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**BURNUP DETERMINATION OF NUCLEAR FUELS
PROJECT REPORT FOR THE
QUARTER APRIL 1 - JUNE 30, 1967**

Edited by

Frederick L. Lisman, William J. Maeck, and James E. Rein

IDAHO NUCLEAR CORPORATION
NATIONAL REACTOR TESTING STATION
IDAHO FALLS, IDAHO

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BURNUP DETERMINATION
OF NUCLEAR FUELS

PROJECT REPORT
FOR THE QUARTER APRIL 1 - JUNE 30, 1967

Edited by

Frederick L. Lisman, William J. Maeck, and James E. Rein

Contributing Personnel

Analytical Development Section

W. A. Emel
R. E. Foster
M. E. Kussy

Mass Spectrometry Group

R. M. Abernathcy
L. W. Butters
L. L. Dickerson
R. E. McAtee
R. A. Nielsen
A. H. Toepfer
G. D. Workman

Operations Analysis (Statistics)

F. W. Spraktes

SUMMARY

In this continuing program to develop methods for the accurate determination of the burnup of nuclear fuels, the main emphasis this quarter was additional measurements of the absolute, thermal fission yields of stable and long-lived isotopes of krypton, xenon, rubidium, and ruthenium for U-235. Also measured was α , the thermal capture-to-fission ratio, for U-235. The determined values are:

<u>Isotope</u>	<u>Fission Yield</u>	<u>Isotope</u>	<u>Fission Yield</u>
Kr-83	0.529	Ru-102	4.21±0.06
Kr-84	0.980	Ru-104	1.84±0.02
Kr-85 (10.76 yr)	0.282	Ru-106	0.414±0.010
Rb-85	1.30±0.02	Ru-106 (radio-metric)	0.404±0.015
Kr-86	1.86	Xe-131	2.72±0.06
Rb-87	2.52±0.03	Xe-132	4.04±0.09
Ru-101	5.05±0.07	Xe-134	7.34±0.17

$$\alpha = 0.176_3 \pm 0.002_2; \alpha_0(2200 \text{ m/sec}) = 0.171_0 \pm 0.003_4$$

These values are tentative and as additional capsules are analyzed, refined data will be reported. Previous project reports are:

Annual - 1962	TID-17385
Annual - 1963	IDO-14649
Annual - 1964	IDO-14656
Quarter, January-March, 1965	IDO-14660
Quarter, April-June, 1965	IDO-14663
Quarter, July-September, 1965	IDO-14667
Quarter, October-December, 1965	IDO-14676
Quarter, January-March, 1966	IDO-14678
Quarter, April-June, 1966	IDO-14681
Quarter, July-September, 1966	IN-1064
Quarter October-December, 1966	IN-1113
Quarter, January-March, 1967	IN-1157 Rev.

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BURNUP DETERMINATION OF NUCLEAR FUELS

INTRODUCTION

The objective of this project is the development of methods for the accurate determination of burnup of nuclear fuels. The emphasis is on power reactor fuels having long residence time and high burnup rather than on short term, experimental irradiations. The principal measurement technique under study is chemical analysis for fission products. The use of long-lived, radiochemically determined fission products is not applicable because the errors of the in-pile decay corrections are magnified with long term irradiations, the nuclear constants are not known with sufficient accuracy, and the few available nuclides, Cs-137 and Sr-90, are volatile. The use of stable fission product nuclides, determined by isotope dilution mass spectrometry, has therefore been selected. The degree of reliability of a burnup determination based on stable fission product analyses depends on three factors; the accuracy of the values used for fission yields, the linearity between the number of fissions and the number of fission product atoms present at the end of the irradiation, and the reliability of the analysis methods.

Specific goals of this project are the accurate measurement of the fission yields of selected nuclides for various fissioning isotopes, the selection of fission product nuclides for use as fission monitors for various fuel types, and the development of separation and mass analysis methods for these nuclides. Presently, experiments are in progress to measure the absolute thermal fission yields for U-233, U-235, Pu-239, and Pu-241 to a high degree of accuracy. Later in the program, fast fission yields will be measured.

Initial values for many U-233 and U-235 thermal fission yields have been reported in previous reports IDO-14660, IDO-14663, IDO-14667, IDO-14676, IDO-14678, IDO-14681, IN-1064, IN-1113, and IN-1157 Rev.

ABSOLUTE U-235 THERMAL FISSION YIELDS
FOR THE STABLE AND LONG-LIVED FISSION
PRODUCTS OF KRYPTON, XENON, RUBIDIUM, AND RUTHENIUM

The major goal of the burnup project is the accurate determination of the thermal fission yields of long-lived and stable fission products. A series of capsules containing weighed amounts of the oxides of highly enriched U-233, U-235, Pu-239, and Pu-241 was prepared and irradiated in the MTR (1). During this quarter, the last of four U-235 capsules was dissolved and the absolute fission yields were measured for isotopes of krypton, xenon, rubidium, and ruthenium. Also measured was α , the thermal capture-to-fission ratio for U-235.

Irradiation History of Capsule

Standard burnup capsule CPP-6-5-3, which contained 122.0 mg of highly enriched U_3O_8 mixed with high-purity aluminum powder and welded in a high-purity aluminum tube, was irradiated for two fuel cycles (1290 Mwd) in the MTR at an estimated unperturbed flux of 6×10^{13} n/cm²-sec (accumulated nvt = 1.7×10^{20}). The cadmium ratio for cobalt for this irradiation was 15. The capsule was discharged from the reactor July 20, 1964, and allowed to cool for about three years before dissolution.

Dissolution of Capsule, (R. E. Foster, Jr., W. A. Emel)

The dissolution of the capsule was carried out in a 4-in. lead-shielded facility. The dissolution apparatus has been previously described (1,2). The dissolver flask and reflux condenser were fabricated from high-purity quartz and an acid mixture of 6M HCl-1.6M HNO₃, prepared from quartz-distilled reagents, was used for the dissolution (1).

The dissolution proceeded smoothly and was complete in 3.5 hr. The fission gases, krypton and xenon, appeared 1 hr after the start of the dissolution and their evolution continued for 2 hr. The final solution (about 200 ml) was water clear and contained no residue.

After cooling, the dissolver solution was quantitatively transferred to a calibrated 250-ml, quartz volumetric flask with the aid of 6M HCl-1.6M HNO₃ rinses. The solution was diluted to volume with quartz-distilled water, a Teflon-coated bar magnet was introduced, and the contents were stirred for 1 hr.

Determination of Number of U-235 Fissions, (R. M. Abernathey, L. W. Buttars, F. L. Lisman, R. A. Nielsen, F. W. Sprakles).

The accuracy of the absolute fission yield measurements directly depends on the accuracy of the determined number of fissions in the

irradiated U-235 capsule. For this reason, a large effort was placed on the pre- and postirradiation analyses for the atoms of the various uranium isotopes from which the number of fissions was calculated.

The U_3O_8 (obtained from ORNL) that was weighed into the capsule was analyzed both for weight percent uranium content and isotopic distribution. For the former, samples of the U_3O_8 were weighed on a microbalance and dissolved in nitric acid, weighed aliquots were mixed with weighed aliquots of a standard U-233 solution, and the mixtures were analyzed by isotope dilution mass spectrometry. Isotopic distribution was determined on unspiked samples. The calculated atoms of each uranium isotope (in the capsule charge of 122.0 ± 0.1 mg U_3O_8) are summarized in Table I. Of interest was the finding that the uranium content of the U_3O_8 corresponded to 99.62% of theoretical.

The postirradiation atoms of each uranium isotope were determined in a similar manner with unspiked and U-233 spiked aliquots of the dissolver solution. The aliquots of the dissolver solution were delivered with a calibrated 1000- μ l pipet. The calculated atoms are presented in Table I.

The number of U-235 fissions was calculated from the relationship:

$$F_5 = U^0 - U$$

in which:

F_5 = number of U-235 fissions

U^0 = preirradiated atoms of uranium

U = total postirradiated atoms of uranium

To accurately use this relationship, one must account for those atoms of U-236 and U-238 lost by capture and subsequent decay to non-uranium isotopes. This was done with data obtained from a computer program which simulated the history of the irradiation. Discussion and details of the buildup and burnout calculations have been reported previously (2).

From the results:

$$U^0 = (2.6355 \pm 0.0029) \times 10^{20}$$

$$U = (2.3649 \pm 0.0016) \times 10^{20}$$

$$\text{The number of fissions, } F_5 = (0.2706 \pm 0.0033) \times 10^{20}$$

The propagated standard deviation error of 0.0033×10^{20} for the computed number of fissions corresponds to a relative error of 1.22%. The atom percent U-235 fissioned (defined as atoms of U-235 fissioned per 100 initial atoms of U-235) is:

$$\begin{aligned} \%F_5 &= \frac{100 (0.2706 \pm 0.0033) \times 10^{20}}{(2.6135 \pm 0.0029) \times 10^{20}} \\ &= 10.35 \pm 0.13 \end{aligned}$$

in which the value of 0.13 is the propagated standard deviation error and corresponds to a relative error of 1.22%.

Table I. ANALYSIS RESULTS OF IRRADIATED U-235 CAPSULE AND
CALCULATED NUMBERS OF ATOMS

<u>Preirradiation Data</u>				
Initial Loading	122.0±0.1 mg U ₃ O ₈			
mg U-235/mg U ₃ O ₈	0.8361±0.0007			
mg U-235 in capsule	102.00±0.11 (0.11%)			
	<u>U-234</u>	<u>U-235</u>	<u>U-236</u>	<u>U-238</u>
Isotopic distribution, wt%	0.4677	99.1633	0.0603	0.3087
Std Dev _{ind}	0.0025	0.0032	0.0015	0.0006
Std Dev _{avg} (n=3)	0.0015	0.0019	0.0009	0.0004
Number of Atoms	1.2381x10 ¹⁸	2.6135x10 ²⁰	1.5829x10 ¹⁷	8.0356x10 ¹⁷
Std Dev _{avg}	0.0042x10 ¹⁸	0.0029x10 ²⁰	0.0237x10 ¹⁷	0.0137x10 ¹⁷
Total atoms of uranium	(2.6355±0.0029)x10 ²⁰ (0.11%)			

<u>Postirradiation Data</u>				
	<u>U-234</u>	<u>U-235</u>	<u>U-236</u>	<u>U-238</u>
Isotopic distribution, atom%	0.5116	97.0644	2.0807	0.3432
Std Dev _{ind}	0.0024	0.0078	0.0050	0.0018
Std Dev _{avg} (n=4)	0.0012	0.0039	0.0025	0.0009
Number of Atoms	1.2099x10 ¹⁸	2.2955x10 ²⁰ (a)	4.9207x10 ¹⁸	8.1164x10 ¹⁷
Std Dev _{avg}	0.0029x10 ¹⁸	0.0016x10 ²⁰	0.0069x10 ¹⁸	0.0219x10 ¹⁷
Total atoms of uranium	(2.3649±0.0016)x10 ²⁰ (0.07%)			
Atoms of uranium lost during irradiation by capture on U-236 and U-238.	0.00002x10 ²⁰			
Total atoms of uranium corrected for capture loss of U-236 and U-238	(2.3649±0.0016)x10 ²⁰ (0.07%)			

(a) Average value of 8 determinations by isotope dilution mass spectrometry. Uncertainty includes uncertainty of U-233 spike solution.

Capture-to-Fission Ratio for U-235 (F. L. Lisman, F. W. Spraktes)

The effective capture-to-fission ratio, $\hat{\alpha}$, is calculated from the data of Table I. The number of U-235 atoms captured to form U-236 equals the postirradiation number of U-236 atoms plus those lost by capture to U-237 (and β^- decay to Np-237) less those atoms of U-236 initially present in the loading. As stated previously, the number of U-236 atoms lost by capture was calculated from a computer program.

Calculation of $\hat{\alpha}$ for U-235

(1) atoms U_6 present at end of irradiation	$= (4.9207 \pm 0.0069) \times 10^{18}$
(2) atoms U_6 lost by capture	$= 0.0077 \times 10^{18}$
(3) (1) + (2)	$= (4.9284 \pm 0.0069) \times 10^{18}$
(4) atoms U_6^0 , initial atoms loaded into capsule	$= (0.1583 \pm 0.0024) \times 10^{18}$
(5) atoms U_5^0 captured to U_6 , (3)-(4)	$= (4.7701 \pm 0.0073) \times 10^{18}$
(6) atoms U_5^0 fissioned (see previous section)	$= (27.06 \pm 0.33) \times 10^{18}$
(7) $\hat{\alpha}$, (atoms U_5^0 captured / atoms U_5^0 fissioned), (5)/(6)	$= 0.1763 \pm 0.0022$

The propagated standard deviation value of 0.0022 corresponds to a relative error of 1.23%.

The capsule was irradiated in the MTR in a location near the beryllium reflector where the cadmium ratio for cobalt is 15. The Wescott "r" value corresponding to $R_{Cd}^{Co} = 15 \pm 3$ is 0.031 ± 0.006 computed from the relationship (3):

$$r = \frac{s_{Co}}{R_{Cd}^{Co} \left[s_{Co} + \frac{1}{k} \sqrt{\frac{T}{T_0}} \right] - s_{Co}}$$

in which:

$$\begin{aligned}
 g_{\text{Co}} &= 1 \\
 R_{\text{Cd}}^{\text{Co}} &= \text{cadmium ration for cobalt} = 15 \pm 3 \\
 s_{\text{Co}} &= 1.795 \text{ (ref. 4)} \\
 K &= 2.2931 \text{ (for 0.040 in. of Cd) (ref. 4)} \\
 T &= 313^\circ\text{K} \\
 T_0 &= 293.6^\circ\text{K}
 \end{aligned}$$

to convert $\hat{\alpha}$ to α_0 , the 2200 m/sec value, the relationship:

$$1 + \alpha_0 = (1 + \hat{\alpha}) (g_f + rs_f) / (g_a + rs)$$

applies. For a neutron temperature of 40°C (313°K) the values are:

$$\begin{aligned}
 g_f &= 0.9735 \pm 0.0016 \text{ (ref. 5)} \\
 s_f &= -0.0316 \text{ (ref. 5)} \\
 r &= 0.031 \pm 0.006 \\
 g_a &= 0.9723 \pm 0.0012 \text{ (ref. 5)} \\
 s_a &= 0.1501 \text{ (ref. 5)} \\
 \hat{\alpha} &= 0.176_3 \pm 0.002_2
 \end{aligned}$$

Therefore:

$$\alpha_0 \text{ for U-235} = 0.171_0 \pm 0.003_4 \text{ (1.98\%)}$$

The quoted error (standard deviation) 0.003_4 includes errors in the measured $\hat{\alpha}$, epithermal parameter "r", and Westcott's quoted uncertainties for the g values.

Krypton and Xenon Fission Yields. (R. M. Abernathey, L. L. Dickerson, R. E. Foster, Jr., F. L. Lisman, F. W. Spraktes)

These two gases were trapped from the capsule dissolver off-gas on molecular Sieve 5A at -190°C . The concentration of each gas was determined by temperature-programmed gas chromatography. The separated gas fractions from the chromatographic column were collected on charcoal at -80°C and then transferred to the mass spectroscopy laboratory for isotopic distribution analysis. Details of the apparatus and the procedure for collection and analysis have been reported (1,2).

The analysis results and calculated absolute thermal fission yields are summarized in Table II for krypton and in Table III for xenon. The error associated with each value for the xenon isotopes is the standard deviation. Due to a power failure during recycling of the krypton , through the gas chromatograph, no error can be assigned to the volume of krypton obtained and hence to the various krypton fission yields.

Table II. THERMAL FISSION YIELDS OF
KRYPTON ISOTOPES FOR U-235

std cc Kr = 0.0363

	<u>Kr-83</u>	<u>Kr-84</u>	<u>Kr-85(10.76 yr)</u>	<u>Kr-86</u>
Isotopic distribution, atom %	14.42	27.43	6.43	51.72
Determined number of atoms	1.4073×10^{17}	2.6771×10^{17}	6.2755×10^{16}	5.0477×10^{17}
Atoms after correction for neutron capture and radioactive decay.	1.4327×10^{17}	2.6526×10^{17}	7.6363×10^{16}	5.0469×10^{17}
Percent fission yield	0.529	0.980	0.282	1.86

Table III. THERMAL FISSION YIELDS OF
XENON ISOTOPES FOR U-235

std cc Xe = 0.2552 ± 0.0051 (2.0%)

	<u>Xe-131</u>	<u>Xe-132</u>	<u>Xe-134</u>	<u>Xe-136</u> ^{a)}
Isotopic distribution, atom %	10.66	16.01	29.09	44.25
Determined number of atoms	7.3141×10^{17}	1.0985×10^{18}	1.9959×10^{18}	3.0361×10^{18}
Std Dev	0.1463	0.0220	0.0399	0.0607
Atom after correction for neutron capture	7.3624×10^{17}	1.0935×10^{18}	1.9867×10^{18}	
Std Dev, % (includes estimated uncertainties in correction factors for neutron capture)	2.0	2.0	2.0	
Percent fission yield	2.72	4.04	7.34	
Std Dev	0.06	0.09	0.17	
Std Dev, %	2.3	2.3	2.3	

a) No attempt was made to distinguish those atoms of Xe-136 resulting from Xe-135 (n, γ)Xe-136 reactions and hence no Xe-136 fission yield can be reported.

Rb-85 and Rb-87 Fission Yields. (R. M. Abernathey, R. E. Foster, Jr., M. E. Kussy, F. L. Lisman, R. E. McAtee, R. A. Nielson, F. W. Spraktes, G. D. Workman)

The absolute thermal fission yields of Rb-85 and Rb-87 were determined by isotope dilution mass spectrometry. Eight aliquots, four spiked with a known amount of Rb-85 and four without spike, of the dissolver solution were chemically separated and then analyzed by mass spectrometry. The chemical separation and mass spectrometric procedures have been previously reported (6).

The analysis results and calculated absolute thermal fission yields are summarized in Table IV. The method of calculation and the error propagation for the determined number of Rb-85 and Rb-87 atoms have been previously reported (6).

To calculate the yield of the 85 mass chain, the number of determined Rb-85 atoms was added to the determined atoms of 10.76-yr Kr-85.

The accurate determination of absolute fission yields, for samples which have undergone long term irradiations, requires corrections for the buildup and burnout of the various fission products due to neutron capture. The method of correcting for buildup and burnout of the fission products is based on a series of computer programs which simulate the irradiation history of the sample. These programs yield the number of individual fission product atoms formed during the irradiation and their manner of formation. In addition, the programs consider the decay of any radioactive nuclides for the time period between termination of the irradiation and sample analysis. Details of these programs have been previously reported (6,7).

Table IV. THERMAL FISSION YIELDS OF Rb-85
AND Rb-87 FOR U-235

	<u>Rb-85</u>	<u>Rb-87</u>
Determined number of atoms	2.8862×10^{17}	6.8075×10^{17}
Std Dev _{avg} (n=4)	0.0219×10^{17}	0.0372×10^{17}
Atom after correction for neutron capture	2.8869×10^{17}	6.8075×10^{17}
Std Dev, % (includes estimated uncertainties in correction factors for neutron capture).	0.76%	0.55%
Determined atoms of 10.76-yr Kr-85 ^{a,b}	$(0.6275 \pm 0.0188) \times 10^{17}$	
Total mass - 85 atoms	$(3.5144 \pm 0.0289) \times 10^{17}$	
Percent fission yield	1.30	2.52
Std. Dev	0.02	0.03
Std Dev, %	1.5	1.2
(a) Not corrected for either out-of-pile or in-pile decay. The sum of the determined Kr-85 and the determined Rb-85 is the total number of mass-85 atoms because the krypton is quantitatively removed from the dissolver solution at time of dissolution.		
(b) The relative standard deviation on the determined number of 10.76-yr Kr-85 atoms is estimated to be $\pm 3.0\%$.		

Ruthenium Fission Yields (R. M. Abernathey, R. E. Foster, Jr., M. E. Kussy, F. L. Lisman, R. E. McAtee, R. A. Nielson, F. W. Spraktes, G. D. Workman)

The absolute thermal fission yields of four ruthenium isotopes, Ru-101, -102, -104, and -106, were determined by isotope dilution mass spectrometry. The atoms of Ru-106 also were measured radiometrically. Four 1000- μ l aliquots from the dissolver solution were spiked with known amounts of Ru-106 and carried through the separation and analysis procedure. The detailed separation and analysis procedures have been previously described (8). The analysis results and the calculated fission yields are summarized in Table V.

As in the case of the thermal fission yields of rubidium, corrections for the buildup and burnout of the ruthenium isotopes are based on a computer program which simulates the irradiation history of the capsule.

Comparison to Previous Results

A comparison of the absolute thermal fission yields determined on the present capsule to previously determined values for other U-235 capsules is given in Table VI. The agreement between the various capsules is well within experimental error.

Table V. THERMAL FISSION YIELDS OF LONG-LIVED AND
STABLE RUTHENIUM ISOTOPIES FOR U-235

	<u>Ru-101</u>	<u>Ru-102</u>	<u>Ru-104</u>	<u>Ru-106</u>	<u>Ru-106</u> (Radiometric)
Determined number of atoms	1.3658×10^{18}	1.1400×10^{18}	4.9938×10^{17}	1.3140×10^{16}	1.3587×10^{16}
Std Dev _{avg} (n=4)	0.0080×10^{18}	0.0072×10^{18}	0.0316×10^{17}	0.0214×10^{16}	0.0434×10^{16}
Atoms after correction for neutron capture and decay	1.3662×10^{18}	1.1400×10^{18}	4.9927×10^{17}	1.1218×10^{17}	1.093×10^{17}
Std Dev (%)	0.59	0.63	0.63	1.9	3.4
Percent Fission Yield	5.05	4.21	1.84	0.414	0.404
Std Dev	0.07	0.06	0.02	0.010	0.015

Table VI. FISSION YIELD COMPARISON

<u>Isotope</u>	<u>This Capsule</u>	<u>6-4-1</u> (Ref.2,6,8)	<u>6-6-2</u> (Ref.7,6,8)
Kr-83	0.529	0.491 ± 0.020	0.499 ± 0.010
Kr-84	0.980	0.919 ± 0.027	0.936 ± 0.019
Rb-85	1.30 ± 0.02	1.30 ± 0.02	1.31 ± 0.02
Kr-86	1.86	1.79 ± 0.06	1.82 ± 0.04
Rb-87	2.52 ± 0.03	2.57 ± 0.03	2.58 ± 0.04
Ru-101	5.05 ± 0.07	5.03 ± 0.06	5.02 ± 0.07
Ru-102	4.21 ± 0.06	4.18 ± 0.08	4.17 ± 0.06
Ru-104	1.84 ± 0.02	1.81 ± 0.02	1.81 ± 0.03
Ru-106	0.414 ± 0.010	0.373 ± 0.006	0.398 ± 0.008
Ru-106 (radiometric)	0.404 ± 0.015	0.382 ± 0.013	0.412 ± 0.015
Xe-131	2.72 ± 0.06	2.78 ± 0.09	2.87 ± 0.06
Xe-132	4.04 ± 0.09	4.15 ± 0.14	4.28 ± 0.09
Xe-134	7.34 ± 0.17	7.53 ± 0.25	7.73 ± 0.20

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