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PRECISION OF THE PHOTOGRAPHIC SPECTROCHEMICAL
ASSAY OF URANIUM 235 AT LOW CONCENTRATIONS

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Since nontechnical and nonessential prefatory material
has been deleted, the first page of the report is page 4 .

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Author: T. Lee, A. L. Hallowell and
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ABSTRACT

A series of experiments was carried out to determine the precision of the spectrochemical procedure for the isotopic analysis of uranium, especially at low uranium 235 concentrations. Standards were prepared from uranium hexafluoride which had been assayed on the mass spectrometer with a 95 per cent confidence interval of 0.5 per cent of the amount present.

For a determination consisting of 16 separate measurements, the best precision obtained at normal concentration on the 95 per cent confidence interval was 0.711 ± 0.112 per cent uranium 235 (16 per cent of the amount present).

PRECISION OF THE PHOTOGRAPHIC SPECTROCHEMICAL
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PRECISION OF THE PHOTOGRAPHIC SPECTROCHEMICAL
ASSAY OF URANIUM 235 AT LOW CONCENTRATIONS

This report presents a summary of the experimental work done up to the present in the K-25 Laboratory on the precision of the emission spectrographic procedure for the determination of uranium 235 in samples of urano-uranic oxide, (U_3O_8). This method, if it can be adapted to routine use, is potentially useful as a supplementary procedure to the mass spectrometer and fission counting, particularly for solid and liquid samples which are contaminated with other compounds.

All the work reported here was carried out using photographic recording. A direct reading spectrometer using photomultiplier tubes to give increased precision and speed of analysis has not been tried.

PREVIOUS WORK

The isotope displacement effect in uranium spectra, first reported by Long and Smith (6) has been utilized by these and others (7) at Y-12 to assay the uranium isotopes. Brody and Fred (1) at Argonne National Laboratory and Metz and Marling (8) at Los Alamos Scientific Laboratory have also carried out studies on this procedure.

Two brief reports of some work done by Tolansky are available (11,12) but no data are given on the use of the method for assay purposes.

Norton (9) using data from Los Alamos found that "quadruplicate spectroscopic measurement of uranium 238 permits inference of the uranium 235 percentage within 0.4 per cent absolute at the 95 per cent confidence level." These data from Los Alamos are based on results from the analysis of 24 samples.

EXPERIMENTAL

A Jarrell-Ash spectrograph with a 21 foot-10 inch grating having 15,000 lines per inch was used for this investigation. The samples were excited in a D. C. arc controlled by an automatic current regulator (10). The current for the arc was supplied by a 250 volt motor generator. Photometric measurements were made on a recording Leeds and Northrup microdensitometer. The photographic emulsions were calibrated with a step sector using an iron bead excited in the D. C. arc. Optical densities were converted to light intensities from an emulsion calibration curve. Background corrections were made by subtracting the background intensities from the combined line and background intensities. Standards were prepared from uranium hexafluoride which were analyzed for uranium 235 on the mass spectrometer and then converted to urano-uranic oxide. The concentration of the uranium 235 was known to 0.5 per cent of the amount present. Precision calculations were made on the 95 per cent confidence interval (2). The work was divided into a series of experiments, a description of which follows. The individual intensity ratios are given in the appendix.

Experiment 1

The intensity ratios of the uranium 235 spectral line at 4244.122 Å. to the uranium 238 spectral line at 4244.373 Å. were determined for 7 isotopic standards of urano-uranic oxide. Five determinations were made of each standard. The source was focussed on the collimating mirror by a 45 cm cylindrical lens (axis horizontal) mounted at the entrance slit. The entrance slit was a 50 micron fixed slit. Corning glass filters numbers 3060, 3389, and 4303 were used. The other conditions are given in table I.

TABLE I
CONDITIONS FOR THE ISOTOPIC ANALYSIS OF URANO-URANIC OXIDE,
EXPERIMENTS 1-5

Sample: 2 mg. of urano-uranic oxide

Upper Electrode: 1/8 inch graphite rod, pointed

Lower Electrode: 1/8 inch graphite rod with a sample cavity 3/32 inch diameter and 1/8 inch deep.

Current: 6 amperes

Exposure Time 35 seconds

Photographic Plates: S. A. - 1

A two-step sector having openings of 39 degrees and 13-1/2 degrees was used. The photographic plates were pre-fogged (13). The average intensity ratios calculated by 2 procedures and their 95 per cent confidence intervals are shown in table II.

A working curve (4) in which the intensity ratios are plotted as a function of the per cent uranium 235 is shown in figure 1. This curve was fitted visually.

The curve in figure 1 is not a straight line because the concentration of both uranium 235 and uranium 238 in the standards varies, and the intensity ratios were calculated for uranium 235 to uranium 238. In conventional spectrochemical procedures, the intensity of the lines from the element being determined are compared with the intensity of a line from a standard element the concentration of which is for practical purposes a constant. To offset this variation a curve was plotted using the ratio of the intensity of uranium 235 to the sum of the intensities of uranium 238 and uranium 235. This curve is shown in figure 2.

WORKING CURVE, DATA FROM
TABLE II, 50 MICRON SLIT

FIGURE 1

WORKING CURVE, DATA FROM
TABLE II, 50 MICRON SLIT

FIGURE 2

TABLE II

AVERAGE INTENSITY RATIOS AND THEIR 95% CONFIDENCE INTERVALS
USING 50 MICRON FIXED SLIT

(Average of 5 measurements)

% U-235	$\frac{I_{4244.122}}{I_{4244.373}} \pm 95\% \text{ Confidence Interval}$	$\frac{I_{4244.122}}{I_{4244.122} + I_{4244.373}} \pm 95\% \text{ Confidence Interval}$
14.94	0.197 $\pm$ 0.006	0.165 $\pm$.0043
27.11	0.339 $\pm$ 0.011	0.253 $\pm$.0062
49.62	1.01 $\pm$ 0.02	0.502 $\pm$.0047
72.37	2.86 $\pm$ 0.09	0.741 $\pm$.0064
83.32	4.54 $\pm$ 0.08	0.819 $\pm$.0026
88.38	7.05 $\pm$ 0.21	0.876 $\pm$.0033

Discussion of Experiment 1

Because the intensity difference between the uranium 235 line at 4244.122 A. and the uranium 238 line at 4244.373 A. at isotopic concentrations below 20% uranium 235 and above 30% uranium 235 is greater than the range over which precise photographic measurements of intensity can be made, it is necessary to decrease the intensity of the stronger spectral line by a fixed amount so that both lines may be recorded simultaneously. A step sector was used for this purpose. This technique requires that the line intensity be uniform from top to bottom, and a test for line uniformity indicated that only the 50 micron fixed slit originally supplied with the instrument gave reasonably uniform lines. Using the 50 micron slit, intensity ratios of the isotopic spectral lines were measured from standards with 15 to 93 per cent uranium 235. At concentrations below 15 per cent uranium 235 it was necessary to improve the resolution of the lines so that there was no interference from the uranium 238 line at 4244.373 A. on the uranium 235 line at 4244.122 A. due to broadening of the uranium 238 line with increasing concentration of uranium 238.

Experiment 2

An "optical" slit (5) formed by a diminished image of an aperture placed outside the spectrograph and shown in figure 3A was used in place of the 50 micron slit. Intensity ratios of the isotopic lines at 4244.122 A. (uranium 235) and at 4244.373 A. (uranium 238) were measured for ten standards. Ten measurements were made of each standard. An 8-step sector having a ratio of 1.585 between steps was used. For each isotopic level, the average ratio and its 95 per cent interval is shown in table III. A working curve in which the ratios are plotted as a function of the per cent uranium 235 is shown in figure 4.

A

B

OPTICAL SLIT

FIGURE 3

WORKING CURVE, DATA FROM TABLE III, USING OPTICAL SLIT

FIGURE 4

TABLE III
AVERAGE INTENSITY RATIOS AND THEIR 95% CONFIDENCE
INTERVALS USING OPTICAL SLIT A
(Average of 10 measurements)

	$I_{4244.122}$	$\pm 95\%$ Confidence Interval
<u>% U-235</u>	<u>$I_{4244.122} + I_{4244.373}$</u>	
0.713	0.0153	± 0.00084
3.069	0.0371	± 0.0019
4.969	0.0613	± 0.0048
14.94	0.170	± 0.0056
27.11	0.296	± 0.0065
49.62	0.495	± 0.0034
72.37	0.706	± 0.0115
83.32	0.806	± 0.0102
88.38	0.853	± 0.0095

Discussion of Experiment 2

Using the optical slit described by King, in which a diminished image formed by a short-focal-length cylindrical lens (axis vertical) of an aperture illuminated by the source was used to replace the regular spectrographic slit, it was found that not only could spectral lines of any practical width be obtained but that the lines were acceptably uniform in intensity along their entire length. The optical arrangement used was improvised from available components, and while not ideal, it permitted evaluation of the isotopic intensity ratios of standards down to the 0.713 per cent uranium 235 level.

Experiment 3

Using a 10 micron mechanical entrance slit, two groups of ratios were obtained from four standards. The first group was based on a measurement of the intensity ratios of the uranium 235 spectral line at 4244.122 Å. to the sum of the uranium 238 spectral line at 4244.373 Å. and the uranium 235 spectral line at 4244.122 Å. A step sector was used to reduce the intensity. The second group of ratios was based on a measurement of the intensity ratios of the uranium 235 spectral line at 4244.122 Å. to a "non-splitting" uranium line at 4243.557 Å. without the use of a step sector. The intensities of the 4244.122 isotopic line and of the 4243.557 "non-splitting" line are of the same order of magnitude for the standards used in this experiment. Ten ratios were obtained for each group. Crossed cylindrical lenses (3) were used. A 60 cm. cylindrical lens, axis horizontal, was used to focus the source on the collimating mirror. A diaphragm was placed at the mirror to mask out the glowing electrodes. In the horizontal plane, a 15 cm. cylindrical lens, axis

vertical, was used to focus the source on the slit. A Corning glass filter number 4308 was used. The average ratios and their 95 per cent intervals are given in table IV.

TABLE IV

AVERAGE INTENSITY RATIOS AND 95% CONFIDENCE INTERVALS USING
10 MICRON FIXED SLIT

(Average of 10 measurements)

% U-235	$I_{4244.122}$	$I_{4244.122}$
	$I_{4244.122} + I_{4244.373}$ ± 95% Confidence Interval Using a Step Sector	$I_{4243.557}$ ± 95% Confidence Interval without a Step Sector
0.4995	0.0135 ± 0.0008	0.424 ± 0.018
0.713	0.0169 ± 0.0009	0.532 ± 0.013
1.208	0.0205 ± 0.0011	0.640 ± 0.022
3.069	0.0457 ± 0.0040	1.423 ± 0.020

Experiment 4

The optical slit as shown in figure 3B was used in place of the 10 micron fixed slit and experiment 3 was repeated without changing any of the other conditions. The results obtained are shown in table V.

TABLE V

AVERAGE INTENSITY RATIOS AND 95% CONFIDENCE INTERVALS
USING OPTICAL SLIT B

(Average of 10 measurements)

% U-235	$I_{4244.122}$	$I_{4244.122}$
	$I_{4244.122} + I_{4244.373}$ ± 95% Confidence Interval Using a Step Sector	$I_{4243.557}$ ± 95% Confidence Interval without a Step Sector
0.4995	0.0174 ± 0.0016	0.460 ± 0.016
0.713	0.0203 ± 0.0010	0.542 ± 0.005
1.028	0.0256 ± 0.0021	0.640 ± 0.019
3.069	0.0550 ± 0.0035	1.455 ± 0.010

Discussion of Experiments 3 and 4

A new set of fixed slits, including a 10 micron slit, was obtained. These slits were found to produce spectral lines that were acceptably uniform along their length. Additional optical components were also received so that a more efficient optical slit could be set up. It was decided to compare the precision obtained with the 10 micron fixed slit with that obtainable with the optical slit. Accordingly two independent sets of measurements were made, one using the 10 micron mechanical slit and the other using the optical slit. Using the two isotopic lines at 4244.122 Å. (uranium 235) and 4244.373 Å. (uranium 238), the precision was found to be approximately the same for both slits (tables IV and V).

Intensity ratios were then obtained of the isotopic uranium 235 spectral line at 4244.122 Å. to a "non-splitting" uranium line at 4243.557 Å. These measurements were made on the same photographic plates used in the earlier part of this experiment. Ratios for both the optical and the 10 micron slit were obtained. The statistical evaluation again showed that the precision obtained with the 10 micron fixed slit and with the optical slit was about the same. However, an appreciable improvement was found in the precision obtained with the ratios of the isotopic line at 4244.122 Å. (uranium 235) to the "non-splitting" line at 4243.557 Å. using no intensity reducing device as compared with the ratios obtained using the two isotopic lines at 4244.122 Å. (uranium 235) and 4244.373 Å. (uranium 238) and a step sector, (tables IV and V, column 3). This improvement is thought to be due to the elimination of the step sector and any effect due to non-uniformity of slit illumination.

Experiment 5

The intensity ratios of 5 isotopic standards ranging from 0.4995 per cent to 1.527 per cent uranium 235 were measured using the isotopic spectral line at 4244.122 Å. (uranium 235) to the "non-splitting" spectral line at 4243.557 Å. Ten measurements were made of each standard using the optical slit arrangement shown in figure 3B. The other conditions are given in table I except that 10 amperes were used instead of 6 amperes. The average ratios and a statistical evaluation of these ratios are given in table VI. A working curve using linear coordinates fitted by the method of least squares in which the average ratios are plotted as a function of the per cent uranium 235 is shown in figure 5. The 95 per cent confidence interval of the line is also shown in figure 5.

WORKING CURVE, DATA FROM TABLE VI, OPTICAL SLIT B

FIGURE 5

TABLE VI

AVERAGE INTENSITY RATIOS AND 95% CONFIDENCE INTERVALS
FOR THE RANGE 0.5 TO 1.5% URANIUM 235

(Average of 10 measurements)

% U-235	$\frac{I_{4244.122}}{I_{4243.557}} \pm 95\% \text{ Confidence Interval}$
	without a Step Sector
0.4995	0.504 $\pm$ 0.023
0.7115	0.566 $\pm$ 0.011
1.028	0.686 $\pm$ 0.009
1.216	0.753 $\pm$ 0.024
1.527	0.884 $\pm$ 0.009

Discussion of Experiment 5

On examining the intensity ratio data from experiments 2, 3, and 4 using the 4244.122 Å. and the 4244.373 Å. isotopic uranium lines at the normal level the precision found in all three cases was approximately the same. This suggested the desirability of preparing a working curve using a much narrower range of concentrations. Accordingly, experiment 5 was carried out using a concentration range of uranium 235 from 0.5 to 1.5 per cent. The working curve shown in figure 5 was used to determine the precision attainable with this procedure at each concentration. The results are shown in table VII.

TABLE VII

EXPECTED PRECISION WHEN USING WORKING CURVE
(Figure 5)

Percent U-235 (from Mass Spectrometer)	95% Confidence Interval) (from Emission Spectrograph)
0.4995	$\pm$ 0.114 or 23%
0.7115	$\pm$ 0.112 or 16%
1.028	$\pm$ 0.119 or 12%
1.216	$\pm$ 0.126 or 10%
1.527	$\pm$ 0.147 or 10%

It is noted that the precision reported for the average of the individual ratios shown in table VI is appreciably better than that shown in table VII. This apparent anomaly is due to the fit of the working curve to the entire group of points. The precision of any point on the working curve based upon the goodness of fit of the line is less than the

reproducibility of the individual ratios. Nevertheless, if the line is to be used as the basis for estimating the concentration of an unknown sample, the precision of the estimate is necessarily based on the fit of the line itself.

This precision is believed to be near the upper limit obtainable with the procedure and equipment now being used. By increasing the number of determinations on each sample, the precision can be improved. By using very carefully prepared standards, and by working in a very narrow range of concentrations with standards having concentrations on either side of the unknown, the accuracy can also be improved.

SUMMARY

Using a Jarrell-Ash 21 foot spectrograph in the second order of a 6 inch, 15,000 line per inch grating, and D. C. arc excitation of the spectra, 5 experiments were carried out to determine the 95 per cent confidence interval of the spectrochemical assay method for uranium 235. Standards were prepared as urano-uranic oxide from uranium hexafluoride which had been assayed with the mass spectrometer. The concentration range studied extended from 0.5 to about 90% uranium 235 but most effort was directed toward determining the precision at low concentrations. From 5 to 10 replicates were made on each standard.

The best precision obtained at normal concentration was 0.711 ± 0.112 per cent uranium 235 for a determination consisting of 10 separate measurements.

Marked improvement in the precision should be obtainable with an instrument especially designed for this work and employing an improved light source, together with a dispersing element of high resolution and dispersion, as well as electronic recording with photomultiplier tubes.

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Experimental work was completed November 1, 1950.

APPENDIX

INDIVIDUAL INTENSITY RATIOS FOR EXPERIMENTS 1 TO 5

TABLE A-1

INTENSITY RATIOS USING 50 MICRON FIXED SLIT

(Experiment 1)

% U-235:	88.38	83.32	72.37	49.62	27.11	14.94
	7.11	4.64	2.75	1.02	0.328	0.191
	6.82	4.53	2.84	1.00	0.333	0.196
	6.94	4.53	2.90	1.02	0.342	0.195
	7.20	4.50	2.93	0.99	0.351	0.201
	7.20	4.48	2.90	1.01	0.340	0.203
Average	7.05	4.54	2.86	1.01	0.339	0.197
	0.877	0.823	0.733	0.505	0.247	0.160
	0.872	0.819	0.740	0.500	0.250	0.164
	0.874	0.819	0.744	0.505	0.255	0.163
	0.878	0.818	0.746	0.497	0.260	0.167
	0.878	0.818	0.744	0.502	0.254	0.169
Average	0.876	0.818	0.741	0.502	0.253	0.165

TABLE A-2

INTENSITY RATIOS USING OPTICAL SLIT A

(Experiment 2)

% U-235:	0.713	3.069	4.969	14.94	27.11	49.62	72.37	83.32	88.38
	0.0153	0.0421	0.0632	0.178	0.283	0.500	0.673	0.798	0.835
	0.0163	0.0386	0.0664	0.175	0.288	0.505	0.698	0.805	0.870
	0.0170	0.0377	0.0724	0.174	0.294	0.495	0.713	0.798	0.839
	0.0129	0.0326	0.0572	0.170	0.291	0.495	0.715	0.819	0.850
	0.0162	0.0372	0.0641	0.175	0.314	0.495	0.686	0.814	0.861
	0.0155	0.0385	0.0677	0.163	0.292	0.487	0.719	0.808	0.861
	0.0155	0.0357	0.0491	0.151	0.296	0.492	0.708	0.807	0.835
	0.0152	0.0380	0.0564	0.171	0.295	0.495	0.718	0.787	0.860
	0.0148	0.0364	0.0583	0.169	0.304	0.492	0.724	0.817	0.853
	0.0139	0.0340	0.0581	0.173	0.305	0.395	0.702	0.808	0.870
Average	0.0153	0.0371	0.0613	0.170	0.296	0.495	0.706	0.806	0.853

TABLE A-3
INTENSITY RATIOS USING 10 MICRON FIXED SLIT
(Experiment 3)

% U-235:	0.713	1.028	3.069
	0.0181	0.0195	0.0404
	0.0175	0.0208	0.0438
	0.0156	0.0211	0.0418
	0.0176	0.0195	0.0468
	0.0174	0.0228	0.0470
	0.0142	0.0194	0.0401
	0.0160	0.0215	0.0459
	0.0172	0.0225	0.0570
	0.0173	0.0189	0.0529
	0.0177	0.0187	0.0466
Average	0.0169	0.0205	0.0457
	0.532	0.642	1.41
	0.516	0.636	1.40
	0.538	0.641	1.40
	0.490	0.660	1.47
	0.549	0.661	1.39
	0.527	0.690	1.47
	0.543	0.557	1.42
	0.537	0.640	1.42
	0.534	0.662	1.42
	0.552	0.645	1.43
Average	0.532	0.640	1.42

TABLE A-4
INTENSITY RATIOS USING OPTICAL SLIT B
(Experiment 4)

% U-235:	0.4995	0.713	1.028	3.069
	0.0206	0.0235	0.0250	0.0562
	0.0182	0.0204	0.0233	0.0578
	0.0148	0.0219	0.0275	0.0590
	0.0189	0.0196	0.0231	0.0519
	0.0143	0.0212	0.0229	0.0548
	0.0179	0.0198	0.0308	0.0562
	0.0156	0.0194	0.0244	0.0581
	0.0154	0.0188	0.0302	0.0618
	0.0199	0.0181	0.0255	0.0482
Average	0.0174	0.0203	0.0256	0.0550
	0.471	0.547	0.662	1.47
	0.442	0.535	0.669	1.46
	0.471	0.547	0.652	1.45
	0.452	0.535	0.620	1.43
	0.463	0.539	0.605	1.44
	0.500	0.546	0.658	1.45
	0.452	0.532	0.661	1.46
	0.422	0.550	0.596	1.48
	0.441	0.550	0.667	1.45
	0.480	0.544	0.655	1.46
Average	0.460	0.543	0.641	1.46

TABLE A-5

INTENSITY RATIOS FOR RANGE 0.5 TO 1.5% URANIUM 235
(Experiment 5)

% U-235:	0.4995	0.7115	1.028	1.216	1.527
	0.530	0.573	0.684	0.744	0.875
	0.507	0.587	0.658	0.734	0.864
	0.495	0.584	0.701	0.786	0.893
	0.500	0.539	0.682	0.774	0.895
	0.456	0.552	0.686	0.700	0.891
	0.504	0.568	0.696	0.745	0.880
	0.474	0.549	0.693	0.816	0.883
	0.476	0.566	0.682	0.721	0.864
	0.565	0.571	0.678	0.738	0.897
	0.534	0.575	0.696	0.775	0.896
Average	0.504	0.566	0.686	0.753	0.884