BURN-UP DETERMINATION OF IRRADIATED URANIUM OXIDE BY MEANS OF DIRECT GAMMA SPECTROMETRY AND BY RADIOCHEMICAL METHOD

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CENTRO DE OPERAÇÃO E UTILIZAÇÃO DO REATOR DE PESQUISAS   COURP
ÁREA DE RADIOQUÍMICA

INIS Categories and Descriptors

B11

BURNUP: U_3O_8
U_3O_8: Burnup
THERMAL NEUTRONS: U_3O_8
FISSION PRODUCTS: Radioactive materials
GAMMA SPECTROSCOPY: Thermal neutrons
SOLVENT EXTRACTION: Cerium
SOLVENT EXTRACTION: Cesium
SOLVENT EXTRACTION: Ruthenium
SILICA GEL: Zirconium
SILICA GEL: Niobium

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ABSTRACT

The burn-up of thermal neutrons irradiated U_3O_8 (natural uranium) samples has been determined by using both direct gamma spectrometry and radiochemical methods and the results obtained were compared.

The fission products ^{144}Ce, ^{103}Ru, ^{106}Ru, ^{137}Cs and ^{95}Zr were chosen as burn-up monitors.

In order to isolate the radioisotopes chosen as monitors, a radiochemical separation procedure has been established, in which the solvent extraction technique was used to separate cerium, cesium and ruthenium one from the other and all of them from uranium. The separation between zirconium and niobium and of both elements from the other radioisotopes and uranium was accomplished by means of adsorption on a silica-gel column, followed by selective elution of zirconium and of niobium.

When use was made of the direct gamma-ray spectrometry method, the radioactivity of each nuclide of interest was measured in presence of all others. For this purpose use was made of gamma-ray spectrometry and of a Ge-Li detector.

Comparison of burn-up values obtained by both methods was made by means of Student's "t" test, and this showed that the results obtained in each case are statistically equal.

INTRODUCTION

The term burn-up is used to express the degree of fuel consumption by the fission process relative to some property of the fuel prior to irradiation.

In the development of nuclear fuels and in the operation of nuclear reactors, it is imperative that there be accurate methods for the evaluation of the fuel and reactor performace. An important criterion in determining fuel performance is an accurate determination of the total fissions and the fission rate. This is accomplished by a burn-up determination.

One of the most accurate techniques used to determine burn-up is that based on the measurement of the amount (number of atoms) formed of selected fission products chosen as burn-up monitors and of the heavy nuclides in the irradiated fuel sample.

In the present paper burn-up will be defined by the following expression[6].

$$\% \text{ Burn-up} = \text{Atom Percent Fission} = \frac{\text{Number of Fissions}}{\text{Initial Number of Heavy Atoms}} \times 100$$

The accuracy of the fission product monitors technique depends on the accuracy of analytical procedures used to determine the amount of fission product formed and the number of atoms of heavy nuclides present at the beginning of irradiation as well as on the accuracy of the fission yields used.

In the present work, both mentioned methods were employed for determining burn-up of irradiated natural uranium oxide (U_3O_8) samples. In both cases the amount (number of atoms) formed of the fission products chosen as burn-up monitors was measured.

The number of the fission products atoms chosen as burn-up monitors, namely: ^{144}Ce, ^{103}Ru, ^{137}Cs and ^{95}Zr was determined and was employed to calculate the number of fissions that occured during the irradiation of uranium sample.

When the direct method was used, the radioactivity of each nuclide of interest was measured in presence of all other nuclides present in the sample. For this purpose, use was made of gamma-ray spectrometry and a Ge-Li detector coupled to 4096-channel analyzer.

By means of non-destructive method Bulovic[1] determined the burn-up occurred in uranium oxide samples, using the radioisotopes ^{95}Zr, ^{103}Ru, ^{137}Cs, ^{140}Ba and ^{144}Ce as burn-up monitors.

In the present work a procedure for radiochemical separation of the selected fission products chosen as burn-up monitors was established. I., this way, each of the selected fission products was isolated from all others before the counting step.

After irradiation of the uranium sample, (U_3O_8 pellet), a cooling time of 55 days elapsed before dissolving the U_3O_8 pellet with 4 M nitric acid solution. After conditioning the solution obtained (acidity and uranium concentration), it was percolated through a silica-gel column on which zirconium and niobium were held. Solvent extraction technique was employed for the isolation of the other fission products burn-up monitors chosen.

Di-(2-ethyl hexyl) fosforic acid was the extracting agent used for the extraction of uranium and of cerium.

Cesium was extracted into a solution of dipicrylamine in nitrobenzene, while ruthenium, in this last step, was left in the aqueous phase.

Cornelis et al.[2], used liquid-liquid extractions to isolate the fission products chosen as burn-up monitors, whose measurement was made by gamma-ray spectrometry.

Krtil et al.[4] have determined the burn-up occurred in a nuclear fuel by means of the liquid-liquid extraction and extraction cromatography techniques. The fission products used as burn-up monitors by Krtil et al.[4] were ^{137}Cs, ^{144}Ce and ^{95}Zr.

Terzaghi et al.[8] presented the comparison of results obtained by means of destructive and non-destructive methods for burn-up of UO_2-PuO_2 samples, using ^{137}Cs as burn-up monitor. The results obtained by both methods are in good agreement.

EXPERIMENTAL

I – DESTRUCTIVE METHOD

I.1 – Operations Adopted

The following set of operations was adopted in order to determine the burn-up of uranium oxide by means of the destructive method.

a) Irradiation of an U_3O_8 pellet (2.74 g) for 93.5 hours, under a thermal neutron flux of 2.5×10^{12} $n.cm^{-2} . s^{-1}$.

b) Dissolution of the uranium oxide pellet with 4 M nitric acid solution and suitable dilution of the solution obtained.

c) Radiochemical separation of the selected fission products burn-up monitors after a cooling time of 104 – 119 days.

d) Measurement of the radioactivity of the isolated fission products, namely: ^{144}Ce, ^{103}Ru, ^{106}Ru, ^{137}Cs and ^{95}Zr by gamma-ray spectrometry.

e) Measurement of the number of heavy atoms (^{235}U + ^{238}U) initialy present in the irradiated sample.

1.2 – Radiochemical Separation Procedure

The first step of the established radiochemical separation procedure consisted in percolating the solution containing uranium and its fission products through a silica-gel column where zirconium and niobium were held. In the following steps, the other radioisotopes of interest were isolated by means of the solvent extraction technique.

Di-(2-ethyl hexyl) prosphoric acid (HDEHP) was the extracting agent used to extract uranium and cerium. Cesium was extracted iton a dipicrylamine-nitrobenzene solution, in such a way that ruthenium was left in the aqueous phase.

Figure 1 presents the sequence of the radiochemical procedure used to isolate the fission products of interest, one from another and from uranium. This radiochemical procedure was run six times in order to verify its reproducibility and to examine the degree of purity of the radioisotopes isolated.

The radiochemical procedure established can be summarized as follows.

1.2.1 – Zirconium and Niobium Separation

The solution containing uranium and its fission products obtained by dissolving the U_3O_8 pellet with 4 M nitric acid solution was diluted to give a solution in which uranium concentration was 0.04 M and the acidity of the solution was adjusted to be 3 M in nitric acid. By percolating this solution through a silica-gel bed (internal diameter 0.8 cm, height of bed 10 cm) previously conditioned with 3 M nitric acid solution, more than 94% of ^{95}Zr and more than 99% of ^{95}Nb were held on it.

After washing the column with water, zirconium and niobium were selectively eluted as follows:

a) ^{95}Zr was eluted with 60 ml of 10 M nitric acid solution. The mean recovery for ^{95}Zr in this operation was 48%, with less than 0.2 % of ^{95}Nb initially present in the fission products mixture. Figure 2 shows the elution curve.

b) ^{95}Nb was eluted with 23 ml of 3 M nitric acid solution containing 5% of H_2O_2. The mean recovery for ^{95}Nb was 82%, with less than 3% of ^{95}Zr initially present in the mixture. Elution curve is shown in Figure 3.

c) ^{95}Zr plus ^{95}Nb remaining on the silical-gel bed were eluted simultaneously with 26 ml of 0.05M HF–0.5 HNO$_3$ solution. This fraction was discarded.
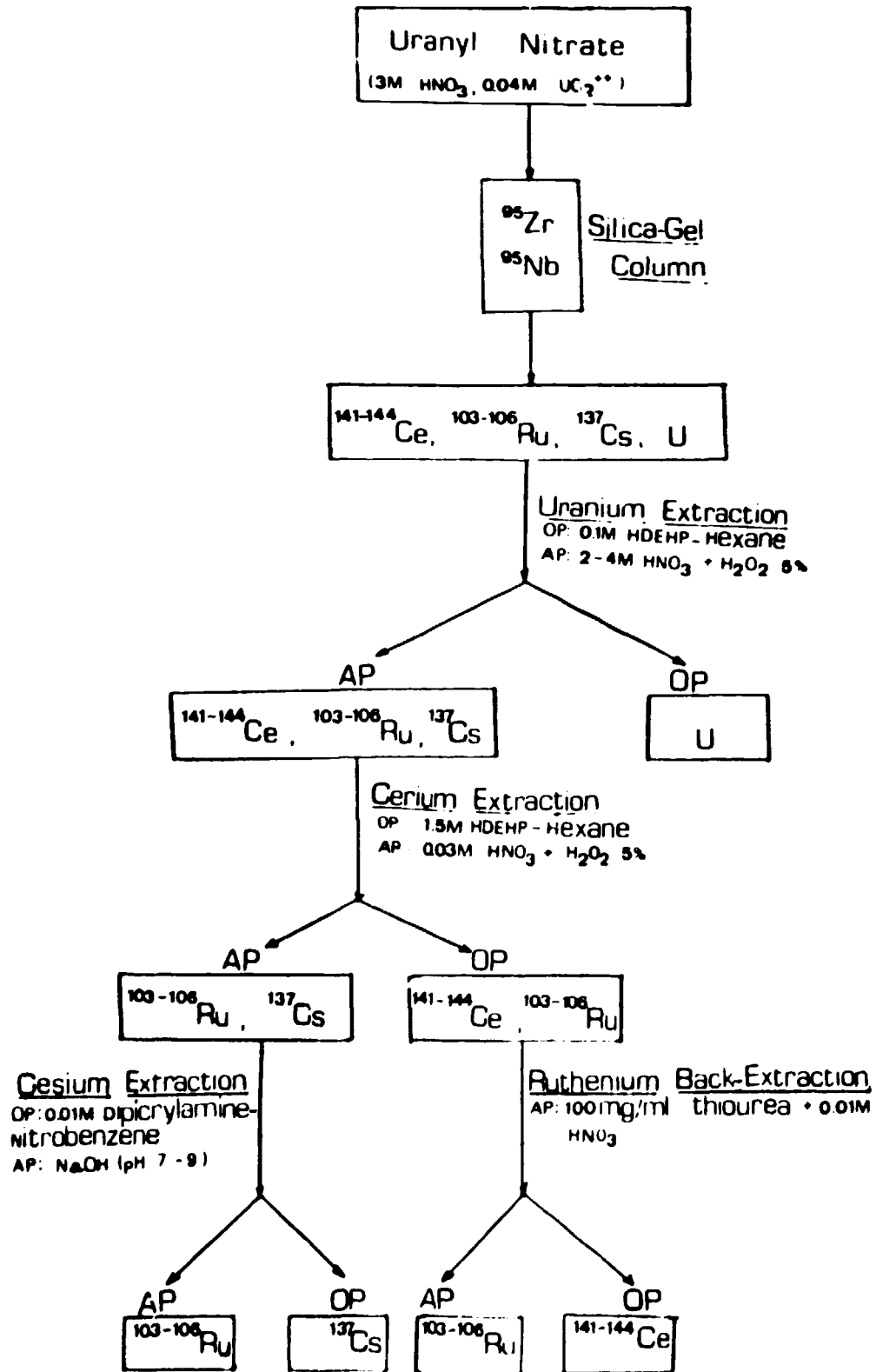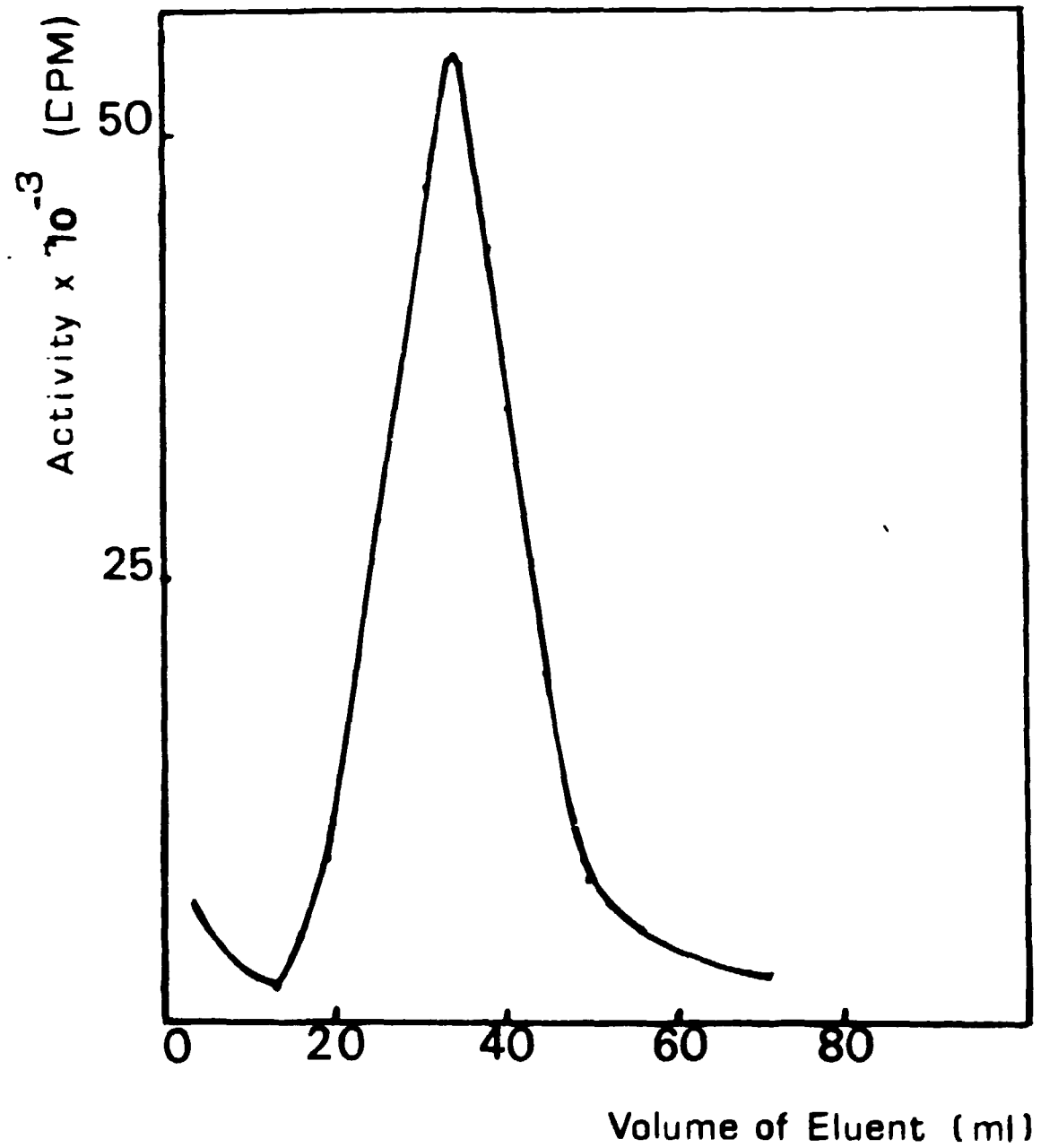
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Figure 1 — Radiochemical separation scheme.
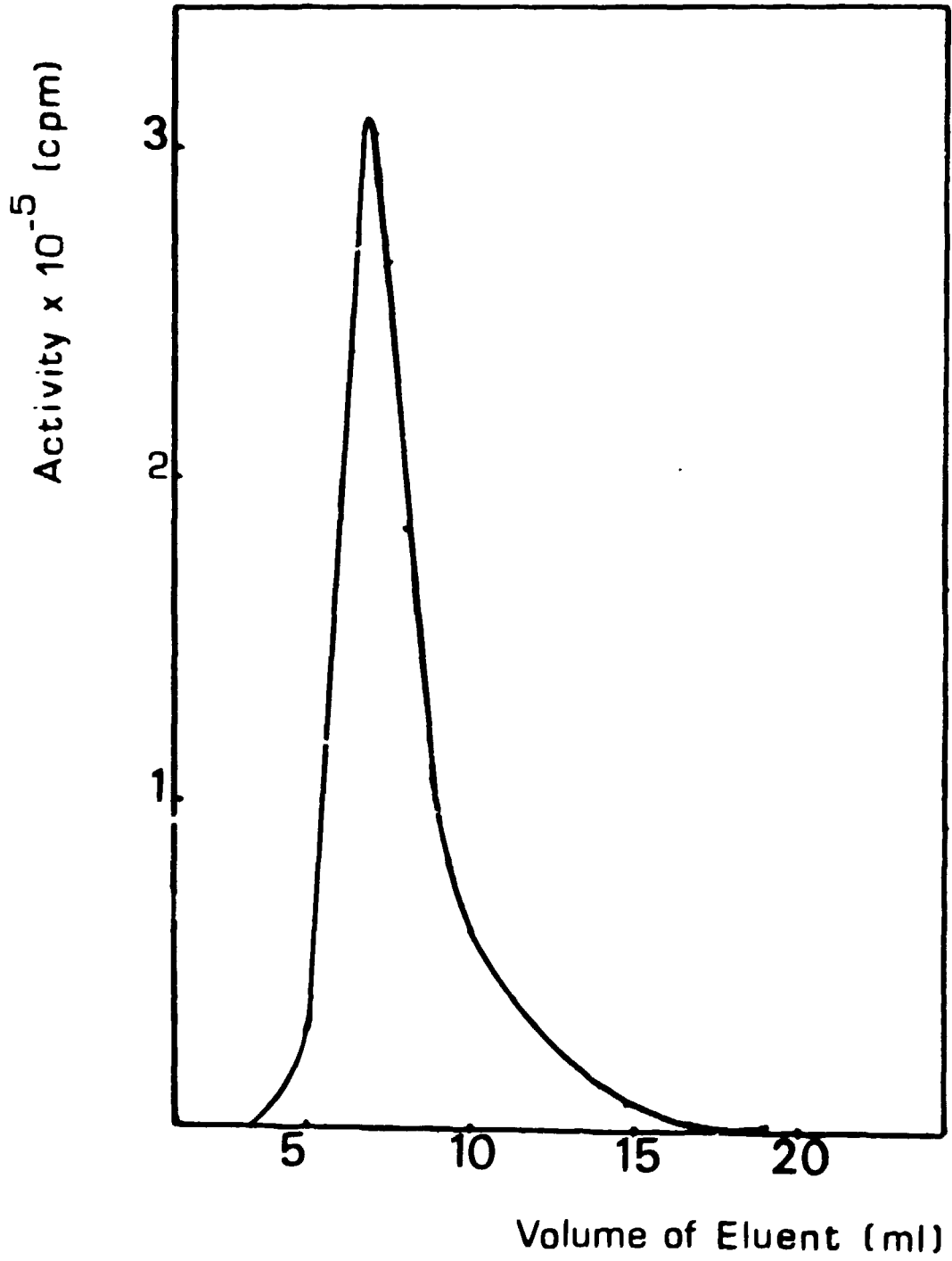
Figure 2 — ^{95}Zr Elution curve.

Figure 3 — ^{95}Nb elution curve.

I.2.2 — Uranium Extraction

Uranium was extracted from the effluent solution of the silica-gel column.

Prior to uranium extraction, performed at an acidity kept in the interval 2 to 4 M nitric acid, H_2O_2 was added to the solution in order to maintain cerium in the trivalent state, remaining, in this way, in the aqueous phase. Uranium, whose concentration in the solution was 0.04 M, was then extracted into a 0.1 M HDEHP solution in hexane. The volume ratio of the phases, organic and aqueous, (O/A) was 1 : 1.

Uranium extraction was complete within 30 minutes of agitation. The resultant aqueous phase contained cerium, cesium and ruthenium.

I.2.3 — Cerium Extraction

The aqueous phase resulting from uranium extraction was adjusted to be 0.03 M in nitric acid. Cerium (III) was then extracted into a 1.5 M HDEHP solution in hexane (O/A ratio = 1 : 1).

Since about 20 to 30% of ruthenium initially present in the fission products mixture was extracted simultaneously with cerium, an additional purification step for cerium was introduced. This additional step consisted in contacting the organic extract (1.5 M HDEHP-hexane) with an aqueous solution of thiourea (100 mg/ml, 0.01 M in HNO_3, shaking time of 1 minute, volume ratio = 1 : 1). In this way, pure cerium remains in the organic phase, since ruthenium is back-extracted into the aqueous phase.

Cerium back-extraction was then obtained by contacting the organic extract (HDEHP) with a 10 M HNO_3 — 5% H_2O_2 mixture. A mean value of 99.9% was obtained for cerium extraction and a mean value of 95% for its back-extraction.

I.2.4 — Cesium Extraction

The aqueous phase resulting from cerium extraction step was made alkaline with sodium hydroxide solution until the final pH of solution was in the range 7 — 9. Cesium was then extracted into a 0.01 M dipicrylamine solution in nitrobenzene. Cesium back-extraction into aqueous phase was obtained by shaking the organic extract with 1.0 M hydrochloric acid solution. The volume ratio was 1 : 1 in both cases.

A mean value of 88.7% was obtained for cesium extraction. Back-extraction operation yielded a mean value of 99.7%. The final cesium solution presented less than 0.6% of ruthenium initially present.

I.2.5 — Ruthenium Recovery

None of the attempts made to extract ruthenium quantitatively into HDEHP solutions succeeded. Most probably, this is due to the irregular chemical behavior of ruthenium which has many possible oxidation states.

Ruthenium was, therefore, isolated by leaving it in the aqueous phase corresponding to cesium extraction with dipicrylamine.

It has been observed that amounts not reproducible of ruthenium, varying in the range from 0% to 27%, were lost by adsorption of the element on the walls of the vessel when, prior to cesium extraction

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with dipicrylamine, the pH of the aqueous phase was adjusted to the required value (interval 7 - 9).

The total final recoveries of ruthenium obtained in the six runs varied within the interval from 41 to 64%.

I.3 — Determination of the Number of Heavy Atoms ($^{235}U + ^{238}U$) and Calculation of the Number of ^{235}U Atoms Initially Present in the Irradiated Sample.

In order to determine the number of heavy atoms ($^{235}U + ^{238}U$) present in the sample after irradiation, the concentration of uranium solution resulting from dissolution of uranium oxide has been determined by means of thiocyanate colorimetric method, according to the procedure described by Currah and Blamish[3]

Since the burn-up was very low, the change in the isotopic compositon is very small, being smaller than the experimental error. Due to this fact, the number of ^{235}U atoms has been calculated by multiplying the total number of uranium atoms by the factor 0.720 (initial ^{235}U enrichment). This value has been used throughout the calculations.

I.4 — Determination of the Number of Fissions

The number of fissions occurred during irradiation was calculated by dividing the number of atoms of one of the selected fission products by its fission yield.

The number of atoms of a given fission product was calculated by dividing its absolute activity by its decay constant.

The absolute activities of the samples used to determine burn-up in the present work were calculated by comparing their countings with the countings of calibrated sources of the respective radioisotopes.

Corrections were made taking into account both the radioactive decay occurred during irradiation and the decay that took place during the period elapsed between the end of irradiation and the counting of the sample. Furthermore, the chemical yields obtained in each case were also taken into account.

II — DIRECT GAMMA SPECTROMETRY METHOD

A suitable aliquot of the fission products mixture solution resulting from dissolution of the irradiated uranium pellet was counted directly, without any chemical processing, by means of a Ge-Li detector coupled to a 4096-channel analyser. Figure 4 presents the gamma-ray spectrum of the fission products mixture.

The cooling time of the fission products mixture in this case was of 58 days.

By comparing the countings due to each of the radioisotopes of interest and those due to the calibrated sources of the same radioisotopes, the absolute activities of the fission products used as burn-up monitors chosen were determined.
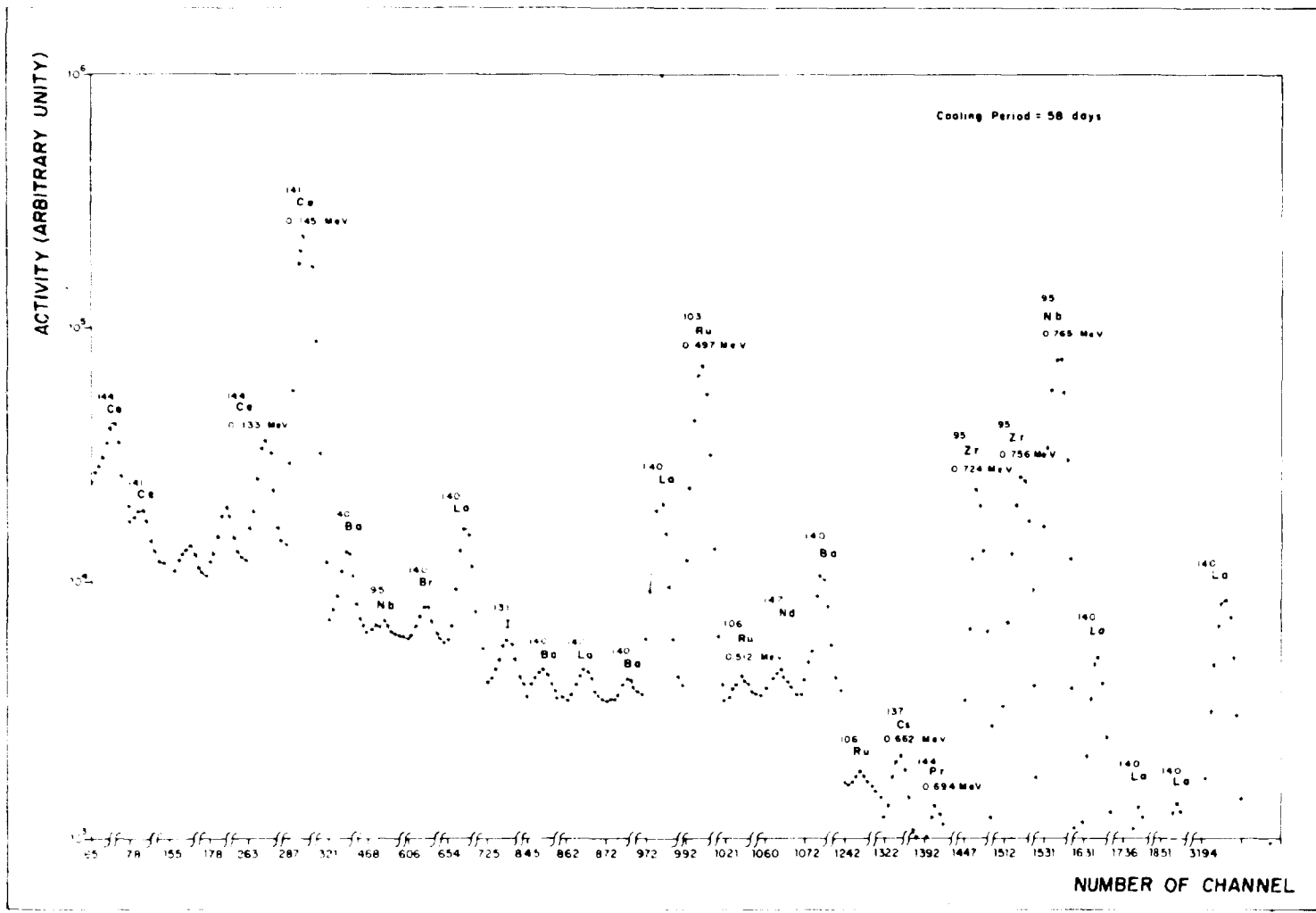
Figure 4 — Gamma-ray spectrum of the fission products mixture.

III — RESULTS FOR BURN-UP DETERMINATION

III.1 — Burn-up Values Obtained by Destructive Method

Burn-up values were calculated by means of the following expression:

$$\% \text{ Burn-up} = \text{Atom Percent Fission} = \frac{\text{Number of Fissions}}{\text{Initial Number of Heavy Atoms}} \times 100$$

In the present work burn-up values were calcualted based on the total number of uranium atoms initially present, that is ^{235}U plus ^{238}U atoms, as well as the number of ^{235}U atoms initially present.

Tables I and II shown burn-up values obtained when the destructive method was employed for both types of calculation, that is, ^{235}U number of atoms and $(^{235}U + ^{238}U)$ number of atoms, respectively.

Variance analysis applied to data of Table I (destructive method and ^{235}U atoms initially present) snowed that burn-up values obtained when ^{106}Ru was used as monitor, must be discarded.

In the same way, variance analysis, applied to data of Table II (destructive method and $(^{235}U + ^{238}U)$ number of atoms as basis) showed that burn-up values obtained when both ^{103}Ru and ^{106}Ru were used as monitors, must be discarded.

In the present work a mean burn-up value was calculated by taking the average between the individual burn-up values obtained considering each of the fission products used as burn-up monitors.

When ^{235}U number of atoms was the basis for calculation, the mean burn-up value was obtained as the average between the burn-up values obtained when the radioisotopes ^{144}Ce, ^{103}Ru, ^{137}Cs and ^{95}Zr were used as monitors. The mean burn-up value found was $(0.158 \pm 0.014)\%$.

In the other case, that is, when $(^{235}U + ^{238}U)$ number of atoms was the basis for burn-up calculation, the mean burn-up value was obtained as the average between the individual burn-up values obtained when the radioisotopes ^{144}Ce, ^{137}Cs and ^{95}Zr were used as monitors. The mean burn-up value obtained in this case was $(1.107 \pm 0.106) \times 10^{-3}\%$.

III.2 — Burn-up Values Obtained By Direct Method

The same expression presented in item III.1 was used for calculating burn-up values by means of the direct method.

As previously, also in the case of direct method, both ^{235}U number of atoms and $(^{235}U + ^{238}U)$ number of atoms were used as bases for calculation of burn-up values.

Table III shows burn-up values obtained by means of direct method (gamma spectrometry without chemical separation) for each of the fission products chosen as burn-up monitors, namely: ^{144}Ce, ^{103}Ru, ^{106}Ru, ^{137}Cs and ^{95}Zr.

Each set of burn-up values shown in Table III $(^{235}U$ number of atoms and $(^{235}U + ^{238}U)$ number of atoms as basis) can be considered homogeneous for a significance level of 0.05, when r maximum and r minimum criterion is applied.

Table I

Uranium Oxide Burn-up [*] Values -- Destructive Method

Monitor Experiment	^{144}Ce	^{103}Ru	^{137}Cs	^{106}Ru	^{95}Zr
1	0.158 ± 0.019	0.169 ± 0.011	0.153 ± 0.012	0.184 ± 0.042	0.155 ± 0.012
2	0.152 ± 0.018	0.160 ± 0.011	0.149 ± 0.015	0.186 ± 0.055	0.148 ± 0.012
3	0.164 ± 0.019	0.173 ± 0.012	0.148 ± 0.011	0.184 ± 0.034	0.169 ± 0.013
4	0.158 ± 0.019	0.174 ± 0.012	0.147 ± 0.012	0.215 ± 0.041	0.141 ± 0.010
5	0.162 ± 0.019	0.172 ± 0.012	0.139 ± 0.009 (discarded)	0.199 ± 0.044	0.145 ± 0.011
6	0.161 ± 0.019	0.169 ± 0.011	0.149 ± 0.012	0.199 ± 0.042	0.155 ± 0.012
Means	0.159 ± 0.019	0.169 ± 0.012	0.149 ± 0.012	0.194 ± 0.043	0.152 ± 0.012

% Burn-up Mean Value: (0.158 ± 0.014)

$$\% \text{ Burn-up}^{(*)} = \frac{\text{Number of Fissions} \times 100}{^{235}\text{U Initial Number of Atoms}}$$

Table II

Uranium Oxide Burn-up[*] Values — Destructive Method

Monitor Experiment	^{144}Ce	^{103}Ru	^{137}Cs	^{106}Ru	^{95}Zr
1	(0.114 ± 0.014)[**]	0.122 ± 0.008	0.111 ± 0.009	0.132 ± 0.030	0.112 ± 0.009
2	0.110 ± 0.013	0.116 ± 0.008	0.107 ± 0.011	0.134 ± 0.040	0.107 ± 0.008
3	0.119 ± 0.014	0.125 ± 0.008	0.107 ± 0.008	0.133 ± 0.024	0.122 ± 0.010
4	0.114 ± 0.014	0.126 ± 0.009	0.106 ± 0.009	0.155 ± 0.030	0.102 ± 0.008
5	0.117 ± 0.014	0.124 ± 0.008	0.100 ± 0.007	0.143 ± 0.031	0.105 ± 0.008
6	0.116 ± 0.014	0.122 ± 0.008	0.108 ± 0.009	0.144 ± 0.030	0.112 ± 0.009
Means	0.115 ± 0.014	0.123 ± 0.008	0.107 ± 0.009	0.140 ± 0.031	0.110 ± 0.009

% Burn-up Mean Value: $(1.104 \pm 0.106) \times 10^{-3}$

$$\% \text{ Burn-up}^{(*)} = \frac{\text{Number of Fissions} \times 100}{(^{235}U + ^{238}U) \text{ Inital Number of Atoms}}$$

(**) All values in this table were multiplied by 10^2.

Table III

Uranium Oxide Burn-up Values — Direct Method

Monitor	^{144}Ce	^{103}Ru	^{106}Ru	^{137}Cs	^{95}Zr
% Burn-up (^{235}U)	0.152 ± 0.019	0.160 ± 0.011	0.202 ± 0.011	0.148 ± 0.011	0.105 ± 0.007
% Burn-up x 10^2 (^{235}U + ^{238}U)	0.110 ± 0.013	0.115 ± 0.008	0.146 ± 0.025	0.107 ± 0.008	0.076 ± 0.006

% Burn-up Mean Values:

^{235}U (0.153 ± 0.019)

^{235}U + ^{238}U (1.108 ± 0.139) x 10^{-3}

When the direct method was used and ^{235}U number of atoms was taken as basis, the mean burn-up value found was $(0.153 \pm 0.19)\%$ and when $(^{235}U + ^{238}U)$ number of atoms was taken as basis the mean burn-up value found was $(1.108 \pm 0.139) \times 10^{-3}\%$.

IV — COMPARISON OF BURN-UP VALUES OBTAINED BY BOTH METHODS

Student's "t" test [7] applied to the comparison of burn-up values determined by both methods showed that within a significance level of 0.10 the results obtained by both methods are equal (calculated t-values are smaller than tabulated t-values).

	% Burn-up (^{235}U taken as basis)	% Burn-up ($^{235}U + ^{238}U$ taken as basis)
Arithmetic means	$x_1 = 0.158$	$x_1 = 1.104 \times 10^{-3}$
	$x_2 = 0.153$	$x_2 = 1.108 \times 10^{-3}$
Variances	$s_1 = 0.014$	$s_1 = 0.106 \times 10^{-3}$
	$s_2 = 0.019$	$s_2 = 0.139 \times 10^{-3}$
Calculated "t"	0.676	0.068
Tabulated "t"	$t(0.10) = 1.70$	$t(0.10) = 1.73$

Indexes 1 and 2 are used to make reference to destructive and direct gamma spectrometry methods, respectively.

DISCUSSION AND CONCLUSIONS

I — RADIOCHEMICAL SEPARATION

The radiochemical separation procedure established in the present work yields:

i) Cessium and niobium with a rather high chemical yield. The mean recovery in both cases was of about 82%. Cesium with less than 1% of ruthenium initially present an niobium with less than 2.5% of zirconium.

ii) Zirconium and ruthenium with a recovery of about 50%. Zirconium presents 0.1% of niobium initially present and ruthenium presents 0.1% of zirconium and niobium.

iii) Virtually total recovery for cerium, although additional separation of ruthenium from cerium is required. (Back-extraction of ruthenium into an aqueous thiourea solution).

II — BURN-UP DETERMINATION

As stated by Bulovic[1], the determination of burn-up as a mean value based on the measurement of several fission products, has several advantages:

i) The mean burn-up value, measured for several fission products is more accurate than the

individual values primarily owing to a reduced effect of the errors of individual fission yields and gamma branching ratios.

ii) The estimation of accuracy of burn-up determination is obtained by comparison of the values determined by measuring the intensities of gamma-rays of various energies.
The folowing factors are among those affecting accuracy of burn-up determinations:

a) error affecting nuclear data (fission yield, half-life, etc.);

b) error in the standards of absolute activity;

c) statistical error in the measurement of activity;

d) error in determination of chemical yield of the separation (destructive method);

e) inaccuracy in the knowledge of the irradiation history.

In the present work, statistical errors in the measurement of the fission products used as monitors were as follows: less than 1% for ^{144}Ce, ^{103}Ru and ^{95}Zr; about 5% for ^{137}Cs and errors within the range 14 to 20% for ^{106}Ru. In this last case the errors were so high because of the low radioactivity of the radioisotope ^{106}Ru.

The errors in the standards of absolute activity were as follows: 2% for ^{137}Cs; 5% for ^{95}Zr; 10% for ^{106}Ru and ^{144}Ce and 3% for ^{103}Ru.

Variance analysis applied to the results obtained by means of destructive method showed that burn-up values obtained by means of ^{106}Ru monitor had to be discarded when ^{235}U number of atoms was the basis for calculation. Probably the factors determining this fact were:

1) rather high error affecting ^{106}Ru standard of absolute activity (10%);

2) rather high statistical error (14-20%) in the measurement of activity in the sample analysed;

3) considerable inaccuracy in the value of ^{106}Ru fission yield (errors in the range 2-5%);

4) possible inaccuracy in the determination of chemical yield.

The same sources of errors most probably explain also why burn-up values calculated by means of both ^{103}Ru and ^{106}Ru monitors had to be discarded when (^{235}U + ^{238}U) number of atoms was taken as basis for burn-up calculation.

In order to cross-check the burn-up value determined in the present work, the value of fission cross section of ^{235}U was calculated[1] as follows:

Irradiation of a cobalt monitor simultaneously with the uranium sample whose burn-up was determined, allowed for calculation of the integrated thermal neutrons flux, by determining the absolute activity of ^{60}Co nuclide.

The value found for the integrated thermal neutrons flux was (2.88 ± 0.04) x 10^{18} n.cm^{-2}, for the experimental irradiation conditions of the present work.

Fission cross section value of ^{235}U was calculated according to the following expression (1):

$$\sigma_f = \frac{N_f}{D\,N^o_{25}}$$

where:

σ_f = fission cross section of ^{235}U

N_f = number of fissions (the same used to calculate burn-up value)

D = integrated thermal neutron flux

N^o_{25} = initial number of atoms of ^{235}U.

The values (549 ± 37) barns and (533 ± 59) barns were found for the fission cross section of ^{235}U. Those values refer to the destructive and direct methods, respectively.

The values found for fission cross section of ^{235}U are in good agreement with those tabulated. For instance, the value presented by Lederer et al.[5] is 577 barns.

RESUMO

Foi determinada a queima ocorrida em amostras de U_3O_8 (urânio natural) irradiadas com nêutrons térmicos, usando-se tanto o método direto de espectrometria gama, como o método baseado em separações radioquímicas.

Os produtos de fissão ^{144}Ce, ^{103}Ru, ^{106}Ru, ^{137}Cs e ^{95}Zr foram escolhidos como monitores.

No procedimento radioquímico estabelecido para isolar os produtos de fissão escolhidos como monitores, utilizou-se a técnica de extração com solventes para separar urânio, cério, césio e rutênio, ao passo que para separar zircônio e nióbio, entre si e dos demais elementos, foi utilizada a adsorção em sílica-gel.

Na determinação da queima pelo método direto, a medida da radioatividade devida a cada um dos monitores escolhidos foi feita em presença de todos os radioisótopos presentes na mistura de produtos de fissão, sendo usado um detetor de Ge-Li para as contagens.

O teste "t" de Student aplicado aos resultados obtidos mostrou que os valores da queima obtidos por ambos os métodos são estatisticamente iguais.

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