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by

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MEASUREMENT OF RADIOACTIVE GASEOUS EFFLUENTS FROM VOLOXIDATION AND DISSOLUTION OF SPENT NUCLEAR FUEL*

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Abstract

Laboratory-scale tests gave data on the release of tritium, ^{14}C, ^{85}Kr, and ^{129}I as radioactive gases from spent nuclear fuels during voloxidation and dissolution. Voloxidation, a proposed reprocessing step, is intended to remove tritium from fuel by oxidation of UO_2 to U_3O_8 prior to dissolution of the fuel with nitric acid. ^{14}C, ^{85}Kr, and ^{129}I may be evolved in both steps. Quantitative data from the tests may be used in designing off-gas treatment processes and equipment. The tests were performed in a shielded cell with a combination voloxidizer-dissolver. With a recirculating off-gas system, tritium and ^{14}C were trapped on molecular sieves; ^{129}I was trapped on silver-exchanged zeolite. ^{85}Kr was measured by online gamma-ray counting. Zircaloy-clad UO_2 fuels from H. B. Robinson-2, Oconee-1, and Saxton reactors, with burnups from ~100 to ~28,000 MWD/MTHM, were tested. The results confirm that voloxidation released most of the tritium but only small fractions of the ^{14}C, ^{85}Kr, and ^{129}I; the remainder of these radioactive gases evolved when the voloxidized fuels were dissolved. Voloxidation off-gases typically contained >99.8% of the tritium, 17 to 22% of the ^{14}C, 7 to 17% of the ^{85}Kr, and <8% of the ^{129}I. Tritium evolved as HTO, with <0.1% as HT.

Introduction

In processing spent nuclear fuel to recover fissile material, volatile radionuclides such as tritium, ^{14}C, ^{85}Kr, and ^{129}I evolve into the off-gas system. Technology is available for trapping the gaseous radioactive species to prevent release to the environment.(1-3) However, proper design of off-gas facilities requires quantitative data on amounts evolved in each process step.

A reference flowsheet for head-end processing steps is shown in Figure 1. The spent fuel rods are sheared into short pieces, and then a voloxidation step drives tritium out of the fuel. The oxidized fuel is dissolved with nitric acid, and the clarified solution is fed to solvent-extraction steps. Most of the gaseous radionuclides enter the off-gas system during the voloxidation and dissolution steps.

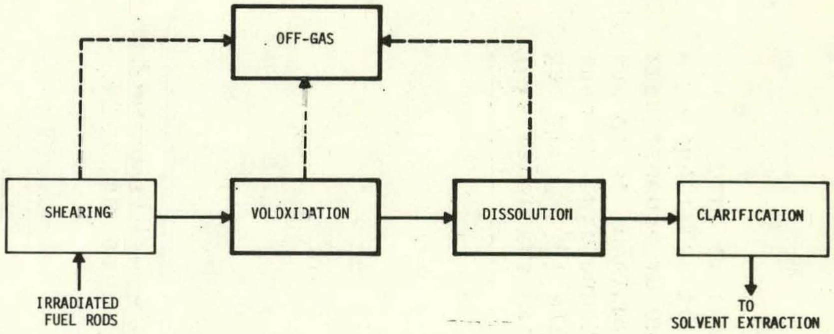

Figure 1. Head-end process operations.

In voloxidation, first developed at Oak Ridge National Laboratory,(4) UO_2 fuel oxidizes to U_3O_8 at about 490°C, promoting evolution of tritium as HTO (water vapor). During oxidation, the fuel expands, disintegrates into fine powder, and separates from the cladding. Tritium removal before fuel dissolution is desirable to avoid extensive isotopic dilution by nontritiated water. Previous studies(4,5) showed that even though tritium evolved quantitatively during voloxidation, species such as ^{85}Kr and ^{129}I evolved incompletely.

In this paper, new laboratory-scale measurements of tritium, ^{14}C, ^{85}Kr, and ^{129}I evolved during voloxidation and dissolution of irradiated power-reactor fuels are reported. We confirm the quantitative evolution of tritium during voloxidation and report the distribution of the other species between voloxidation and dissolution.

Experimental Procedure

Apparatus

Voloxidizer-Dissolver. A stainless-steel reaction vessel, shown in Figure 2, serves for both voloxidation and dissolution of irradiated fuel. The apparatus is in a shielded cell for remote operation and is connected to off-gas collection facilities. The voloxidizer-dissolver is a 2-liter reaction chamber equipped with numerous heaters, thermocouples, cooling coils, a rotary agitator, and orifices for off-gases. The reaction chamber rotates on a horizontal axis to provide two operating positions: 1) tilted as shown in Figure 2 for voloxidation or 2) vertically upright for dissolving. Gases emerge through a reflux condenser during dissolving or through another orifice during voloxidation.

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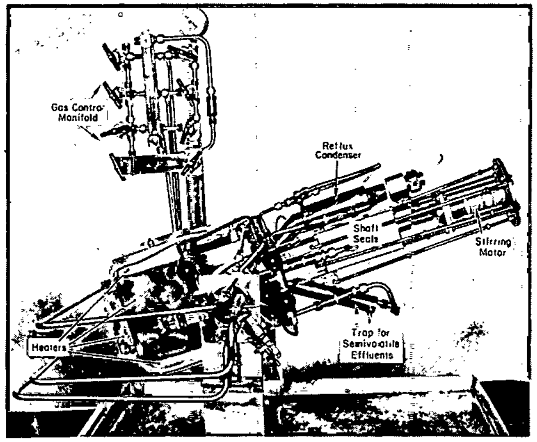

Figure 2. Voloxidizer-dissolver.

Off-Gas System. The voloxidizer-dissolver is coupled to laboratory-scale off-gas equipment both inside and outside the shielded cell; the arrangement for voloxidation is shown schematically in Figure 3. A small pump recirculates helium carrier gas through the closed system. Oxygen consumed in the system is replaced to maintain constant oxygen content in the recirculating gas. ^{85}Kr is measured by online gamma-ray counting; tritium, ^{14}C, and ^{129}I are collected on a series of molecular sieve traps for later analyses. The off-gas system was tested and calibrated with tracer amounts of radioactive gases.

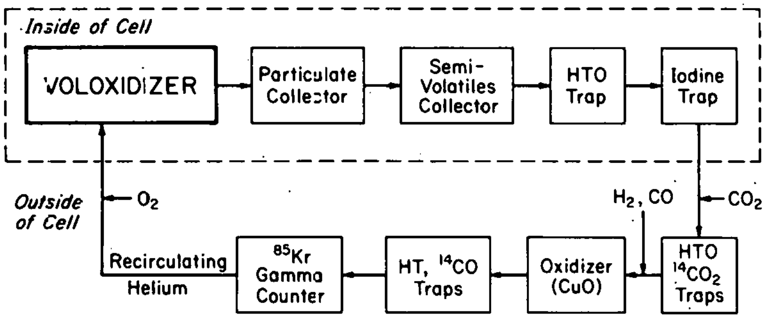

Figure 3. Off-gas system.

Particulates in the off-gas are collected on impacter plates at the mouth of the voloxidizer. Semivolatile elements such as cesium, antimony, and ruthenium may plate out along a temperature-gradient tube following the particulate collector. Data on particulates and semivolatiles are not discussed in this paper.

Three Type 3A molecular sieve traps collect tritium as HTO. One trap closely coupled to the voloxidizer collects evolved HTO quantitatively. Water vapor introduced into the off-gas recirculation line carries the HTO. The other two traps are in a hood outside the shielded cell, one upstream and one downstream from an oxidizing bed of CuO at 300°C. The upstream trap is a backup for collecting residual HTO, whereas the downstream trap collects HTO converted from HT in the off-gas. Hydrogen gas introduced upstream from the CuO bed carries any HT.

Two Type 13X molecular sieve traps collect ^{14}C as $^{14}CO_2$. These traps are also outside the shielded cell, one on each side of the heated CuO bed. Any ^{14}CO in the off-gas is oxidized to $^{14}CO_2$ and collected in the second trap. Carrier CO_2 is added before the first trap, and carrier CO is added before the CuO bed. As discussed in the following section, separation of CO and CO_2 in this system is not entirely satisfactory.

The off-gas circulates through a calibrated chamber for counting the 514-keV gamma ray of ^{85}Kr with a high-resolution Ge-Li detector. These rapid online measurements allowed the course of voloxidation and dissolution reactions to be monitored.

A bed of silver-exchanged zeolite (Ag^0-X) at 180°C traps iodine in the off-gas. During voloxidation, the iodine trap, although near the voloxidizer in the shielded cell, is downstream from the tritium trap. Because any ^{129}I that deposited in the upstream lines or in the tritium trap would not have been detected, the measured ^{129}I is possibly less than the total amount evolved.

The arrangement of the off-gas system for dissolution is similar to that shown in Figure 3. Equipment outside the shielded cell is identical. In the cell, dissolver off-gases pass through a reflux condenser directly to an iodine trap.

Analytical Methods

After each voloxidation or dissolution test, tritium, ^{14}C, and ^{129}I are removed from their primary sorbents by a heat treatment. The methods used are summarized in Table I. HTO and $^{14}CO_2$ are desorbed from molecular sieve pellets to obtain forms suitable for liquid scintillation beta-counting. Used pellets are discarded.

Because silver interferes with neutron-activation analysis for ^{129}I, the iodine is removed from silver zeolite as HI and resorbed on lead zeolite, which does not interfere. This method was adapted from the work of Staples, Murphy, and Thomas.(6,7) The regenerated silver-zeolite traps are reused.

Table I. Analyses for gaseous radioactive species.

Radionuclide	Primary Sorbent	Bakeout[a]	Carrier Gas	Secondary Trap	Analytical Method
^{3}H	3A	Yes	H_2O	Cold trap[b]	β counting
^{14}C	13X	Yes	CO_2	CO_2mMET[c,d]	β counting
^{85}Kr	-	-	He	-	Online γ counting
^{129}I	Ag^0-X	Yes[e]	H_2	Pt-X(150°C)	Neutron activation

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a. Heat treatment at 500°C for 8 to 16 hours.

b. Refrigerated to -50°C.

c. Trademark of Amersham/Searle carbon dioxide trapping agent.

d. Preceded by cold trap for H_2O.

e. Heated at 500°C with flowing hydrogen gas.

Irradiated Fuels

Representative fuels with a wide range of burnups from three different light-water reactors, listed in Table II, were used for the tests described in this paper. Each of these fuels was UO_2 and was originally clad with Zircaloy. Fuels from H. B. Robinson-2 and Oconee-1 reactors were from single rods. Saxton fuel was contained in four rods with different irradiation histories.(8)

Similar unirradiated UO_2 fuel was tested in a control experiment to establish lower limits of detection for the gaseous radioactive species.

Table II. Characteristics of Zircaloy-clad UO_2 fuel rods.

Reactor	Rod[a]	Burnup, MWD/MTHM	Cooling Period, yr	Initial ^{235}U Enrichment, %
H. B. Robinson-2	-	∿28,000	3	2.55
Oconee-1	-	∿11,000	3	2.0
Saxton	A	∿6,000	5	12.5
	B	∿3,000	5	12.5
	C	∿3,000	5	12.5
	D	∿100	5	12.5

---

a. Arbitrary designations.

Test Conditions

Voloxidation. About 200 g of irradiated UO_2 fuel in cladding and/or as loose fragments was charged to the voloxidizer. Cladding pieces were from 2.5 to 3.8 cm long. The fuel was heated for 4 hours at 490°C and was tumbled with the rotary agitator at 3.5 rpm. Oxygen content of the recirculating gas was monitored periodically with an inline oxygen analyzer and was held at a nominal 20% in most tests, by adding oxygen as needed. Eleven tests were run with irradiated fuels. Control tests were run with unirradiated fuel and without fuel.

Figure 4 shows the behavior of a typical voloxidation test. Reaction temperature, incremental oxygen additions, and ^{85}Kr evolution were measured as a function of time. In this example, for Saxton fuel, the reaction appeared to be complete after one hour at 490°C. Part of the added oxygen was consumed in the off-gas system by reactions in the heated CuO bed; this fraction was determined in a separate blank experiment and was subtracted from the data to obtain the amount of oxygen consumed by voloxidation.

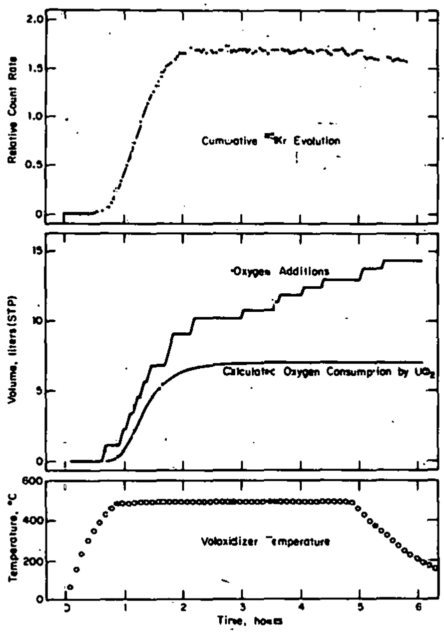

Figure 4. Typical voloxidation behavior (Saxton fuel).

Dissolution. Fuel dissolution was initiated at ambient temperature with a batch quantity of 1 to 3M HNO_3. As the dissolution proceeded, 10M HNO_3 was metered in over 60 to 75 minutes, to slowly increase acid concentration. Ninety minutes after the initial acid charge, the solution temperature was increased to 90°C to complete the dissolution. Gas circulated through the dissolver and off-gas system, and radioactive species were trapped as with voloxidation. Additional steps included leaching the spent cladding hulls and any undissolved residue with 10M HNO_3 and then rinsing with 1 to 3M HNO_3.

Figure 5 shows the behavior of a typical dissolution test. Dissolved uranium concentration and ^{85}Kr evolution were measured as a function of time. The data show that total dissolution time was about 200 minutes and that ^{85}Kr evolution correlated well with the fuel dissolution rate.

Each batch of voloxidized fuel (11 irradiated and 1 unirradiated) and single batches of unvoloxidized Robinson and Oconee fuels were dissolved. A few experiments(5) in a glass apparatus with less complete off-gas facilities are reported for comparison. In a blank experiment, dissolving operations without fuel were also performed.

Dissolver solutions were analyzed for tritium, ^{14}C, and ^{129}I. Negligible ^{14}C or ^{129}I was found in solution. The tritium decontamination factor (DF) for voloxidation was calculated from the amount of tritium in the dissolver solution.

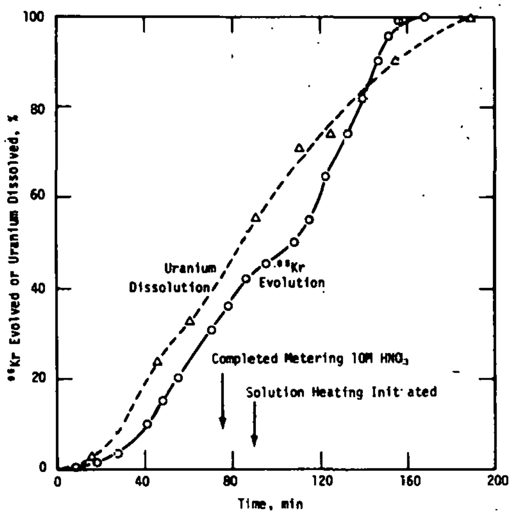

Figure 5. Typical dissolution behavior (Saxton fuel).

Results and Discussion

Tritium

Table III shows, for the cases studied, that nearly all of the tritium in spent fuel evolved during voloxidation, thus confirming the effectiveness of voloxidation for tritium removal. In most of the tests, >99.8% of tritium in the fuel was removed by voloxidation. The small amount of tritium remaining in the fuel after voloxidation was not released to off-gas by dissolution; it was found as HTO in the dissolver solution.

In two tests, the fraction of tritium evolved during voloxidation was somewhat low (87 and 98% released). Conversion of UO_2 to U_3O_8 was incomplete in both tests. In every test with complete conversion to U_3O_8, the tritium DF exceeded 650.

The total amount of tritium for each test is the sum of amounts found in all off-gas traps and in the dissolver solution. Most of the total in each test was found in the trap nearest the voloxidizer, as expected. Duplicate tests with Saxton rods gave good reproducibility. Total tritium from Robinson and Saxton fuels appears to be correlated with burnup, but tritium from Oconee fuel is less than that expected on the basis of burnup.

Table III. Tritium distribution.

	Tritium, Ci/MTU			Voloxidation		
Fuel	Voloxidizer Off-gas	Dissolver Solution	Total	Tritium DF	Tritium, % of Total	HT, %[a]
Robinson	133	0.004	133	33,150	>99.9	0.03
	b,c	250	250	-	-	-
Oconee	6	0.91	7	8[d]	87.5	0.05
	16	0.002	16	6,560	>99.9	0.02
	27	0.013	27	2,170	>99.9	0.01
	7	0.035	7	195	99.5	0.07
	b	35	35	-	-	-
	b,c	7	7	-	-	-
Saxton						
A	31	0.57	32	55[d]	98.2	0.01
B	25	0.027	25	925	99.9	0.1
B	29	0.044	29	665	99.8	0.1
C	28	0.003	28	8,450	>99.9	0.05
C	28	0.007	28	4,150	>99.9	0.03
D	10	0.012	10	805	99.9	0.02
Unirradiated	0.2	0.033	0.2	-	-	-
Blank	1.2	0.008	1.2	-	-	-

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a. Upper limit.

b. Unvoloxidized UO_2 fuel.

c. Dissolved in glass apparatus (Reference 5).

d. Incomplete oxidation.

Tritium in irradiated fuel arises principally from ternary fission and activation of ^{6}Li impurities. Thus, total tritium will depend upon the lithium impurity level and upon irradiation history of the fuel. A calculation with the ORIGEN code(9) for Robinson fuel predicted about 470 Ci/MTU of tritium from fission and a comparable amount from activation. Observed amounts are less. Possible sources of variation in the results include: loss of tritium in earlier operations, such as shearing and storage; differences in burnup along a fuel rod; migration of tritium within a rod, because of thermal gradients; tritium trapped in the cladding.

Carbon-14

From 17 to 22% of the total ^{14}C in the fuels evolved during voloxidation in most of the tests (Table IV). The remainder of the ^{14}C was released to the off-gas system during dissolution. The amounts found were 1000 times greater than the limit of detection for ^{14}C, as determined by the control experiments. Duplicate tests with Saxton fuel showed that the measurements were reproducible.

Somewhat less than the typical ~20% of the ^{14}C evolved during the two voloxidation tests with incomplete conversion to U_3O_8; about 9% was released in these tests. Saxton fuel with very low burnup was another exception, evolving only 2% during voloxidation.

Table IV. Carbon-14 distribution.

Fuel	^{14}C, Ci/MTU Voloxidizer Off-gas	 Dissolver Off-Gas	 Total	Voloxidation ^{14}C, % of Total	 ^{14}CO, %[a]	Dissolution ^{14}CO, %[a]
Robinson	0.066	0.243	0.309	21.3	59	0.04
Oconee	0.010	0.104	0.114	9.1[b]	22	72
	0.025	0.124	0.149	16.8	27	65
	0.024	0.104	0.128	18.7	2	31
	0.035	0.141	0.176	19.7	51	24
	c	0.081	0.081	-	-	0.2
Saxton						
A	0.0037	0.0389	0.0426	8.7[b]	0.07	0.14
B	0.0055	0.0235	0.0290	19.1	75	29
B	0.0068	0.0234	0.0302	22.4	51	58
C	0.0053	0.0216	0.0269	19.7	64	29
C	0.0047	d	d	d	69	d
D	0.00001	0.0005	0.0005	2.0	76	4
Unirradiated	0.000005	0.00005	0.00005	-	50	55
Blank	0.000005	0.00003	0.00003	-	50	20

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a. Upper limit (see text).

b. Incomplete oxidation.

c. Unvoloxidized UO_2 fuel.

d. Not determined.

^{14}C in irradiated fuels is formed primarily by the $^{14}N(n,p)^{14}C$ reaction on nitrogen impurities in the fuel and secondarily by the $^{17}O(n,\alpha)^{14}C$ reaction. Thus, the amount of ^{14}C produced depends upon the amount of nitrogen impurity and upon fuel burnup. A typical calculated value is 0.5 Ci/MTU for fuel with 20-ppm nitrogen irradiated to about 30,000 MWD/MTHM. The amounts of ^{14}C measured in Robinson, Oconee, and Saxton fuels are correlated with the burnups and are in the range expected for 10 to 20 ppm nitrogen in the fuels.

Krypton-85

Table V shows that, in most of the tests, 7 to 17% of the total ^{85}Kr in the fuels evolved during voloxidation. The dissolution tests confirmed that the major fraction of ^{85}Kr remained in voloxidized fuel but was totally released by dissolving. Duplicate tests with Saxton fuel agreed well. Again, somewhat lower results (4 to 6% released during voloxidation) were obtained for the two tests with incomplete conversion to U_3O_8.

The small fraction of ^{85}Kr from voloxidation agrees with results of other workers with different fuels.(4) Apparently, krypton from fission is dispersed throughout the fuel as individual atoms rather than accumulated as bubbles at the grain boundaries. Oxidation of UO_2 does not release much of the krypton from the matrix, even though a very fine U_3O_8 powder is formed. Since tritium is released completely under the same conditions, the results imply that krypton diffuses much slower than tritium in the U_3O_8 crystal lattice.

Table V. Krypton-85 distribution.

Fuel	^{85}Kr, Ci/MTU Voloxidizer Off-Gas	 Dissolver Off-Gas	 Total	Voloxidation ^{85}Kr, % of Total
Robinson	137	1651	1788	7.7
Oconee	41	969	1010	4.1[a]
	123	1087	1210	10.2
	77	1011	1088	7.1
	115	1058	1173	9.8
	b	1070	1070	-
Saxton				
A	62	944	1006	6.2[a]
B	75	377	452	16.6
B	62	396	458	13.5
C	60	388	448	13.4
C	61	c	c	c
D	0.7	5.1	5.8	11.3
Unirradiated	0.07	1.00	1.07	-
Blank	0.07	0.08	0.15	-

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a. Incomplete oxidation.

b. Unvoloxidized UO_2 fuel.

c. Not determined.

Total ^{85}Kr in fuel is related to burnup, cooling period, and perhaps previous thermal history. A calculation with the ORIGEN code predicted about 7000 Ci/MTU of ^{85}Kr for the Robinson fuel. The amounts of ^{85}Kr measured in Robinson, Oconee, and Saxton fuels correlate well with burnups but are somewhat lower than calculated values. An unknown amount of krypton may have been released on initial puncture of the rods and subsequent storage of the fuel.

Iodine-129

Table VI gives results for ^{129}I collected during voloxidation and/or dissolution of the fuels. The data are less consistent than for the other radioactive gases, possibly because of experimental difficulties. We conclude from our experience with these tests that separate experiments designed specifically to measure iodine will yield more accurate results.

In all of the tests, very little ^{129}I (from 0.02 to 1.7 g/MTU) was collected during voloxidation. These amounts also were small fractions of total ^{129}I found, ranging from 0.1 to 7.9% evolved during most of the voloxidation tests. In two tests, the fractions from voloxidation were 59 and 84%; however, material balances for iodine in these two tests did not agree well with amounts expected.

Table VI. Iodine-129 distribution.

	^{129}I, g/MTU			Voloxidation
Fuel	Voloxidizer Off-gas	Dissolver Off-gas	Total	^{129}I, % of Total
Robinson	0.18	58	58	0.3
	a,b	171	171	-
Oconee	0.03	27	27	0.1
	0.76	31	32	2.4
	1.7	20	22	7.9
	a,b	104	104	-
	a,c	102	c	c
Saxton				
A	1.5	27	28	5.4
B	0.16	0.03	0.19	84
C	0.03	c	c	c
C	0.02	c	c	c
D	1.1	0.8	1.9	59
Unirradiated	0.007	0.016	0.023	-
Blank	0.014	0.009	0.023	-

---

a. Dissolved in glass apparatus (Reference 5).

b. Unvoloxidized UO_2 fuel.

c. Not determined.

Also shown in Table VI are previously reported results(5) for Robinson and Oconee fuels dissolved in a glass apparatus; the iodine was collected in an NaOH off-gas scrubber. About three times more ^{129}I was obtained with the glass apparatus than with the metal equipment used in this work. Reasons for the difference have not been determined, although results from the glass apparatus are believed to be more accurate. The earlier work showed little difference in ^{129}I evolved during dissolution of voloxidized or unvoloxidized Oconee fuel; this is consistent with the fraction of ^{129}I evolved during voloxidation being small.

Total ^{129}I should be proportional to burnup only. A typical calculated value for Robinson fuel is 187 g/MTU.

Chemical Species

Upper limits on the amounts of tritium evolved as HT and ^{14}C evolved as ^{14}CO were determined from tritium or ^{14}C found in the traps downstream from the oxidizing bed of CuO. Table III gives the fraction of tritium from voloxidation found in the downstream tritium trap. Table IV gives similar information for ^{14}C from both voloxidation and dissolution.

The results show that nearly all of the tritium from voloxidation was in the form of HTO, as expected. Less than 0.1% could have been in the form of HT. Although these amounts are 10 to 100 times greater than the background level of the system, they are within the range of possible leakage from upstream traps and therefore should be regarded as upper limits for HT. From dissolution of voloxidized fuel, no tritium as HT was observed; only background levels of tritium were found in the downstream trap.

The distribution between $^{14}CO_2$ and ^{14}CO is uncertain. During the required conditions of prolonged flow, some CO_2 desorbed from the upstream trap and collected in the downstream trap. This effect was shown in a calibration test with $^{14}CO_2$; the ^{14}C was found equally distributed between the two traps even though no ^{14}CO was in the system. In voloxidation tests, up to 76% (in a test with Saxton fuel) of the ^{14}C was found in the downstream trap. In dissolution tests, up to 72% (in a test with Oconee fuel) of the ^{14}C was found in the downstream trap. Because an unknown part of the ^{14}C on this trap was originally $^{14}CO_2$ and not ^{14}CO, the values shown represent only upper limits on ^{14}CO; the actual amounts could have been much less. In one test with Saxton fuel, only about 0.1% of the ^{14}C from either voloxidation or dissolution appeared as ^{14}CO. Total ^{14}C evolved was accurately measured because the total capacity of the upstream and downstream traps for CO_2 was adequate for the quantities involved.

Conclusions

From the data of Tables III - VI, we conclude the following:

- Voloxidation of spent nuclear fuel releases tritium to the off-gas system quantitatively but releases only small fractions of the ^{14}C, ^{85}Kr, and ^{129}I in the fuel.
- Dissolution of voloxidized fuel in nitric acid releases the remaining ^{14}C, ^{85}Kr, and ^{129}I to the off-gas system.
- Typical values for the fractions evolved during voloxidation are >99.8% of the tritium, 17 to 22% of the ^{14}C, 7 to 17% of the ^{85}Kr, and <8% of the ^{129}I.
- Tritium is evolved as HTO during voloxidation; less than 0.1% is HT.

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