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Semi-Quantitative Uranium Analysis by X-Ray Spectrography

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SEMI-QUANTITATIVE URANIUM ANALYSIS BY X-RAY SPECTROGRAPHY

I N T R O D U C T I O N

In the past the Laboratory has used only the radiation counter to estimate uranium. The counter is sensitive to radiation from sources other than uranium and so the estimate was one of "equivalent uranium" (eU). There has existed a need for a method for the determination of uranium per se. This investigation was designed to that end.

E X P E R I M E N T A L

Standards were formed using analytical reagent grade uranium nitrate hexahydrate ($UO_2(NO_3)_2.6H_2O$) as the uranium source. The matrix material into which the uranium nitrate was added is a very fine grained coal ash. The coal ash was chosen to represent a nominal rock composition.

A 10 gram sample of coal ash containing enough uranium nitrate to give the final composition of 2.00 weight per cent uranium was first compounded. The sample was thoroughly ground and then checked for uranium homogeneity. A series of dilutions were made to form standards at 2.00%, 1.00%, 0.500%, 0.250%, 0.125%, and 0.060% U. Those powders were pressed into Spec-Caps at 24,000 psig to form permanent standards.

The SrK_a line (25.09°2θ) occurs fairly near the U_{La} line (26.14°2θ) but is readily resolved. The Hg_{La} line at 25.73°2θ may offer an interference as could possibly the Hf_{Ka2} at 26.06°2θ. The Hf line would be weak. It and the Hg line could be eliminated by PHA or voltage control. The weak Pb_{LB9} at 26.61° could possibly interfere but it too could be eliminated by PHA.

The U_{La} line used for this analysis has a wave length of 0.910Å. Therefore, the elements Cu, Ni, Co, Fe, Mn, and Cr (in decreasing order of importance) would be strong matrix absorbers. No system of correction or control for those matrix affects are incorporated into this analysis. Error would result from the presence of a considerable amount of any of those elements. Strontium has been used as an internal standard for matrix control. However, strontium is so frequently a constituent of rocks it would be unsafe to use it routinely. Bromine could possibly be used, except the Br K_B interferes with the U_{La}. Br internal standard, using a very low Br concentration, could possibly be used but it was not investigated. This being a semi-quantitative analysis, I elected to accept the error caused by matrix affects, knowing some samples

(high in interfering elements) could not be analyzed by this procedure. This procedure incorporates some matrix correction by using the peak to background intensity ratios rather than simply the peak intensity. The x-ray intensity data obtained on the prepared standards are shown on table 1.

The data were obtained by scanning rather than by fixed 2θ (because of temporary x-ray machine mechanical problems). The 0.00% standard is the coal ash without any addition of uranium nitrate. There was evidence of a trace of U in the coal ash and that is probably the reason for the 0.20 ratio at the 0.00% U level. The analytical line formed from the data of table 1 is nearly straight with some upward tailing below 0.1% U.

The precision of the analysis was tested at 0.25% U and at 0.066% U. Using five specimens for each test the coefficient of variation was 4% of the value at 0.25% U and 27% of the value at 0.066% U. The latter value is near the detectability limit and so 27% of the value is acceptable precision. The analytical limit is estimated to be about 0.03% U and the detectability limit is estimated to be between 0.01 and 0.005% U. The 0.066% sample is a specimen from our "counting standards" that was labled by the supplier to contain 0.1% U. The stated figure is probably eU rather than per cent U. All the other "counting standards" were also found to contain about one half the stated uranium values and are probably eU values. The samples that were used in the precision test, in addition to one other of the "counting standards", will be sent out for analysis for the purpose of comparison.

Table 1

X-RAY INTENSITY DATA ON THE U_{La} LINE

SAMPLE	PEAK INTENSITY	BACKGROUND INTENSITY @ 27.5°2θ	INTENSITY RATIO $\frac{\text{PEAK-BACKGROUND}}{\text{BACKGROUND}}$
2.00	8,600	600	13.3
1.00	6,000	600	9.00
0.50	3,700	600	5.17
0.25	2,200	600	3.66
0.125	1,460	600	1.43
0.06	1,080	600	0.80
0.00	720	600	0.20