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VARIATIONS IN U^{234} CONCENTRATION
OF NATURAL URANIUM

R. F. SMITH
J. M. JACKSON

UNION CARBIDE CORPORATION
NUCLEAR DIVISION
PADUCAH GASEOUS DIFFUSION PLANT

UNION
CARBIDE

PADUCAH GASEOUS DIFFUSION PLANT
P. O. Box 1410
Paducah, Kentucky 42001

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VARIATIONS IN U²³⁴ CONCENTRATION OF NATURAL URANIUM

R. F. Smith and J. M. Jackson

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OF NATURAL URANIUM

A B S T R A C T

Uranium ore concentrates from sixteen world sources have been analyzed to determine variations in the U^{234} content of natural uranium. A spread of approximately 7.5 percent of the U^{234} content was apparent among the various sources. The lowest concentration, 0.00500 ± 0.00002 Wt.% U^{234} , was obtained on ore concentrate from Mines Development in Edgemont, South Dakota. The highest concentration, 0.00539 ± 0.00002 Wt.% U^{234} , was obtained on Belgian Congo Pitchblende. The latter is in substantial agreement with "best values" quoted by Glenn T. Seaborg.

VARIATIONS IN U^{234} CONCENTRATION OF NATURAL URANIUM

In 1961, the Paducah Laboratory analyzed 17 domestic and foreign ore concentrates to evaluate variations in the U^{235} content of natural uranium^{1/}. Sixteen of the same concentrates have recently been used to measure variations in U^{234} concentrations, the other having been exhausted.

Previous work^{2/} has shown wide variations in U^{234} content for specific uranium deposits. This study was designed to reveal variations which might be expected among ore concentrates (yellowcake) received from foreign and domestic processing mills. The overall range found for the 16 concentrates was 0.00500 to 0.00539 Wt.% U^{234} . The latter is in substantial agreement with "best values" quoted by Glenn T. Seaborg^{3/}.

SAMPLE PREPARATION

Uranium was extracted from each lot of ore concentrate and ignited to stoichiometric U_3O_8 . A U^{233} spike was added to each and appropriate samples were prepared for spectrometer measurements.

Preparation of U_3O_8 From Sample

Approximately 5 grams of uranium were extracted from each lot of ore concentrate. New glassware was used for processing to minimize the possibility of isotopic contamination. It was not thought necessary to analyze all processing solutions for traces of uranium impurity as was done in the previous work^{1/}; however, precautions were taken to prevent contamination from other laboratory uranium sources.

Recovered uranium was treated with hydrofluoric acid, evaporated to dryness, and ignited for 16 hours at $850^{\circ}C$ (the first hour in a steam atmosphere). This procedure has been shown to produce stoichiometric U_3O_8 ^{4/}. Outside uranium contamination was precluded by strenuously cleaning ignition furnace and platinum dishes prior to use.

Prepared U_3O_8 was analyzed spectrographically and found to contain less than 100 ppm total metallic impurity. This quantity could not contribute significant error in making U^{234} measurements.

U^{233} Spike

Stoichiometric U_3O_8 aliquots from each of the 16 samples were spiked with approximately 54 ppm U^{233} . The U^{233} was obtained from ORNL in 1961 and further purified by the Paducah Laboratory^{5/}. Four flasks of U^{233} solution were prepared in 1964^{6/}, and the integrity of those solutions has been maintained by sealing and performing evaporation corrections. Two of those flasks were used here to provide necessary U^{233} spikes. One lot from each source was spiked from Flask No. 2 which contained 70.577 micrograms U^{233} per gram of solution. Approximately 2

grams of U_3O_8 and 1.3 grams of U^{233} solution were mixed for each of these spikes. The other lot from each source was spiked from Flask No. 4 which contained 57.242 micrograms U^{233} per gram of solution. In this case approximately 3 grams U_3O_8 and 2.4 grams solution were used in each mix. Weighing burets were employed to measure the quantity of U^{233} solution dispensed. Spiking data is summarized in Appendices A and B.

Final Sample Preparation

The U_3O_8 and U^{233} for each spike were dispensed into a new beaker and sufficient nitric acid was added to put all contents in solution. Resulting solutions were allowed to come to dryness slowly under heat lamps to provide convection current mixing. Just prior to the time uranium started precipitating, an aliquot of solution was removed from each beaker for surface ionization spectrometer measurements. The final precipitates were re-ignited to U_3O_8 according to the procedure previously described. Aliquots of resulting U_3O_8 were then fluorinated² to UF_6 for determination of the U^{234} content by gas source spectrometers.

SPECTROMETER MEASUREMENTS

Each spiked sample was analyzed on three different mass spectrometers, one CEC Model 21-702B surface ionization instrument and two CEC Model 21-220 gas analysis instruments. Magnetic scanning was employed for all measurements.

Mass Spectrometer Setup

The conventional measuring circuit rather than an electron multiplier was used for the 21-702B. The preamplifier was evacuated and a 1×10^{12} ohm input resistor was employed. The $\text{U}^{234}/\text{U}^{233}$ valley was about 4% of peak height with essentially all the lack of complete resolution contributed by the tail of the U^{238} peak.

A narrow slit collector (8 mils) and a 5×10^{11} ohm input resistor were installed in each of the Model 21-220 instruments before measurements were performed. In addition the spectrometer tubes were carefully adjusted within the magnetic field for optimum resolution. The $\text{U}^{234}/\text{U}^{233}$ valleys were approximately 10% and 4% of peak heights respectively for Spectrometers No. 4 and No. 6 with any lack of resolution being chiefly contributed by the U^{238} peak tail. Instruments were operated at a pressure of about 2×10^{-8} Torr to minimize the possibility of bias due to residual spectrometer impurity.

Analytical Measurements

A mass spectrometer determination consisted of alternately sweeping up over the U^{233} and U^{234} peaks then down over the U^{234} and U^{233} peaks

until approximately 12 peaks of each isotope had been recorded. Replicate determinations on the same spectrometer were taken at least two weeks apart to assure independence. Three or more replicate determinations were taken on each sample lot by surface ionization spectrometer. In general, two determinations per lot were performed on each of the two gas analysis spectrometers.

DATA EVALUATION AND CALCULATIONS

Data were examined to see if any obvious biases existed between independent variables, after which measurement precisions were evaluated. Then the U^{234} concentrations of the various lots were calculated.

Agreement Between Independent Variables

Average mass spectrometer measurements are summarized in Appendices C and D. An examination of the analytical data revealed that U^{233} spikes prepared from Flask No. 2 were not different from those prepared from Flask No. 4. (Flask No. 2 spikes minus Flask No. 4 spikes by gas source spectrometer = -0.000002 ± 0.000009 Wt.% U^{234}). Also, there were no significant differences among the three spectrometers used to perform the measurements.

Analytical Precision

All data obtained on each instrument were used to evaluate the precision of a single determination for that instrument. Differences between independent measurements on all 32 sample lots were compiled for each spectrometer and instrument precision calculated therefrom. Single determination precisions calculated by this method were 2.0, 1.4, and 0.8 per cent of the analytical values respectively for the surface ionization instrument, Spectrometer No. 4, and Spectrometer No. 6. In general, these precisions were used to calculate the 95% symmetrical confidence interval of the average spectrometer measurements for each lot. For a few lots with highest interval variance, the estimated internal lot precision rather than the smaller composite spectrometer precision is quoted for conservatism.

U^{234} CONCENTRATIONS

Appendices E and F summarize U^{234} measurements along with confidence intervals. A tabulation of spectrometer results for individual lots is presented. It will be observed that the results from Spectrometer No. 6 are much more precise than those from the surface ionization instrument. Thus, the average concentration quoted for each sample is a weighted average of the three instruments rather than an arithmetic average. Equations used to calculate weighted averages and confidence interval

of same are presented in Appendix G. Confidence intervals are quoted to only one significant figure and were rounded up in all cases.

For 11 of 16 ore concentrate samples, the two lots were not significantly different, and all data were pooled to give weighted averages for those eleven processing mills. Lots were different for the remaining five ore concentrate samples, and weighted averages are quoted for each lot. Since tests for significance were applied before rounding confidence intervals, the difference between lots for some of the five may not be immediately apparent from the appendices.

CONCLUSIONS

There are significant differences among U^{234} concentrations of unaltered uranium ore concentrates produced in various parts of the world. A spread of approximately 7.5% of the average U^{234} assay was found among concentrates from 16 world processing mills. Although this variation is substantial, it is small compared to that reported for specific minerals^{2/}.

It will be noted from the appendices that independent lots from five of the 16 processing mills gave U^{234} results which were significantly different. There were no differences between lots from 11 of the mills. The maximum difference observed between lots from the same mill was about 1.5% of the U^{234} assay.

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APPENDIX A.

SPIKES PREPARED FROM U²³³ FLASK NO. 2

<u>Sample</u>	<u>Lot No.</u>	<u>Grams of Sample</u>		<u>Grams of Spike</u>		<u>Weight Ratio</u> <u>U²³³/Sample U</u>
		<u>Oxide</u>	<u>Uranium</u>	<u>Solution*</u>	<u>U²³³**</u>	
Homestake	56	2.0005	1.6964	1.3261	93.592 x 10 ⁻⁶	55.17 x 10 ⁻⁶
Rayrock	R-41	2.0005	1.6964	1.2713	89.725 x 10 ⁻⁶	52.89 x 10 ⁻⁶
Faraday	F-93	2.0005	1.6964	1.2673	89.442 x 10 ⁻⁶	52.72 x 10 ⁻⁶
Kermac	92	2.0008	1.6967	1.2612	89.012 x 10 ⁻⁶	52.46 x 10 ⁻⁶
Uranium Reduction	624	2.0011	1.6969	1.2307	86.859 x 10 ⁻⁶	51.19 x 10 ⁻⁶
Calprods	142	2.0006	1.6965	1.3781	97.262 x 10 ⁻⁶	57.33 x 10 ⁻⁶
Climax	311	2.0011	1.6969	1.3666	96.451 x 10 ⁻⁶	56.84 x 10 ⁻⁶
Radium Hill	Z-62	2.0011	1.6969	1.2844	90.649 x 10 ⁻⁶	53.42 x 10 ⁻⁶
Lucky Mc	62	2.0003	1.6963	1.2254	86.485 x 10 ⁻⁶	50.98 x 10 ⁻⁶
Lakeview	2	2.0001	1.6961	1.3368	94.347 x 10 ⁻⁶	55.63 x 10 ⁻⁶
Algom	942	1.9999	1.6959	1.3216	93.275 x 10 ⁻⁶	55.00 x 10 ⁻⁶
Pitchblende	NBS	2.0003	1.6963	1.2909	91.108 x 10 ⁻⁶	53.71 x 10 ⁻⁶
Rum Jungle	120	1.8730	1.5883	1.2077	85.236 x 10 ⁻⁶	53.66 x 10 ⁻⁶
Lorado	160	2.0004	1.6963	1.3097	92.435 x 10 ⁻⁶	54.49 x 10 ⁻⁶
Dawn Mining	55	2.0001	1.6961	1.2788	90.254 x 10 ⁻⁶	53.21 x 10 ⁻⁶
Mines Development	108	1.9996	1.6957	1.2555	88.609 x 10 ⁻⁶	52.26 x 10 ⁻⁶

* Net solution weights were corrected by adding 0.0012 grams per gram of solution to compensate for air inleakage to buret while dispensing.

** Flask Number 2 contained 70.577 micrograms U²³³ per gram of solution after correcting for evaporation.

APPENDIX B.

SPIKES PREPARED FROM U²³³ FLASK NO. 4

<u>Sample</u>	<u>Lot No.</u>	<u>Grams of Sample</u>		<u>Grams of Spike</u>		<u>Weight Ratio</u> <u>U²³³/Sample U</u>
		<u>Oxide</u>	<u>Uranium</u>	<u>Solution*</u>	<u>U²³³**</u>	
Homestake	80	2.9999	2.5439	2.4179	138.405 x 10 ⁻⁶	54.41 x 10 ⁻⁶
Rayrock	R-26	3.0009	2.5448	2.3176	132.664 x 10 ⁻⁶	52.13 x 10 ⁻⁶
Faraday	F-103	3.0005	2.5444	2.3813	136.310 x 10 ⁻⁶	53.57 x 10 ⁻⁶
Kermac	14	3.0001	2.5441	2.4416	139.762 x 10 ⁻⁶	54.94 x 10 ⁻⁶
Uranium Reduction	555	3.0002	2.5442	2.3762	136.018 x 10 ⁻⁶	53.46 x 10 ⁻⁶
Calprods	253	2.9986	2.5428	2.5035	143.305 x 10 ⁻⁶	56.36 x 10 ⁻⁶
Climax	291	3.0008	2.5447	2.3293	133.334 x 10 ⁻⁶	52.40 x 10 ⁻⁶
Radium Hill	Z-71	3.0010	2.5448	2.3572	134.931 x 10 ⁻⁶	53.02 x 10 ⁻⁶
Lucky Mc	20	2.9999	2.5439	2.4882	142.430 x 10 ⁻⁶	55.99 x 10 ⁻⁶
Lakeview	16	2.9998	2.5438	2.4721	141.508 x 10 ⁻⁶	55.63 x 10 ⁻⁶
Algom	753	3.0001	2.5441	2.4882	142.430 x 10 ⁻⁶	55.98 x 10 ⁻⁶
Pitchblende	NBS	3.0008	2.5447	2.4219	138.634 x 10 ⁻⁶	54.48 x 10 ⁻⁶
Rum Jungle	124 and 125	3.0005	2.5444	2.5284	144.731 x 10 ⁻⁶	56.88 x 10 ⁻⁶
Lorado	111	3.0002	2.5442	2.3583	134.994 x 10 ⁻⁶	53.06 x 10 ⁻⁶
Dawn Mining	72	3.0004	2.5443	2.4581	140.707 x 10 ⁻⁶	55.30 x 10 ⁻⁶
Mines Development	121	3.0001	2.5441	2.3925	136.951 x 10 ⁻⁶	53.83 x 10 ⁻⁶

* Net solution weights were corrected by adding 0.0012 grams per gram of solution to compensate for air inleakage to buret while dispensing.

** Flask Number 4 contained 57.242 micrograms U²³³ per gram of solution after correcting for evaporation.

APPENDIX C.

SPECTROMETER RATIOS FOR DOMESTIC ORES

Sample	Source	Lot No.	<u>U²³³ Spiking Information</u>		<u>Average Weight Ratio, U²³⁴/U²³³*</u>		
			Flask No.	Weight Ratio U ²³³ /Sample U	Surface Ionization Spectrometer	Gas Analysis Spectrometers	
						No. 4	No. 6
Climax	Grand Junction Colo.	311	2	56.84 x 10 ⁻⁶	0.9357	0.9397	0.9408
		291	4	52.40 x 10 ⁻⁶	1.0252	1.0206	1.0192
Homestake	Ambrosia Lake Dist. Grants, New Mexico	56	2	55.17 x 10 ⁻⁶	0.9735	0.9705	0.9762
		80	4	54.41 x 10 ⁻⁶	0.9915	0.9880	0.9882
Kermac	Ambrosia Lake Dist. Grants, New Mexico	92	2	52.46 x 10 ⁻⁶	1.0144	1.0164	1.0175
		14	4	54.94 x 10 ⁻⁶	0.9738	0.9595	0.9590
Uranium Reduction	Moab, Utah	624	2	51.19 x 10 ⁻⁶	1.0133	1.0198	1.0182
		555	4	53.46 x 10 ⁻⁶	0.9890	0.9845	0.9896
Mines Development	Edgemont, South Dakota	108	2	52.26 x 10 ⁻⁶	0.9516	0.9613	0.9554
		121	4	53.83 x 10 ⁻⁶	0.9368	0.9392	0.9297
Lucky Mc	Fremont County, Wyoming	62	2	50.98 x 10 ⁻⁶	0.9991	1.0005	1.0017
		20	4	55.99 x 10 ⁻⁶	0.9223	0.9229	0.9245
Dawn Mining	Ford, Washington	55	2	53.21 x 10 ⁻⁶	0.9982	0.9999	1.0029
		72	4	55.30 x 10 ⁻⁶	0.9569	0.9574	0.9605
Lakeview	Lakeview, Oregon	2	2	55.63 x 10 ⁻⁶	0.9173	0.9260	0.9201
		16	4	55.63 x 10 ⁻⁶	0.9268	0.9298	0.9286

* Mass Spectrometer ratio times 234/233

APPENDIX D.

SPECTROMETER RATIOS FOR FOREIGN ORES

Sample	Source	Lot No.	<u>U²³³ Spiking Information</u>		<u>Average Weight Ratio, U²³⁴/U²³³*</u>		
			Flask No.	Weight Ratio U ²³³ /Sample U	Surface	Gas Analysis Spectrometers	
					Ionization Spectrometer	No. 4	No. 6
Pitchblende	Belgian Congo, Africa	NBS	2	53.71 x 10 ⁻⁶	1.0025	1.0083	1.0013
		NBS	4	54.48 x 10 ⁻⁶	0.9840	0.9876	0.9893
Calprods	South Africa	142	2	57.33 x 10 ⁻⁶	0.9237	0.9244	0.9279
		253	4	56.36 x 10 ⁻⁶	0.9296	0.9288	0.9308
Radium Hill	Radium Hill, Australia	Z-62	2	53.42 x 10 ⁻⁶	0.9865	0.9824	0.9808
		Z-71	4	53.02 x 10 ⁻⁶	0.9812	0.9900	0.9862
Rum Jungle	Rum Jungle, Australia	120	2	53.66 x 10 ⁻⁶	0.9971	0.9928	0.9956
		124 and 125	4	56.88 x 10 ⁻⁶	0.9411	0.9420	0.9408
Algom	Blind River Area, Ontario, Canada	942	2	55.00 x 10 ⁻⁶	0.9465	0.9556	0.9515
		753	4	55.98 x 10 ⁻⁶	0.9314	0.9378	0.9367
Faraday	Bancroft Area Eastern Ontario	F-93	2	52.72 x 10 ⁻⁶	1.0158	1.0197	1.0151
		F-103	4	53.57 x 10 ⁻⁶	0.9940	1.0007	0.9968
Lorado	Lake Athabaska Area Saskatchewan, Canada	160	2	54.49 x 10 ⁻⁶	0.9723	0.9698	0.9708
		111	4	53.06 x 10 ⁻⁶	1.0013	1.0041	1.0005
Rayrock	Marion River Area, Northwest Territory Canada	R-41	2	52.89 x 10 ⁻⁶	0.9987	0.9962	1.0047
		R-26	4	52.13 x 10 ⁻⁶	1.0131	1.0180	1.0172

* Mass Spectrometer ratio times 234/233.

APPENDIX E.

U²³⁴ CONCENTRATIONS OF DOMESTIC ORES

Sample	Source	Lot No.	U ²³³ Flask No.	U ²³⁴ Concentration in Sample Wt.% U ²³⁴ x 10 ⁻⁴			
				Surface Ionization	Gas Analysis Spect. No. 4	Gas Analysis Spect. No. 6	Weighted Average
Climax	Grand Junction Colo.	311	2	53.2±0.6	53.4±0.5	53.5±0.3	53.4±0.2
		291	4	53.7±0.6	53.5±0.5	53.4±0.3	
Homestake	Ambrosia Lake Dist. Grants, New Mexico	56	2	53.7±0.6	53.5±0.5	53.9±0.3	53.8±0.2
		80	4	53.9±0.6	53.8±0.4	53.8±0.3	
Kermac	Ambrosia Lake Dist. Grants, New Mexico	92	2	53.2±0.9	53.3±0.5	53.4±0.2	53.4±0.2
		14	4	53.5±0.9	52.7±0.5	52.7±0.2	52.7±0.2
Uranium Reduction	Moab, Utah	624	2	51.9±0.4	52.2±0.5	52.1±0.3	52.1±0.3
		555	4	52.9±0.4	52.6±0.7	52.9±0.2	52.9±0.2
Mines Development	Edgemont, South Dakota	108	2	49.7±0.4	50.2±0.5	49.9±0.3	50.0±0.2
		121	4	50.4±0.7	50.6±0.5	50.0±0.3	
Lucky Mc	Fremont County, Wyoming	62	2	50.9±0.6	51.0±0.4	51.1±0.3	51.0±0.3
		20	4	51.6±0.8	51.7±0.4	51.8±0.3	51.7±0.3
Dawn Mining	Ford, Washington	55	2	53.1±0.6	53.2±0.4	53.4±0.3	53.2±0.2
		72	4	52.9±0.6	52.9±0.5	53.1±0.3	
Lakeview	Lakeview, Oregon	2	2	51.0±0.6	51.5±0.5	51.2±0.4	51.3±0.3
		16	4	51.6±0.6	51.7±0.5	51.7±0.4	51.7±0.3

APPENDIX F.

U²³⁴ CONCENTRATIONS OF FOREIGN ORES

Sample	Source	Lot No.	U ²³³ Flask No.	U ²³⁴ Concentration in Sample Wt.% U ²³⁴ x 10 ⁻⁴			
				Surface Ionization	Gas Analysis Spect. No. 4	Gas Analysis Spect. No. 6	Weighted Average
Pitchblende	Belgian Congo, Africa	NBS	2	53.8±0.8	54.2±0.4	53.8±0.3	53.9±0.2
		NBS	4	53.6±0.8	53.8±0.5	53.9±0.3	
Calprods	South Africa	142	2	53.0±0.6	53.0±0.5	53.2±0.3	53.1±0.3
		253	4	52.4±0.5	52.3±0.5	52.5±0.3	52.4±0.3
Radium Hill	Radium Hill, Australia	Z-62	2	52.7±0.6	52.5±0.5	52.4±0.4	52.4±0.2
		Z-71	4	52.0±0.6	52.5±0.5	52.3±0.3	
Rum Jungle	Rum Jungle, Australia	120	2	53.5±0.6	53.3±0.4	53.4±0.3	53.4±0.2
		124 and 125	4	53.5±0.6	53.6±0.7	53.5±0.3	
Algom	Blind River Area, Ontario, Canada	942	2	52.1±0.8	52.6±0.7	52.3±0.3	52.4±0.2
		753	4	52.1±0.7	52.5±0.5	52.4±0.3	
Faraday	Bancroft Area, Eastern Ontario Canada	F-93	2	53.6±0.6	53.8±0.5	53.5±0.3	53.5±0.2
		F-103	4	53.2±0.6	53.6±0.5	53.4±0.3	
Lorado	Lake Athabaska Area, Saskatchewan, Canada	160	2	53.0±0.5	52.8±0.4	52.9±0.3	53.0±0.2
		111	4	53.1±0.5	53.3±0.5	53.1±0.4	
Rayrock	Marion River Area, Northwest Territory, Canada	R-41	2	52.8±0.6	52.7±0.4	53.1±0.3	53.0±0.2
		R-26	4	52.8±0.6	53.1±0.5	53.0±0.4	

APPENDIX G.

U²³⁴ WEIGHTED AVERAGE FROM THREE INSTRUMENTS

Equations 1 and 2 which follow were used to calculate weighted average and symmetrical confidence interval of weighted average respectively.

$$(1) \text{ Weighted Average} = \frac{\frac{x}{LE_x^2} + \frac{y}{LE_y^2} + \frac{z}{LE_z^2}}{\frac{1}{LE_x^2} + \frac{1}{LE_y^2} + \frac{1}{LE_z^2}}$$

$$(2) \text{ LE of Weighted Average} = \sqrt{\frac{1}{\frac{1}{LE_x^2} + \frac{1}{LE_y^2} + \frac{1}{LE_z^2}}}$$

Where: x = Wt.% U²³⁴ from surface ionization spectrometer

y = Wt.% U²³⁴ from spectrometer number 4

z = Wt.% U²³⁴ from spectrometer number 6

LE_x = 95% symmetrical confidence interval for x

LE_y = 95% symmetrical confidence interval for y

LE_z = 95% symmetrical confidence interval for z