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QUANTITATIVE ANALYSIS OF URANIUM IN ORE DEPOSIT BY CALIBRATION STANDARDIZATION OF XRF

P. ARIKAN and A. ÖZMEN*

Ankara Nuclear Research and Training Center Beşevler-Ankara, Turkey

*Physics Department, Gazi University, Beşevler-Ankara, Turkey

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ARSTRACT

The X-ray fluorescence method was studied for uranium amount determination in uranium deposit from Saskatchewan area of Canada. The ^{57}Co and ^{109}Cd radioisotope sources were employed to excite the characteristic K and L lines of uranium respectively. The calibration method was supplemented by the thin film, dilution and scatter methods with respect to the selection of either K and L lines of analyte.

INTRODUCTION

X-Ray fluorescence is the most widely used technique for the analysis of uranium in geological samples. Properly chosen method can also compensate for the matrix effects. The direct comparison method involves comparing the intensity of X-ray line of samples with standards having the same geometry, same physical form and matrix composition.

X-ray fluorescence technique was applied by several authors for quantitative determination of uranium in various samples. Klecka (1966) has reported uranium results by using ^{125}I source with a Si spectrometer. Karttunen and Harmon (1969) have analyzed uranium in the range 8-20 mg U / ml. Shenberg and Amiel (1971) have studied analytical significance of uranium peaks in X-ray fluorescence analysis. More recently, X-ray fluorescence analysis of uranium employing another analytical techniques has been reported by Arıkan and Özmen (1986, 1987) such as filter, internal standard and Compton profile normalization methods.

The method has the capability for rapid and accurate repetitive analysis of uranium bearing samples of ore deposit. In present study, the method was based on the measurement of the uranium K-X rays and

L-X rays. Elemental calibration constants and calibration factors for uranium lines were determined experimentally using International Atomic Energy Agency (IAEA) and Canada Centre for Mineral and Energy Technology (CANMET) standards of known total composition.

MATERIAL and METHOD

An energy-dispersive X-ray fluorescence method has been applied for the direct determination of uranium in RL-1 and BL-3 uranium ores from Canada. BL-3 is a sample of uranium from Beaverlodge area of northwestern Saskatchewan, is relatively free of thorium and in secular equilibrium. Uranium characteristic L-X rays were excited in the BL-3 ore sample using 22.1 keV radiation from ^{109}Cd source. If exciting radiation is monoenergetic, line is given following equation (Adler, 1970).

$$I_A = \frac{I_0 k \operatorname{cosec} \theta_1 \, (1 - e^{-\alpha \rho d})}{\alpha \rho} \qquad (1)$$

where,

I_A	intensity of characteristic line of element
k	a proportionality constant
I_o	the intensity of the incident radiation
α	$\mu_1 \operatorname{cosec} \theta_1 + \mu_2 \operatorname{cosec} \theta_2$
μ_1, μ_2	the mass absorptions for the incident radiation of energy E_1, and the characteristic radiation E_2
ρ	the density of the sample
d	the sample thickness
θ_1, θ_2	the incident and emerging angles

A lithium drifted silicon detector with a 80 mm^2 active area was used for detection of uranium L-X rays. The detector was coupled through a preamplifier and a spectroscopy amplifier to 4096 channel analyzer (CANBERRA Industries). During the experiments, the resolution (FW HM) was about 200 eV at Mn Kα (5.9 keV). The ore samples and standards, supplied by CANMET-Canada were prepared in thin region to minimize the matrix effects. 50 mg amount of ore powder was used and 300 mg cellulose was added as a binder. In order to avoid a possible particle size effect of ore powder was sieved to about 200 mesh. The powder

samples were pressed in diameter 32 mm at 25 ton pressure. The linear calibration curve for ULα by using uranium ore standards of CANMET is shown in Fig. 1.

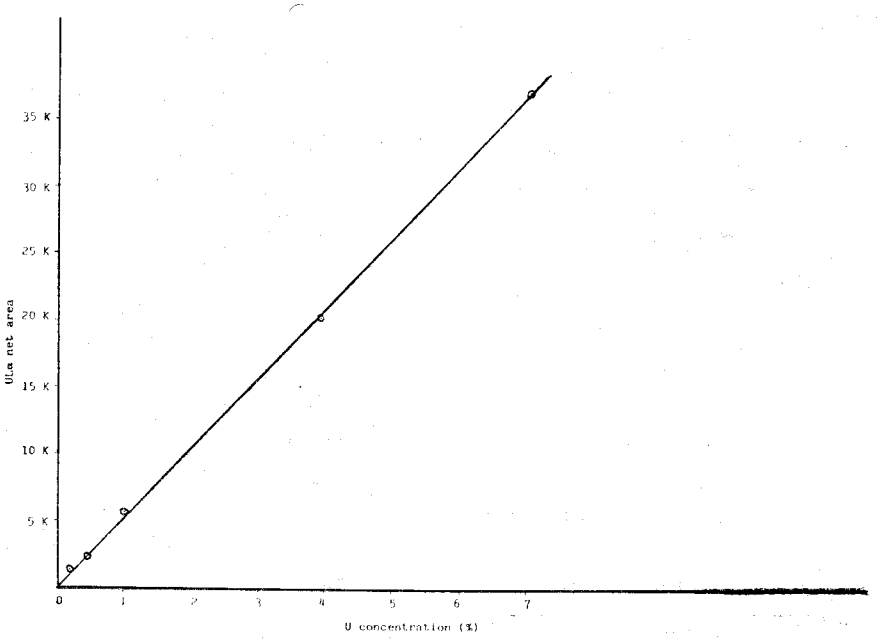

Fig 1. Standard calibration curve for BL-3 uranium ore

RL-1 is a sample of uranium ore from Rabbit Lake, Saskatchewan. RL-1 was ground to -74 μm, blended and analyzed for homogeneity of powder ore in CANMET. Five separate subsamples were prepared for RL-1 on an asreceived basis. For each sample, 5 g powder ore and 1 g cellulose were mixed in pulveriser. The homogeneous mixture was then briqueted in a press at 25 tons. Samples of uranium bearing ores were excited by primary radiation 122.1 keV gamma rays from the ^{57}Co annular source. Ge (Li) semiconductor detector was used to detect the uranium K-X rays from thick samples. The calibration curve of IAEA uranium ore standards was evaluated by the least squares method which is given in Fig. 2. The spectrum of L-X rays of BL-3 ore sample is illustrated in Fig. 3.

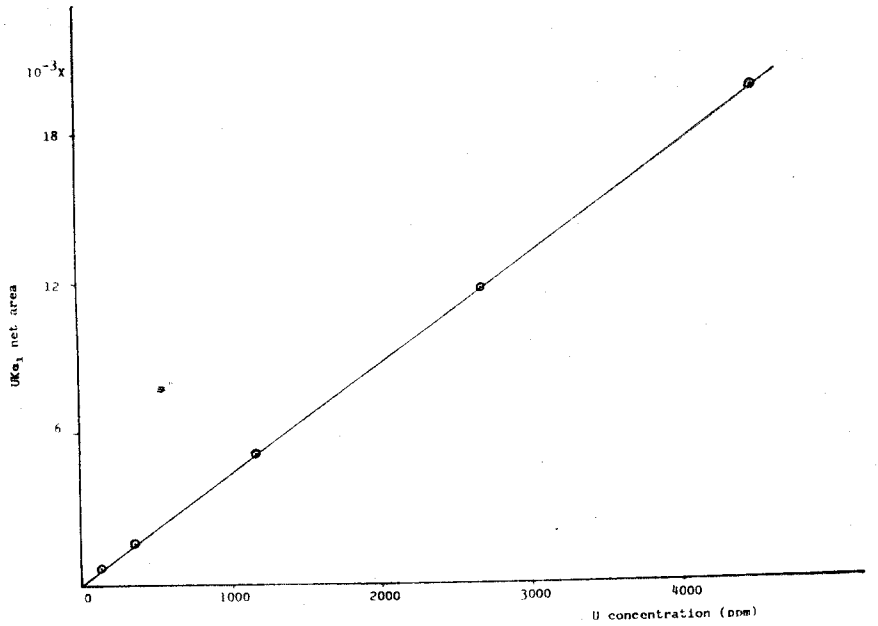

Fig 2. Standard calibration curve for RL-1 uranium ore

RESULTS and DISCUSSION

Radioisotope excited X-ray fluorescence was employed for the analysis of uranium in uranium bearing ores from Canada. The calculation of uranium concentration was performed by the calibration standardization. The results of measured uranium concentrations by using CANMET and IAEA standards were summarized in Table 1. As can be seen from Table 1, good agreement was obtained between the results of XRF and

Table 1 Analysis of uranium bearing ores

Sample	RL-1 U, ppm (XRF)	BL-3 % U (XRF)
1	2000	1.03
2	2000	1.01
3	2003	1.02
4	1998	1.02
5	1998	1.02
CANMET	2010	1.02

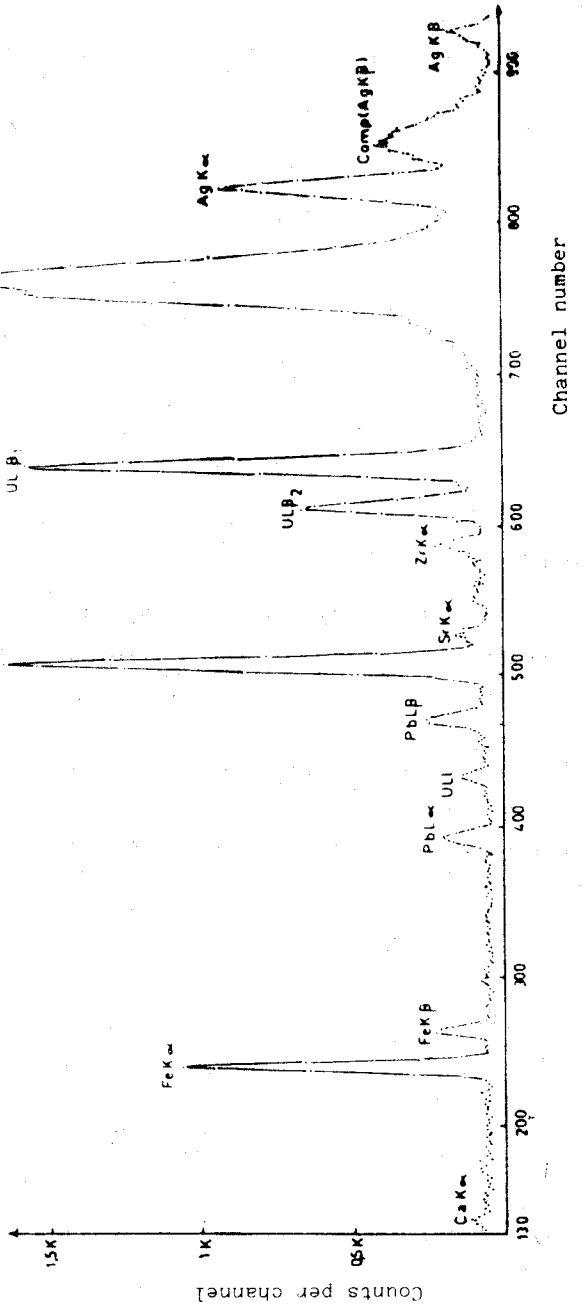

Fig 3. The spectrum of BL-3 ore sample

CANMET.

The calibration method was supplemented by thin film, dilution and scatter methods with respect to the usage of L and K-X rays of uranium respectively. The method was carefully conducted and that all significant corrections for analyte peak were applied. In thin film method, the samples were prepared so thin that absorption-enhancement effects substantially disappear. Another method, standardization with scattered photons was used to compansate the matrix effects that appear in thick sample. In dilution method, the ore samples were diluted in an excess of cellulose. This minimized the variations in matrix composition and tended to make the fluorescence intensities nearly proportional to concentrations. The results of the experiments described clearly show the capability of radioisotope excited x-ray fluorescence method for the analysis of uranium in uranium bearing ores.

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