A Field Chromatographic Method for Determination of Uranium in Soils and Rocks

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By CHARLES E. THOMPSON *and* H. W. LAKIN

A CONTRIBUTION TO GEOCHEMISTRY

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A rapid and inexpensive field method for determination of trace quantities of uranium in soils and rocks.

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A CONTRIBUTION TO GEOCHEMISTRY

A FIELD CHROMATOGRAPHIC METHOD FOR DETERMINATION OF URANIUM IN SOILS AND ROCKS

By Charles E. Thompson and H. W. Lakin

ABSTRACT

A simple and rapid field method for the semiquantitative determination of uranium in soils and rocks was needed to supplement the Geiger and scintillation counter techniques now used extensively in prospecting for uranium. In the proposed method an aliquot of a nitric acid-aluminum nitrate solution of the sample is placed on a special paper, and the uranium is separated from the other sample constituents by the upward flow of a solvent mixture through the paper.

The suggested procedure is applicable to samples containing 4 to 1,200 ppm of uranium, and with a slight modification it can be used for samples containing larger amounts.

By means of the proposed method relatively unskilled workers can use inexpensive and easily obtainable reagents and equipment to determine uranium in the field in at least 60 samples of soils and sedimentary rocks ground to 100 mesh during an eight hour day.

INTRODUCTION

Although the first use of chromatographic techniques was in the separation of organic compounds, the development of partition chromatography using efficient ion-exchange resins and methods of filter paper chromatography has made chromatographic techniques very useful in inorganic research and analysis. Many of these procedures have been compiled by Pollard and McOmie (1953) and Smith (1953). It is possible to separate closely related elements such as tantalum and niobium; chromatographic methods have been developed for the separation and quantitative estimation of these elements.

The requirements for paper chromatographic techniques are: (1) a solution of the ions to be separated, (2) a strip of paper or column of cellulose to support the solution aliquot; the filter paper strips are used for micro amounts; the column of cellulose for macro amounts, (3) a solvent to effect the separation of ions in the paper, and (4) a color-producing reagent to indicate the position and relative amounts of the ions separated.

The possibilities of rapid and inexpensive chromatographic methods useful in geochemical prospecting were explored by Hunt, North, and Wells (1955). Most chromatographic techniques are time consuming and the necessary apparatus is bulky. However, these authors described rapid methods requiring little apparatus for the determination of uranium, lead, copper, cobalt, and nickel in soils and rocks.

The method described herein is an improvement of the Hunt, North, and Wells paper chromatographic procedure for uranium. The interference of chloride, fluoride, sulfate, and phosphate ions has been greatly reduced, and serious difficulties due to variation of the salt content of the sample solution have been eliminated. Also, standard conditions for drying the paper have been established which permits the use of the method in any climate.

The authors are grateful for the assistance given them by L. B. Riley in the statistical study of the results obtained by the method.

REAGENTS AND APPARATUS

All reagents used are of analytical grade.

Nitric acid, concentrated.

Nitric acid-aluminum nitrate solution. To 1 part concentrated nitric acid and 3 parts water, add aluminum nitrate until a few crystals remain undissolved two hours after the last addition.

Standard uranium solution, 0.1 percent.

Dissolve 0.295 gram of U_3O_8 in 65 ml of concentrated nitric acid, transfer the solution to a 250-ml volumetric flask, add 100 ml of water, add aluminum nitrate until a few crystals remain undissolved and dilute to volume with water.

Dilute standard uranium solutions, used in preparation of standard uranium chromatograms. Pipet the appropriate volume, given below, of the standard 0.1 percent uranium solution into 10-ml volumetric flasks containing 3 ml of nitric acid-aluminum nitrate solution, and finally dilute to the 10-ml mark with the nitric acid-aluminum nitrate solution.

Volume of standard 0.1 percent uranium solution required	Uranium content of dilute solutions	
Milliliter	*Percent*	*Micrograms per ml*
0. 1	0. 001	10
. 2	. 002	20
. 4	. 004	40
. 8	. 008	80
1. 5	. 015	150
3	. 03	300
6	. 06	600

Ethyl acetate.

Aluminum nitrate, crystalline.

Hydrofluoric acid, 48 percent.

Potassium ferrocyanide solution, 5 percent, aqueous.

Calcium chloride (anhydrous).

Whatman C.R.L./1 chromatographic paper.
Culture tubes, 16 x 125 mm, borosilicate glass.
Test tube rack, capacity 40 tubes.
Cylinders, graduated, 50 ml and 10 ml.
Balance, sensitivity 2 mg.
Beakers, 600 ml.
Dishes, platinum, 50 ml.
Dishes, porcelain, 50 ml.
Stirring rod, plastic.
Watch glasses, 125-mm diameter.
Indicator spray bottle or atomizer.
Flasks, volumetric, 250 and 10 ml.
Pipets, serological, 0.1 and 1 ml, graduated.
Pipets, volumetric, 1 and 2 ml.
Pipets, micro, 0.01 and 0.05 ml.[1]
Stove, gasoline, portable
Water bath
Desiccator, inside diameter 150 mm.

PROCEDURE

PREPARATION OF SAMPLE SOLUTION

Sedimentary rocks.—Weigh 1 g of finely powdered soil or rock sample and transfer to a 16 x 125 mm culture tube (0.1 g can be used for samples containing more than 1,200 ppm). Add 2 ml of nitric acid-aluminum nitrate solution and digest the sample for one hour in a boiling water bath. Allow the insoluble residue to settle. (Alternatively the sample solutions may be centrifuged for three minutes at 1,500 rpm to obtain a clear aliquot.)

Soils and materials containing large amounts of organic matter.—Weigh 1 g of finely powdered soil or rock sample, transfer to a porcelain dish, and ignite for 15 minutes over a gasoline stove. Transfer the ignited sample to a culture tube, add 2 ml of nitric acid-aluminum nitrate solution, and digest the sample for one hour in a boiling water bath. Allow the insoluble residue to settle.

Crystalline rocks.—Weigh 1 g of finely powdered sample and transfer it to a platinum dish. Add about 10 ml of hydrofluoric acid and 3 ml of concentrated nitric acid, and stir to effect contact of the acids with the sample. Let stand 4 hours at room temperature, and then evaporate the contents of the dish to dryness on a hot water bath. Transfer the contents of the dish to a culture tube using a plastic stirring rod; add 2 ml of nitric acid-aluminum nitrate solution to the dish, swirl, and transfer this wash solution to the culture tube. Digest the solution in the culture tube for one hour in a boiling water bath. Allow the insoluble residue to settle.

[1] Micropipets convenient for removing small aliquots of sample solution from the digestion tubes may be made as follows: Draw one end of 3-mm outside-diameter glass tubing into a capillary. To calibrate the micropipet, place the orifice of a standard 0.1-ml pipet, graduated in 0.01 ml, next to the fine capillary tip of the micropipet and allow the desired volume to enter the micropipet by capillary flow. Then mark the micropipet at the meniscus with a small file. Repeat to assure accuracy. Suitable micropipets are also commercially available.

PREPARATION OF SOLVENT MIXTURE

Transfer 30 ml of ethyl acetate, 3.5 ml concentrated nitric acid, and 10 ml of water into a 600-ml beaker. Cover the beaker with a 125-mm diameter watch glass and allow the solvent mixture to stand in the beaker 20 minutes before using.

PREPARATION OF CHROMATOGRAMS

Withdraw an appropriate aliquot of the supernatant sample solution with a micropipet and spread the aliquot evenly starting about 1.5 cm from the bottom of one of the strips on a piece of Whatman C. R. L./1 filter paper (fig. 29), and placing 10 sample aliquots of equal volume on each sheet. For samples containing 4 to 240 ppm uranium, use a 0.05-ml aliquot. For samples containing 240 to 1,200 ppm uranium, use a 0.01-ml aliquot of the sample solution. Use only one size of aliquot on each sheet of paper. Fasten the top corners (A and B in fig. 29) together with a paper clip to form a cylinder and place upright in a desiccator (with an inside diameter of 150 mm) containing dry calcium chloride for the time specified in the following tabulation chart.[2]

Temperature (° F)	*Aliquot size (ml)*	*Drying time (minutes)*
<60	0.05	20
60–90	.05	10
>90	.05	8
<60	.01	12
60–90	.01	8
>90	.01	5

After the aliquots have dried, place the paper cylinder upright in the 600-ml beaker containing the solvent mixture. Cover the beaker with a watch glass. Leave the paper cylinder in the beaker until the solvent front rises 8 cm above the bottom of the paper. The solvent front must rise 2 cm above the area wetted by the sample aliquot but should not rise above the top of the slit. Remove the paper from the beaker, unfold, and place it on a clean surface.

Allow the ethyl acetate to evaporate, then spray the chromatogram with a 5 percent aqueous solution of potassium ferrocyanide. If uranium is present a ferrocyanide complex of uranium forms a brown stain at the highest point the solvent had risen.

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[2] In connection with the development of a method for determining uranium in natural water, F. N. Ward and A. P. Marranzino of the Geological Survey have recently found that drying the paper in a desiccator containing a saturated solution of magnesium nitrate in contact with an excess of the salt provides a proper humidity for this operation. A paper may be left in the desiccator of the size described above for 20 minutes and be sufficiently dry for the development of a good chromatogram. Leaving the paper in the desiccator overnight does not change the results. The new desiccant is recommended to replace the one described here.

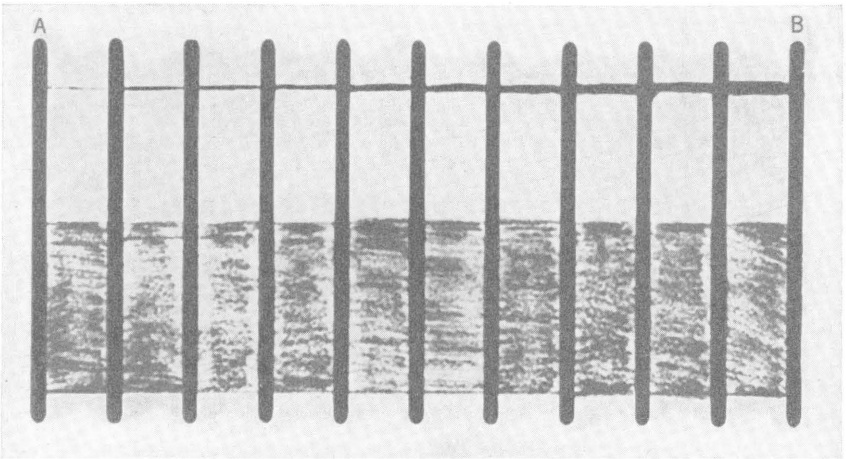

FIGURE 29. Standard series of uranium chromatograms on a sheet of C. R. L./1 Whatman paper.

PREPARATION OF STANDARDS

Prepare a series of standards containing 0.1, 0.2, 0.4, 0.8, 1.5, 3, and 6 micrograms of uranium on a sheet of C. R. L./1 Whatman paper as follows: Place a 0.01-ml aliquot of each of the standard uranium solution containing 10, 20, 40, 80, 150, 300, and 600 micrograms of uranium per ml on 7 strips of a sheet of C. R. L./1 Whatman paper. Proceed as directed for sample solutions.

ESTIMATION OF URANIUM

Compare the brown stain of the uranium complex obtained from the sample aliquot with standards prepared at the same time, and record the number of micrograms found in the sample aliquot. Use the following equation to calculate parts per million uranium in the sample:

$$\frac{\text{Total vol. of sample solution}}{\text{Wt. of sample} \times \text{vol. of aliquot}} \times \text{micrograms found} = \text{U ppm}$$

DISCUSSION

EFFECTIVENESS OF SAMPLE SOLUTION

The paper chromatographic method of separation and estimation of uranium requires that the sample be dissolved in 1+3 nitric acid and that aluminum nitrate be present in the sample solution to prevent interference of phosphate and other anions. Dilute nitric acid-aluminum nitrate solution has been found to be a very satisfactory reagent for dissolving the uranium in several hundred samples from the Colorado Plateau (table 6). However, this reagent alone would

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not extract all of the uranium in a number of igneous rock samples (table 5). Fusion with fluxes such as potassium bisulfate, potassium hydroxide, sodium hydroxide, lithium hydroxide and potassium nitrate, sodium carbonate, sodium carbonate and potassium nitrate, ammonium fluoride, or combinations of these proved cumbersome or otherwise unsatisfactory. The most satisfactory attack on this type of sample was the hydrofluoric-nitric acid digestion.

INTERFERING IONS

One hundred micrograms of uranium were added to duplicate samples of salts of a number of cations and anions, and one of the duplicate mixtures was digested with 1+3 nitric acid and the other with nitric acid-aluminum nitrate solution. The effect of these ions on the recovery of uranium is shown in table 1. The cations Ba, Sr, Li, Fe (III), Ca, Mg, K, and Na did not interfere when 100 micrograms of U was added to their nitrates. Where the 1+3 nitric acid acid digestion was used, phosphate, chloride, fluoride, sulfate in small amounts, and carbonate in extremely large amounts interfered with the separation of uranium. Carbonate is easily recognized by effervescence and can be eliminated by first evaporating the nitric acid solution of the sample to dryness and dissolving the residue in nitric

TABLE 1.—*The effect on interfering ions by the addition of* $Al(NO_3)_3$ *to nitric acid containing 100 micrograms of uranium*

Salt added (grams)	Uranium found (micrograms)	
	1+3 HNO_3 digestion	HNO_3-$Al(NO_3)_3$ digestion
1 NH_4VO_3	100	100
1 $(NH_4)_6Mo_7O_{24}$	100	100
1 $NaNO_3$	100	100
1 H_3BO_3	100	100
.5 $NaHSO_4$	0	12
.2 $NaHSO_4$	12	30
.1 $NaHSO_4$	24	60
.5 $KHSO_4$	0	0
.2 $KHSO_4$	0	30
.1 $KHSO_4$	12	60
.5 NaCl	0	30
.2 NaCl	0	60
.1 NaCl	0	100
.5 NH_4F	0	30
.2 NH_4F	0	60
.1 NH_4F	12	100
.5 Na_2HPO_4	0	100
.2 Na_2HPO_4	0	100
.1 Na_2HPO_4	0	100
1 Na_2CO_3	0	0
.5 Na_2CO_3	0	100
.2 Na_2CO_3	100	100

acid. However, with the Al $(NO_3)_3$ present it was not necessary to take to dryness and redissolve. The interference of phosphate is eliminated, and interference of the other anions is reduced substantially by the use of the nitric acid-aluminum nitrate solution for digestion of the sample.

To test further the effect of aluminum nitrate on the interference of phosphate, 20 samples containing as much as 35 percent phosphate (PO_4^{-3}) were analyzed, once by digesting with just 1+3 nitric acid, and a second time by digesting them with 1+3 nitric acid saturated with aluminum nitrate. Less than 4 ppm of uranium was found when 1+3 nitric acid was used alone. However, when these samples were digested with nitric acid-aluminum nitrate solution and the uranium estimated chromatographically, the results compared favorably with those obtained by fluorimetric estimation as shown in table 2.

TABLE 2.—*Comparison of estimation of uranium in phosphate rocks by chromatographic and fluorimetric methods*

Sample no.	Uranium by chromatographic estimation (ppm) after nitric acid-aluminum nitrate digestion	Uranium by fluorimetric estimation (ppm) after nitric acid digestion
54–473	160	140
54–468	100	120
54–539	80	120
54–464	80	90
54–472	120	90
54–476	60	90
54–480	80	90
54–495	120	90
54–466	60	70
54–475	60	70
54–494	80	60
54–504	30	50
54–533	25	40
54–492	25	30
54–406	40	30
54–515	25	20
54–516	25	20
54–544	10	20
54–546	15	20
54–620	15	20

OPTIMUM DRYING OF SAMPLE ALIQUOTS ON THE PAPER

There is an optimum moisture content of the paper wet by the aliquot at which the uranium moves in the best tight band in the solvent front. If the paper is too moist, the uranium trails behind the solvent front; and if too dry, the uranium either does not move or moves slowly. Iron moves more rapidly the drier the spot, conse-

quently excessive drying after addition of the aliquot to the paper results in the uranium being concealed in the wide blue band of the iron ferrocyanide complex.

It was observed that the optimum drying time for paper containing aliquots of 1+3 nitric acid digestions of samples varied appreciably between samples. The variation in optimum drying time that was observed is attributed by the authors to the lowering of the vapor pressure of the sample aliquot by the soluble salts occurring in the samples. In any event, the addition of a constant large amount of a salt (aluminum nitrate) to every sample overshadows the relatively small variations between samples and makes it possible to give a specific drying time for a given humidity and temperature. The data presented in table 3 illustrate that the digestion of the sample with (1+3) nitric acid saturated with aluminum nitrate results in a sample aliquot of sufficiently uniform salt content to permit prediction of optimum drying time. The optimum drying time for an 0.05-ml aliquot in a desiccator with an inside diameter of 150 mm containing anhydrous $CaCl_2$ is 10 minutes at 73° F (22.8° C). The optimum drying times for various temperatures and aliquots given on the chart in the procedure were similarly obtained.

TABLE 3.—*Comparison of quality of chromatograms obtained on a 0.05 ml aliquot at 73° F after various periods of drying. (Desiccator containing $CaCl_2$)*

[N=No band, D=Difficult to estimate, F=Fair band, G=Good band]

	Drying time, minutes				
Material	5	10	15	20	30
1+3 nitric acid					
Phosphate rock	N	N	N	N	N
Sandstone	F	F	F	F	F
Soil	D	F	F	D	D
1+3 nitric acid-aluminum nitrate solution					
Phosphate rock	D	G	F	D	D
Sandstone	F	G	F	D	D
Soil	D	G	F	D	D

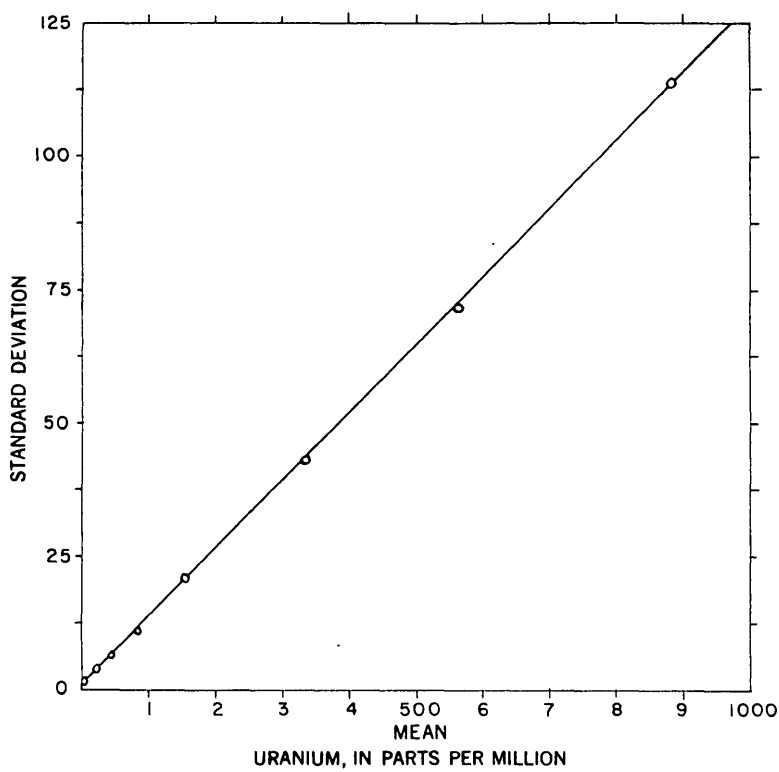
SOLVENT MIXTURE USED FOR SEPARATION OF URANIUM

The relative proportions of ethyl acetate, water, and concentrated nitric acid affect the movement of the uranium in the solvent front. Use of 35 ml of ethyl acetate, 10 ml of water, and 3.5 ml of concentrated nitric acid resulted in the best movement of the uranium with the solvent front. If these proportions are changed materially, the

uranium is not concentrated in the solvent front but is spread along the length of the strip. It has been found that this solvent mixture, because of changes in composition, is satisfactory for a maximum of about three hours only or for five sheets of paper, whichever is first.

RESULTS

The precision of the proposed method was estimated by a statistical study of 97 pairs of duplicate determinations on 97 different materials ranging in uranium content from 4 to 1,000 ppm. These samples were analyzed in eight groups according to their apparent uranium content with no less than 10 samples in each group. The mean and standard deviation of each group were calculated by the procedure described by Youden (1951, p. 17). These data are given in table 4. The mean values were then plotted against the standard deviation (fig. 30) and a straight line relationship obtained. Using the equation for a straight line $SD=a+b\ x$ where SD equals estimated standard deviation and x is the quantity of uranium found in ppm, we obtained $SD=0+0.13\ x$ or $SD=0.13\ x$ for values ranging from 6 to 900 ppm.



FIUGRE 30. Variation of standard deviation with uranium content.

TABLE 4.—*Mean and standard deviation of duplicate uranium determinations*

Range of uranium content in groups of samples (ppm)	No. of pairs of duplicate determinations	Mean (ppm)	Standard deviation (ppm)
4–12	12	6. 8	1. 7
12–30	11	20. 7	3. 3
30–60	12	42	6. 7
60–100	11	81	10. 9
100–200	16	150	20. 9
200–400	11	332	42. 6
400–600	10	567	71. 5
800–1000	15	887	112. 6

Thus we find that the estimated standard deviation for the method is 13 percent of the value obtained and, assuming normal distribution of values about their mean, that about 1 out of 20 replicate determinations will differ by more than 26 percent of the value obtained. This is adequate precision for a rapid semiquantitative field method suitable for geochemical exploration.

Data presented in table 5 illustrate the accuracy of results obtained by the proposed procedure for crystalline rocks. The figures obtained by the paper chromatographic procedure are given in the third column; the fourth and fifth columns show the range of plus and minus two standard deviations for this method.

TABLE 5.—*Comparison of uranium in crystalline rocks by chromatographic and fluorimetric estimation methods*

Sample no.	Type of material	Uranium found by chromatographic estimation (ppm)	Calculated 95 percent confidence limits [1] (ppm)		Uranium found by fluorimetric estimation (ppm)
54–4597	Hornblende syenite	4	(2)	(2)	6. 6
54–4598	Bostonite (sodic)	4	(2)	(2)	6. 7
54–4599	Quartz monzonite	4	(2)	(2)	8. 2
54–4600	Quartz monzonite	8	6	10	10. 0
54–4601	Bostonite (potassic)	16	12	20	13. 8
54–4602	Bostonite	16	12	20	17. 6
54–4603	Sodic granite	16	12	20	18. 1
54–4604	Bostonite (potassic)	16	12	20	18. 9
54–4605	Quarts monzonite (sodic)	16	12	20	19. 4
54–4606	Bostonite (sodic)	16	12	20	21. 7
54–4607	Bostonite (potassic)	24	18	30	32. 5
54–4608	Bostonite (sodic)	30	22	38	38. 3
54–4609	Bostonite (potassic)	30	22	38	41. 9
54–4610	Bostonite (potassic)	40	30	50	46. 9
54–4611	Bostonite (potassic)	40	30	50	57. 8
54–4612	Bostonite (potassic)	60	44	76	69. 8
54–4613	Bostonite (potassic)	120	88	152	138. 6

[1] 95 percent confidence limits based on calculations from data presented in table 4.
[2] These samples are outside the range of data on which confidence limits were calculated.

The fluorimetric results (last column) are consistently higher than those obtained by the proposed procedure. The low values given by the paper chromatographic procedure are probably due to incomplete solution of the sample as the proposed attack of the sample with hydrofluoric and nitric acid fails to decompose many refractory minerals such as zircon. However, the results by the paper chromatographic procedure increase in the same order as the results obtained by complete solution.

TABLE 6.—*Comparison of uranium in sedimentary rocks (other than phosphates) by chromatographic and fluorimetric methods of estimation*

Sample no.	Uranium found by chromatographic estimation (ppm)	Calculated 95 percent confidence limits [1] (ppm)		Uranium found by fluorimetric estimation (ppm)
211104	20	15	25	20
72772	20	15	25	20
72491	20	15	25	30
70256	30	22	38	20
70258	30	22	38	30
72490	30	22	38	40
D-92411	30	22	38	40
211105	40	30	50	30
D-79695	40	30	50	30
70254	40	30	50	40
72227	40	30	50	50
D-91018	50	37	66	50
72773	60	44	76	40
D-79711	60	44	76	50
D-79705	60	44	76	60
71505	80	60	100	40
211102	100	74	126	90
72866	100	74	126	60
211103	100	74	126	100
211099	100	74	126	90
211094	100	74	126	120
211093	150	112	188	110
D-91095	300	220	380	310
211095	300	220	380	270
211109	500	370	630	500
D-91016	800	600	1000	720
D-91012	900	700	1100	800
211110	1000	(2)	(2)	1000
211108	1500	(2)	(2)	1500
211106	1500	(2)	(2)	1500
211111	2000	(2)	(2)	2000
211107	2000	(2)	(2)	2000
211101	3000	(2)	(2)	1500
211097	3000	(2)	(2)	4000
211096	3000	(2)	(2)	3400
211100	3500	(2)	(2)	1700
D-91011	4000	(2)	(2)	4800
D-91012	6000	(2)	(2)	6000
211098	9000	(2)	(2)	6100

[1] 95 percent confidence limits based on calculations from data presented in table 4.
[2] These samples are outside the range of data on which confidence limits were calculated.

Thirty-nine samples of sedimentary rocks low in phosphate were run independently by the fluorometric and chromatographic methods (table 6). The upper and lower value for the 95 percent level of precision for the paper chromatographic method are given in the third and fourth columns and only two of the fluorometric values are substantially outside of these ranges. Assuming that the fluorometric values are correct, the accuracy of the proposed method for sedimentary rocks is approximately the same as the precision of the method.

The method is fast and simple enough to enable a relatively unskilled worker to determine the uranium content in 60 or more samples in the field in an 8-hour day. In this laboratory a chemist has determined uranium content chromatographically in as many as 120 samples in an 8-hour day. The method permits the estimation of as little as 4 parts per million of uranium and as much as 1,200 ppm without modification. Larger uranium contents can be determined by simple modifications.

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