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SHORT-TERM STABILITY TEST FOR URANIUM SOIL CANDIDATE A REFERENCE MATERIAL

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ABSTRACT

This work describes a methodology to determine the soil short-term stability after the steps of production in laboratory. The soil is a candidate of reference material for chemical analysis of uranium with metrological traceability to be used in environmental analysis, equipment calibration, validation methods, and quality control. The short-term stability of the soil is an essential property to be determined in order to producing a reference material. The slopes and their uncertainties were obtained from the regression lines at temperatures of 20 ^{o}C and 60 ^{o}C, the control temperature was -20 ^{o}C and t-test was applied. At 20 ^{o}C the t-value obtained was 0.34 and the critical value was 2.78. At 60 ^{o}C the t-value was 1.19 and the critical value was 3.18. Since in both cases the calculated t-value is lower than the critical value, we can conclude with 95% confidence level that no significant changes happened during the period studied concerning uranium concentration in soil at temperatures of 20 ^{o}C and 60 ^{o}C, showing stability at these temperatures. This paper describes one of the steps of the procedures to produce a soil reference material to the analite U.

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1. INTRODUCTION

The Metrology Laboratory (LNMRI) of Instituto de Radioproteção e Dosimetria (IRD) has a long tradition in preparing matrix reference materials and conducting laboratory intercomparison studies. In recent years the IRD has also begun to regularly conduct a proficiency test "Programa Nacional de Intercomparação" (PNI). The PNI is conducted to support laboratories in Brazil involved in radionuclide analysis of environmental samples. They are involved in various fields of application and cover a wide range of objectives of regional and interregional projects. To cover the vast range of requirements and the increasing demand for PNI, the Reference Materials Group at the IRD's laboratory, uses either natural matrix reference materials or matrix materials spiked with calibrated standard solutions as reference material. Characterization of natural matrix reference materials is expensive and time-consuming. Despite all efforts, the availability of matrix reference materials remains limited and it is not always possible to meet the target criteria for a specific proficiency test, e.g. type of matrix, concentration of analytes, total combined uncertainty associated with the target values. Brazil has large deposits of uranium and the radio analytical procedures to determine uranium concentration in natural matrices is critical to an accurate assessment of the content of this element in the various minerals of interest. The environmental control of the radionuclides concentration in the vicinity of uranium mines is another area which also requires reliable measurements and standards. Radio analytical

determinations require standards not only for instruments calibration but also for evaluation of the analysis performance. Therefore the production of a uranium soil reference material is extremely important in the chemical analysis of this element.

2. MATERIALS AND METHODS

2.1 Sample collection and preparation

About 600 kg of wet material were collected to a depth of around 50 cm from the ground on a farm in Pocos de Caldas, Minas Gerais, coordinates 21 "55 '47.9 S, 046' 35 '13.2 N. The collected soil was taken to IRD for Reference Materials Laboratory / LNMRI and was passed by several steps. The raw soil material was initially clean by hands with the removal of stones, roots, leaves and other macroscopic impurities. The material was dried in a laboratory oven with air flow at 50^0C overnight. The material was sieved to discharge all the parts bigger than 3mm. The presence of small pieces of vegetables mixed in the soil lead the procedure to the elimination of organic matter by calcination in a furnace at 450^0C during 24 hours. The soil free of organic matter was milled in a Ball Mill (PM400 Retsch Gmbh), passed through a 75-µm sieve, homogenized, submitted to automatic bottles filling and sterilized with ^{60}Co gamma rays. The total material obtained after this process was 120 kg.

Figure 1. Ball Mill

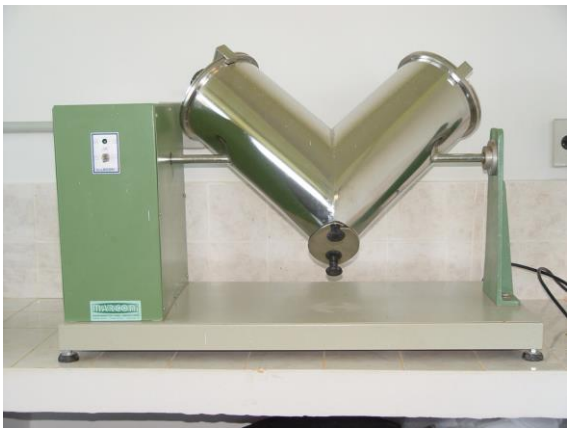

Figure 2. Type “V” Homogenizer

Figure 3. Sieve system

Figure 4. The soil final package

2.2 Uranium concentration analysis

The test performed in this study used neutron activation analysis for uranium determination in soil samples. The vials containing soils were chosen randomly. For short-term stability the uranium concentration was measured periodically at temperatures of 20° C and 60°C and the reference temperature of -20°. The neutron activation analysis of the samples and reference standard materials were conducted in a nuclear research reactor IEA-R1 of IPEN / CNEN-SP by 8 hours of irradiation under a thermal neutron flux range in the order of 6.5-4.5 x 10^{12} n cm^{-2} s^{-1}. The measures of induced gamma activity were made with POP TOP model hyperpure germanium detector of EG & G ORTEC, with a resolution of 1.90 keV for peak 1332.49 keV 60Co, coupled to an associated electronics.

3. RESULTS

Three control samples were kept at -20°C and then analyzed. The average uranium concentration measured was 69.7 mg / kg with a standard deviation of 2.4 mg / kg. The samples to determine the short term stability were maintained in a constant temperature of 20^0C in a controlled weighting room with air conditioning system, and 60^0C in a laboratory oven. The samples were measured in time intervals of 7, 14, 21, 28 and 60 days. Table 1 shows the obtained values of uranium concentration (mg/kg).

Table 1. Uranium concentration (mg/kg) at 20^0C and 60^0C

Days	Temperature (^{0}C)	
	20	60
7	71.3 ± 0.3	65.5 ± 0.2
14	71.0 ± 0.4	68.7 ± 0.4
21	65.5 ± 0.4	66.5 ± 0.4
28	66.0 ± 0.3	-
60	69.6 ± 0.3	68.9 ± 0.3

The uranium concentrations determined in soil samples obtained at 20°C and 60°C were divided by the sample concentrations obtained at -20°C (control sample) to give Qt ratios. Q_T values were plotted against time, as shown in figures 5 and 6.

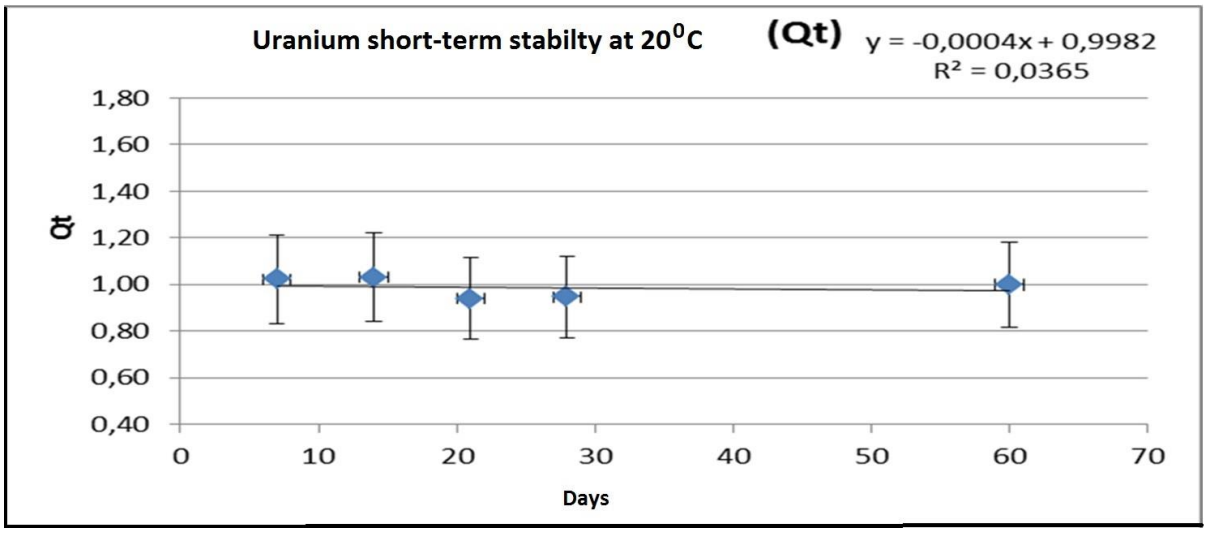

Figure 5. Ratio Qt versus time at 20°C

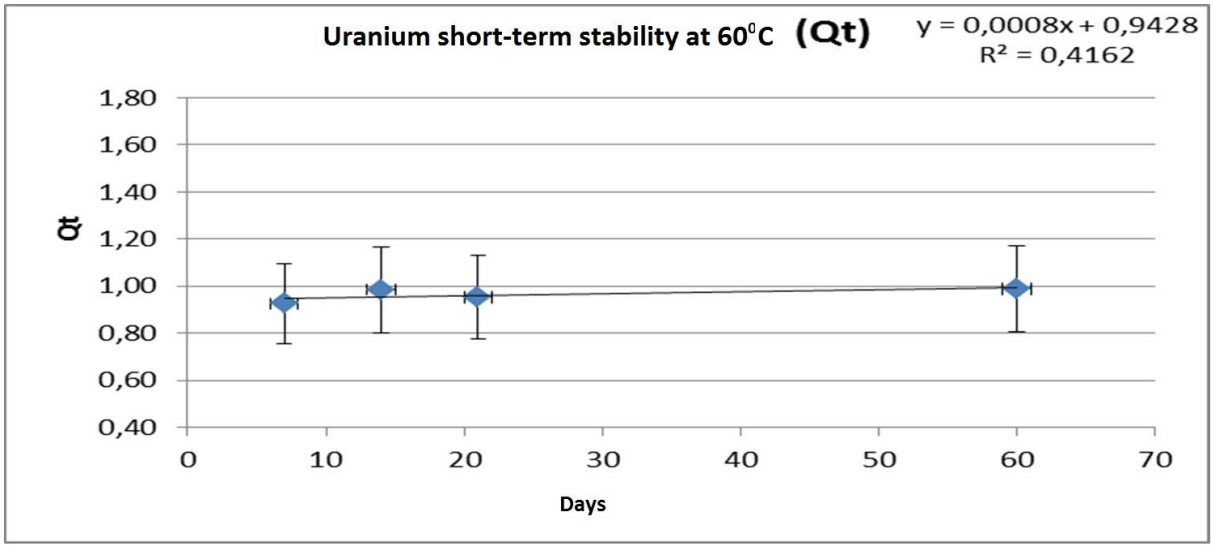

Figure 6 . Ratio Qt versus time at 60°C

The slopes are calculated by the equation:

$y = -0.0004 x + 0.9982$ ($T = 20^0C$) and

$y = 0.0008 x + 0,9428$ ($T = 60^0C$)

3. CONCLUSION

Using the slope and their uncertainties obtained from the regression lines at temperatures of 20 ^{o}C and 60^{o}C the t test was applied. At 20° C was obtained t equal to 0.34 and the value of t critical was 2.78. At the temperature of 60°C t value was 1.19 with the t critical value of 3.18. Since, in both cases, the calculated value of t is under the critical value, we can conclude that with 95% confidence level no significant changes in concentrations of U during the studied time in temperatures 20^{o}C and 60^{o}C, demonstrating stability in short term at these temperatures for uranium concentration.

4. REFERENCES

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