. in excess, noting the approximate amount of Al precipitated as the neutral point is passed (see notes, paragraphs 22 and 23).

7. Heat to boiling and reduce the volume of the combined filtrates to about 150 ml. (Some time may be saved here if the filtrate from the 1st carbonate precipitation is evaporated down during the 2nd carbonate precipitation and filtration). Add $AlCl_3$ solution (1 ml. = 5 mg. Al_2O_3), 7-10 ml. if the solution contains no Al, 4-5 ml. if in the process of neutralization the solution was found to contain some Al. If a precipitate forms, add sufficient 1-1 HCl to dissolve it. Add 1 g. of dry $(NH_4)_2HPO$ and, with the solution at the boiling point, add NH_4OH until the solution is just alkaline to methyl red, then add 25 ml. of ammonium acetate-acetic acid buffer solution, containing 308 g. of ammonium acetate and 460 ml. of acetic acid per liter. Again heat the solution (with ppt.) to boiling, and digest on the steam bath for 1/

hour. Filter through #42 Whatman 9 cm. paper and wash the precipitate 5 times with a solution containing 20 ml. of the ammonium acetate-acetic acid buffer solution above in 100 ml. of water. (See Notes, paragraph 22).

8. After the precipitate has drained, transfer it from the paper to a 100 ml. beaker with a fine jet of distilled water, keeping the volume below 50 ml. Add 5 ml. of 50% NaOH solution to dissolve the precipitate and 3 drops of superoxol (see Notes, paragraph 23) to develop the color. Remove paper fibers and possible traces of Fe and Ce by filtering through a paper that has been treated with a NaOH-H_2O_2 solution of the same concentration. (See Confirmatory experiments, paragraph 34.) Compare visually or with a spectrophotometer (at 370 mμ) with U standards containing the same amount of NaOH and of superoxol in the same volume.

Verification

9. The accuracy of the method is shown by the results of Table 1.

Table 1. Verification of method A

No.	Sample	Mg. U added	Mg. U recovered 1st filtrate	Mg. U recovered 2nd filtrate	Total U recovered
1	a/35 ml. synthetic mixture	.25	.23	... b/	.23
2	.....Do.....	.50	.45	... b/	.45
3	.....Do.....	1.00	.80	.20	1.00
4	.....Do.....	1.50	1.15	.32	1.47
5	.....Do.....	2.00	1.42	.50	1.92
6	..15ml.....Do.....	2.00	2.01	... b/	2.01
7	.....Do.....	5.00	4.65	.34	4.99
8	.....Do.....	10.00	8.30	1.62	9.92
9	.....Do.....	20.00	18.00	1.76	19.76

a/ Synthetic mixture--10 g. $CaCl_2$, 20 g. $Na_3PO_4.12H_2O$, 30 g. $AlCl_3.6H_2O$, 5 g. $MgCl_2.6H_2O$, 1 g. $MnCl_2.4H_2O$ in 1 liter of 10% (by vol.) HCl.

b/ U not determined in 2nd filtrate.

Procedure

10. Evaporate the combined water layers to dryness. Add 10 ml. of 1-1 HCl. Digest gently. Add 30 ml. of water, digest and cool. (See Notes, paragraph 24).

11. Pass the solution, 50 ml. in volume, through a small Jones reductor. (See Notes, paragraph 25). Collect the solution directly into 15 ml. of 6% cupferron solution contained in a 125 ml. Erlenmeyer flask, immersed in an ice bath, shaking the flask during the passage. Wash the reductor column with 10 ml. of 5% HCl and then with enough water to make the contents of the flask 100 ml. Let stand in the ice bath for about 5 minutes, stirring occasionally. Mix in a little paper pulp and filter. Wash with cool 6% HCl containing 1.5 g. per liter of cupferron. (See Notes, paragraphs 26 and 27).

12. Burn the cupferron precipitate in porcelain. Start the ignition at a low heat until the paper is carbonized, then increase the heat until the carbon is burned off. The final temperature should be about 750°C. (See Notes, paragraph 28).

13. Fuse with a little potassium pyrosulfate containing a little niter. (See Notes, paragraph 29). The melt is kept in quiet fusion until all the nitrate is gone and the residue is dissolved. Cool and dissolve the melt in 30 ml. of 10% by volume HCl.

14. Transfer the solution to a separatory funnel. Add 15 ml. of 6% cupferron solution. Shake for about a minute. Add 20 ml. of ethyl acetate, shake, allow to settle, and separate the layers. Make 2 more extractions of the water layer with ethyl acetate. Wash the combined ethyl acetate layers once with 15-20 ml. of cold 6% HCl. Wash this HCl layer once with ethyl acetate. Combine all aqueous acid layers. Reject the ethyl acetate layers. (See Notes, paragraph 30).

15. Evaporate the water layer to a small volume. Add 15 ml. of HNO_3 and take to dryness. Add 1 ml. H_2SO_4, cover with watch glass, fume gently on the hot plate. Destroy the last trace of organic matter by cautious dropwise addition of 1 ml. of nitric or fuming nitric acid. Allow to fume for 5 minutes after the nitric acid is gone. Repeat twice with 1 ml. portions of fuming nitric acid until the organic matter is completely destroyed. Cool.

16. Add 30 ml. of water and boil gently until a solution is obtained. Cool to about 50°C. Add 6 drops of superoxol, then 50% NaOH solution drop by drop until neutral, then 1 ml. in excess. Stir in some paper pulp and make to 50 ml. in a graduated cylinder. Filter through a 7 cm. Whatman #40 paper but do not wash. The solution is now ready for color comparison. (See Notes, paragraph 31).

17. Compare the solution visually or with a spectrophotometer with solutions containing known quantities of uranium made up to 50 ml. volume and containing 1 ml. of 50% NaOH solution and 5 drops of superoxol.

Verification

18. Table 2. Verification of method B

Sample	Mg. U in sample	Mg. U added	Total mg. U found
Synthetic mixture a/	None	0.3	0.3
.....Do.....	None	.6	.6
.....Do.....	None	1.30	1.25
.....Do.....	None	2.5	2.5
65-7 (sandstone)	.15	None	.15
.....Do.....	.15	.6	.75
108-54 (shale)	.5	None	.5
.....Do.....	.5	.3	.8

a/ Synthetic mixture: 50 g. quartz, 50 g. albite, 50 g. $Na_3PO_4.12H_2O$, 50 g. $CaCO_3$, 50 g. Fe_2O_3, 10 g. $MgCO_3$, 10 g. MnO_2, 1 g. chrome alum, 1 g. V_2O_5, 0.2 g. TiO_2.

19. The procedure for decomposition of the sample (paragraphs 2-3) is suitable for most materials, but has to be modified for some unusual samples. A few examples will illustrate. Samples containing much iron oxide may be digested with 1-1 HCl, evaporated to dryness, taken up in HCl and filtered. The residue is then treated with HNO_3 +HF as usual and the HCl filtrate added back. Samples containing much fluorite are best attacked by fuming to dryness with $HClO_4$. If $HClO_4$ is used, diethyl ether should be used in the subsequent extraction of Fe instead of ethyl acetate, which tend to form emulsions in the presence of $HClO_4$. Some samples contain appreciable amounts of minerals such as cassiterite, rutile or columbite not readily attacked by the procedure of paragraph 3. Fusion with $KHSO_4$ in porcelain or with KOH in a nickel crucible should be substituted for fusion with Na_2CO_3 for such material.

20. The residue insoluble in 1-1 HCl and filtered off (at end of paragraph 3) is usually NaCl and occasionally $PbCl_2$. This is dissolved in hot water and added back to the main portion after iron has been removed (paragraph 4). The procedure may leave residues containing Ta, Cb, Sn, Ti, Zr, W, Th, and P_2O_5. These may be ignored as they do not occlude uranium if decomposition was complete. (See Confirmatory experiments, paragraph 32).

21. Copper, if present, is largely precipitated as the carbonate (paragraph 6). If precipitation of copper is not complete as the carbonate, as indicated by a blue hue in the filtrate from the carbonate precipitation, acidify and reduce the volume of the filtrate as in paragraph 7, adjust the acidity to 5% and pass in H_2S to precipitate the remainder of the Cu. After filtering off this precipitate, boil off H_2S from the filtrate before proceeding to the phosphate precipitation.

22. The amount of U retained in the 1st carbonate precipitation depends on the amount of U present and on the bulk of the carbonate precipitate. With the amount of precipitate to be expected from solutions resulting from the decomposition of a 5 gram sample, less than 4% of the U is retained after 2 precipitations if the U

content is less than 2 mg. (see Table 1, paragraph 9). With the amount of precipitate to be expected from solutions resulting from the decomposition of a 2 gram sample less than 2% of the U is retained after 2 precipitations if the U content is less than 20 mg.

23. The elements Cr and Ti which might interfere in the colorimetric determination of U with H_2O_2 in alkaline solution are removed by the carbonate precipitation but V is not. The phosphate precipitation is essentially for the purpose of separating U from V and is not necessary if the V content of the solution is less than 1 mg. The U can then be determined directly on the filtrate from the carbonate precipitatio after the volume has been reduced. However the salt content of the filtrate is usually so great that the volume cannot be reduced sufficiently when the U content is very low and precipitation of the U as phosphate, using $AlPO_4$ as a collector, with subsequent solution in NaOH, makes it possible to obtain the U in a smaller volume for comparison.

24. A clear solution is generally obtained at this point (paragraph 10). Sometimes, however, some additional TiO_2 may separate out. This should be filtered off and rejected. (See Confirmatory Experiments, paragraph 32).

25. A miniature Jones reductor is used (paragraph 11), 10 inches in length and of about 1/2 inch bore, containing zinc amalgamated with 3% of mercury. With ores containing copper or lead, it may be necessary to pretreat the solution with zinc to avoid precipitation of copper or lead in the reductor. The solution to be passed through the reductor should have as small a volume as is consistent with solution of the salts. The solution should contain approximately 10% by volume of HCl. Passage of the solution through the reductor reduces uranium to the trivalent and quadrivalent states, both of which are quantitatively precipitated by the cupferron in the receiver flask. The cupferron precipitation is conducted at acidities of HCl of 4 to 8% by volume. Perchlorate and sulfate do not interfere.

26. The cupferron will precipitate U (paragraph 11), along with all Fe, Ti, Zr, Hf, V, Cb, Ta, Mo, Sn and part of the W that escaped previous separation. The process separates these elements from the bulk of the elements, such as Al, P, Mn, Co, Ni, Ca and Cr present in the solution.

27. Hillebrand-Lundell points out that bivalent Cr will be partially precipitated by cupferron. Tests were made to determine to what extent this takes place under the conditions used in the analysis. Duplicate solutions containing 25 mg. Cr_2O_3 in 50 ml. of 10% by volume of HCl were passed through the Jones reductor and caught in 15 ml. of 6% cupferron solution according to the procedure of paragraph 11. The cupferron precipitates were ignited and found to be free of chromium.

28. These precautions (paragraph 12) during the ignition of the precipitate are necessary, since if much vanadium is present a fused mass may be obtained which may enclose carbon.

29. The niter is necessary (paragraph 13) in case all the carbon had not been completely burned off in the previous ignition. Uranium must be in the sexivalent stage to prevent its precipitation in the next step of the procedure.

30. This step (paragraph 14) effects the quantitative separation of uranium from Fe, Ti, Zr, V, Mo, and other elements that form insoluble precipitates with cupferron; U^{+6} is not precipitated. These cupferron precipitates are soluble in ether or ethyl acetate and are completely extracted, leaving the uranium quantitatively in the aqueous layer. For perfect separation from uranium, vanadium must be in the quinquevalent state, as quadrivalent vanadium precipitates out slowly with cupferron and some may escape separation. The acidity of the solution at the start of the extraction of the cupferrates is 8% by volume of HCl. In the extraction, practically all of the excess cupferron dissolves in the ethyl acetate. (See Confirmatory Experiments, paragraph 33).

31. The solution is filtered (paragraph 16) to remove hydroxides of Ce, and Th which may have escaped previous separation. The color produced by cerium in NaOH-H_2O_2 solution is about twice as strong as that produced by an equal weight of uranium. The cupferrate of cerium is not extracted by ethyl acetate (see Confirmatory Experiments, paragraph 33); any cerium precipitated with U^{+3} and U^{+4} will accompany uranium and interfere in the final color comparison unless it is removed at this point. If 25 mg. Ce^{+3} is originally present, about 0.6 mg. Ce is found in the first cupferron precipitate. If 2 mg. Ce^{+3} is originally present, enough Ce escapes separation to give a very faint but definite yellow in the final NaOH-H_2O_2 test. It is furthermore likely that even more cerium is carried down in the cupferron precipitate when elements such as U^{+4}, Fe, V, Ti, etc. are present. Filtration of the final NaOH-H_2O_2 solution separates uranium quantitatively from cerium. (See Confirmatory Experiments, paragraph 34).

Confirmatory Experiments

32. Even though the sample is completely decomposed, it may not be completely dissolved by 1-1 HCl. The insoluble may contain Cb, Ta, W, Ti, Zr, Sn, Th, with or without phosphorus. Hydrolyzed oxides of these elements do not occlude uranium when precipitated in the procedure, either when present alone or in combination, as shown in the experiments in Table 3.

To a mixture of the elements under investigation, as soluble ions, was added 10 ml. of HCl and 5 ml. of HNO_3. The solution was evaporated to dryness three times with intermediate addition of 10 ml. portions of HCl. The dry salts were then digested with 8 ml. of 1-1 HCl, diluted to 35 ml., again digested. The insoluble was filtered off, ignited and weighed, and uranium was determined in the filtrate.

Table 3. Non-occlusion of uranium in insoluble residues

Amount Taken, g.		Wt. insoluble residue, g.	U found in filtrate, g.
1. U	.0006		
TiO_2	.300	0.23	.0006
2. U	.0006		.0006
TiO_2	.300	0.315	
H_3PO_4, 85%	2 drops		
3. U	.0003		.0003
Cb_2O_5 a/	.100	.098	
4. U	.0003		.0003
Cb_2O_5 a/	.100	.112	
H_3PO_4, 85%	2 drops		
5. U	.0003		.0003
Ta_2O_5 a/	.100	.098	
6. U	.0003		.0003
Ta_2O_5 a/	.100	.103	
H_3PO_4, 85%	2 drops		
7. U	.0003	< .001	.0003
ZrO_2	.070		
8. U	.0003		.0003
ZrO_2	.070		
H_3PO_4, 85%	2 drops	.144	
9. U	.0003		.0003
SnO_2	.100	None	
10. U	.0003		.0003
SnO_2	.100	.002	
H_3PO_4, 85%	2 drops		
11. U	.0003		.0003
WO_3	.105	.105	
12. U	.0003		.0003
WO_3	.105	None	
H_3PO_4, 85%	2 drops		
13. U	.0003		.0003
Ce_2O_3	.100	None	
14. U	.0003		.0003
Ce_2O_3	.100	None	
H_3PO_4, 85%	2 drops		

Table 3. Non-occlusion of uranium in insoluble residues--Continued

Amount Taken, g.		Wt. insoluble residue g.	U found in filtrate, g.
15. U	.0003		.0003
Sb_2O_3	.100	.005	
16. U	.0003		.0003
Sb_2O_3	.100	None	
H_3PO_4,85%	2 drops		
17. U	.0003		.0003
Bi	.060	None	
18. U	.0003		.0003
Bi	.060	None	
H_3PO_4,85%	2 drops		
19. U	.0003		.0003
Cb_2O_5, a/	.100		
Ta_2O_5 a/	.100	.37	
TiO_2	.300		
ZrO_2	.070		
SnO_2	.100		
WO_3	.105		
20. U	.0003		.0003
Cb_2O_5 a/	.100		
Ta_2O_5 a/	.100	.67	
TiO_2	.300		
ZrO_2	.070		
SnO_2	.100		
WO_3	.105		
H_3PO_4,85%	4 drops		
21. U	.0025		.0027
Cb_2O_5 a/	.100		
Ta_2O_5 a/	.100	.67	
TiO_2	.300		
ZrO_2	.070		
SnO_2	.100		
WO_3	.105		
H_3PO_4,85%	4 drops		

a/ Added after Na_2CO_3 fusion.

33. The following tests were made to determine the effectiveness of ether and ethyl acetate in extracting the precipitates formed with cupferron (Procedure, paragraph 14). The elements taken were contained in 30 ml. of solution containing 4 ml. of HCl. This solution was cooled in an ice bath and 10 ml. of cool 6% cupferron solution was added. The mixture was extracted 3 times with 15 ml. portions of ether or ethyl acetate. The aqueous layer or organic solvent layer was then tested for the elements in question after destruction of the organic matter.

Table 4. Effectiveness of ether and of ethyl acetate in extracting cupferron precipitates

Elements Taken, g.	Amount in g. left in aqueous layer after extraction with Ethyl Acetate	Ether	Method of Detection
0.018 Fe^{+3}	.000005 Fe	.000003	KCNS-acid
.015 Ti^{+4} + .004 Fe^{+3}	No Ti	No Ti	H_2O_2-acid
.025 Ce	.025 Ce	....	NaOH-H_2O_2 b/
.014 V^{+5} + .004 Fe^{+3}	< .000005	< .000005	Phosphotungstic acid
.017 Mo^{+6} + .004 Fe^{+3}	.000005 Mo	....	$SnCl_2$-KCNS
.024 W^{+6} + .7 ml. 85% H_3PO_4	.020 W	....	Evaporation of ethyl acetate and ignition to WO_3
.024 W^{+6} + .7 ml. 85% H_3PO_4 + .004 Fe^{+3}	.020 W	....	Acid cinchonine b/
.017 Cr^{+3} + .004 Fe^{+3}	.017 Cr	....	Na_2O_2 Fusion b/
.018 Zr^{+4} + .004 Fe^{+3} a/	No Zr	....	NH_4OH
.022 Th^{+4} + .004 Fe^{+3}	.002 Th	....	NH_4OH
.020 Sn^{+4}	No Sn	....	Acid N_2O

a/ Zr cupferrate did not dissolve completely in ethyl acetate but floated in it and was just as effectively removed from the water layer.

b/ Ethyl acetate layer tested.

34. The removal of ~~kkm~~ cerium by the $NaOH-H_2O_2$ precipitation is essentially complete, as shown in the experiments in Table 5.

Table 5. Separation of Ce from U by $NaOH-H_2O_2$

Ce taken, mg.	U taken, mg.	U found in filtrate, mg.
5.0	None	None
5.0 (Repeat)	None	None
5.0	0.3	0.3
2.0	None	None
2.0	0.3	0.3
1.0	None	None
1.0	0.3	0.3
0.2	None	None
0.2	0.3	0.3
0.1	None	None
0.1	0.3	0.3
0.03	None	None
0.03	0.3	0.3

Comparison of results by methods A and B

Table 6. Comparison of results by methods A and B

Sample No.	Type of material	% U Method A	% U Method B
65-7	Sandstone	.003	.003
150-4-20	Shale	.006 a/	.006
150-4-29	Glauconite-shale	.005 a/	.0075
150-4-23	Shale	.007 a/	.007
150-4-24	Clayey shale	.007 a/	.009
150-4-28	Glauconite-shale	.007 a/	.009
150-7-50	Shale	.008 a/	.008
108-54	Shale	.008 a/	.010
21-3	Gold concentrate	.013	.012
150-9-64	Phosphate rock	.016	.014
21-2	Gold concentrate	.026	.028

a/ These samples were decomposed by treatment with HNO_3 + HF, followed by fusing with H_2SO_4, and the decomposition may not have been complete.