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Behavior of Fission Product Iodine in the Head-end Reprocessing of HTGR Fuel: Iodine-129 Studies (Series I)

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[redacted]

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BEHAVIOR OF FISSION PRODUCT IODINE IN THE HEAD-END REPROCESSING OF HTGR FUEL: IODINE-129 STUDIES (SERIES I)

C. E. Lamb
C. L. Fitzgerald
V. C. A. Vaughen

Chemical Technology Division

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BEHAVIOR OF FISSION PRODUCT IODINE IN THE HEAD-END REPROCESSING OF HTGR FUEL: IODINE-129 STUDIES (SERIES I)

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ABSTRACT

The transport of ^{129}I in the head-end reprocessing of High-Temperature Gas-Cooled Reactor (HTGR) fuel was investigated using fuel removed from the Peach Bottom Reactor. This fuel consisted of TRISO-coated $(Th,U)C_2$--TRISO-coated ThC_2 microspheres in a graphite matrix.

A modified grind-burn-leach flowsheet was selected to simulate the secondary burner step. To minimize the number of samples required to study the iodine behavior, the fissile and fertile fractions were crushed and processed together. Filters and suitable sorbers were positioned in the separate parts of the system for characterization purposes. Sintered Hastelloy X and high-efficiency particulate air (HEPA) absolute filters were used to remove entrained particles, whereas activated charcoal and silver-exchanged zeolite were chosen as the iodine sorbents.

The fission-product data resulting from radiochemical analysis and gamma radiation measurements were compared with values predicted by the ORNL Isotope Generation and Depletion Code (ORIGEN). About 15-50% of the total iodine found remained with the Hastelloy X filter, and about 5-20% remained with the ash removed from the burner. Approximately 99% of the iodine in the gas phase leaving the burner housing was sorbed by the first activated-charcoal trap. The dissolved burner ash and insoluble residue also contained approximately 99% of the ^{144}Ce and more than 90% of the ^{134}Cs, ^{137}Cs, and ^{154}Eu.

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1. INTRODUCTION

The first set of experiments in a series designed to study the release and transport of ^{129}I in the head-end reprocessing of High-Temperature Gas-Cooled Reactor (HTGR) fuel was completed. Highly radioactive fuel removed from the Peach Bottom HTGR was used in these experiments. This fuel was composed of TRISO-coated $(Th,U)C_2$--TRISO-coated ThC_2 microspheres embedded in a graphite matrix. The microspheres

were fabricated with an inner core of $(Th,U)C_2$ or ThC_2 surrounded by a layer of low-density pyrolytic carbon, a layer of high-density pyrolytic carbon covered by a layer of SiC, and an outer shell of high-density pyrolytic carbon.

The head-end process for the HTGR fuel consists of (1) steps to crush and burn the matrix graphite, separate the fissile particles from the fertile particles, and process each fraction separately through a secondary crush to open the microspheres, (2) a secondary burn to remove the exposed carbon and oxidize the carbides of uranium and thorium, and (3) a dissolution in Thorex reagent (13 $\underline{M}$ HNO_3, 0.1 $\underline{M}$ Al^{3+}, 0.05 $\underline{M}$ F^-) to recover the heavy metal oxides. To minimize the number of samples required to study the iodine behavior, the fissile and fertile fuel fractions were processed together using a grind-burn-leach flowsheet;[1] this simulated the secondary burner operation in the process.

Although the primary goal was to study the behavior of iodine, the results were also obtained for other fission products and heavy metals and were compared with values calculated by the ORNL Isotope Generation and Depletion Code (ORIGEN), a computer program used for predicting quantities of activation products, actinides, and fission products from irradiation data and nuclear data libraries.[2]

The experiments were performed in duplicate to provide a basis for a comparative study. A complete fuel rod from General Atomic Company (GAC) Fuel Test Element (FTE)-16 was used in each experiment. Each rod was ground to a fine powder, thus blending the fissile and fertile particles. The grinding was performed in an argon atmosphere because the carbide fuel is pyrophoric.

After completion of the experiment, the equipment was disassembled into basic components. The sorbers and filters were isolated and packaged for analysis. The ashes were removed from the burner, weighed, and transferred into a dissolver vessel, where they were leached with Thorex reagent. During the leaching process, the solution was purged with a stream of air. The gas was discharged through a solution of NaOH to trap any evolved iodine. These scrubber solutions, along with the insoluble microsphere hulls, sorbers, and filters, were submitted to the Analytical Chemistry Division laboratories for analysis. A radiochemical separation and activation technique was used to determine the ^{129}I, whereas gamma-ray

spectrometry was employed to identify other fission products present in sufficient quantities for detection.

2. EXPERIMENTAL

2.1 Fuel Description and Irradiation Data

The fuel used in these experiments was of the Fort St. Vrain HTGR type and was irradiated in the Peach Bottom Reactor for 512 effective full-power days (EFPD) between January 7, 1972, and October 31, 1974. The fuel rods were composed of TRISO-coated $(Th,U)C_2$--TRISO-coated ThC_2 microspheres embedded in a graphite matrix; each rod contained 3.0 g of thorium and 0.5 g of uranium (preirradiation).[3] Each rod was cylindrical (12 mm in diameter and 50 mm in length) and weighed approximately 11.7 g.

All radioactivity measurements and ORIGEN nuclide values were corrected for decay to October 1, 1977. (Postirradiation examination data are to be published by GAC.)[4]

2.2 Equipment

Cell 1 in the High-level Chemical Development Facility was used in this study. The cell was equipped with in-line instrumentation to support the experimental system (Fig. 1). The layout consisted of metered gas supply lines, temperature controllers, off-gas monitors, CO/CO_2 detectors, radiation monitors, and gas collection equipment. It was interfaced with subassemblies for grind, burn, and leach operations (Fig. 2).

The subassembly for grinding the fuel consisted of a standard commercial blender (Fig. 3) modified with a gas supply and exhaust line to carry any iodine liberated during the operation to an activated-charcoal adsorber and an absolute filter. The discharged gas was collected and analyzed at the end of the experiment.

The burner subassembly was constructed of stainless steel and consisted of a burner vessel (mounted in a well-type furnace) designed to contain the fuel rod during the burn operation. Preheated inlet gas was supplied through a small stainless steel frit welded in a position about 1 cm above

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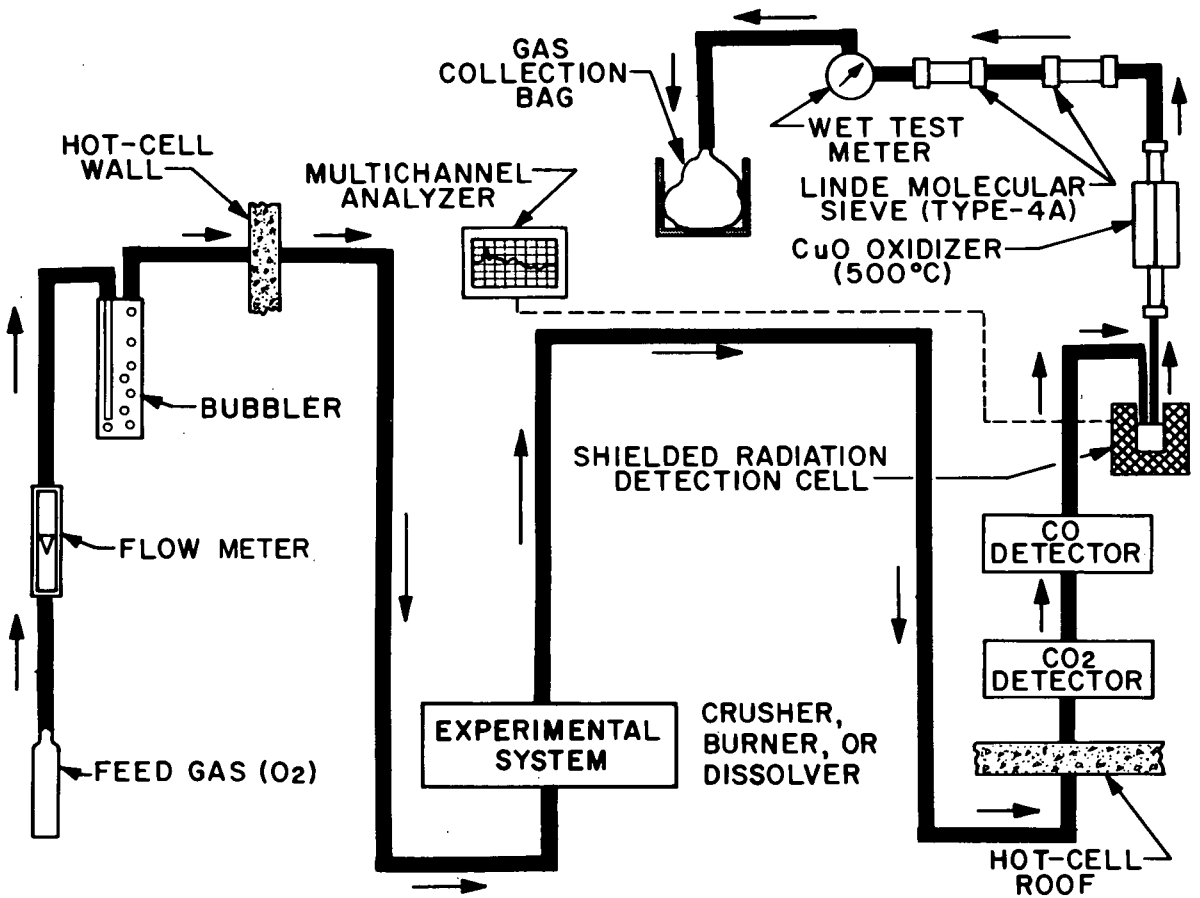

Fig. 1. In-line equipment supporting the experimental system.

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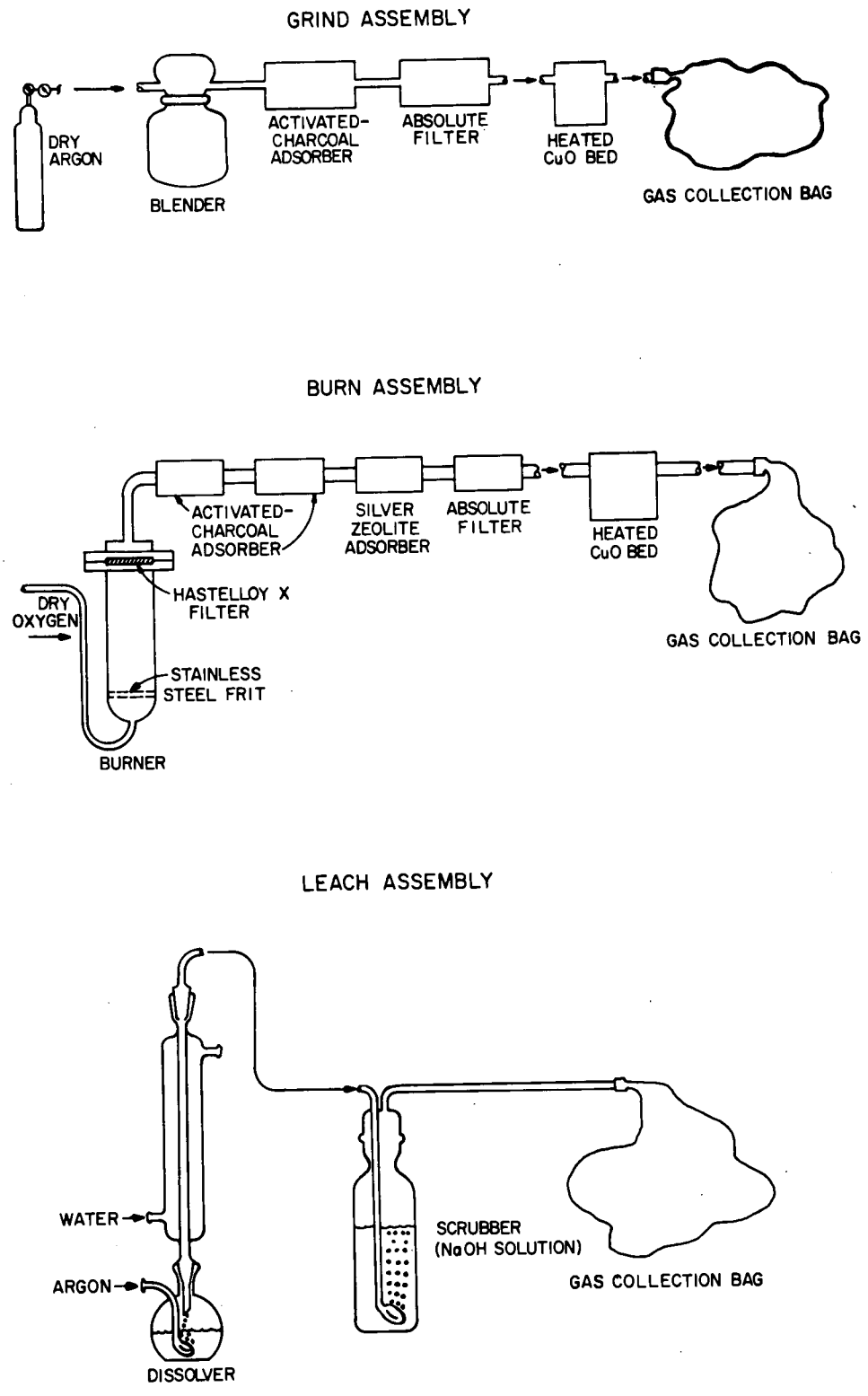

Fig. 2. Equipment layout.

Fig. 3. Sample grinder/blender with purge gas-line connectors.

the bottom of the vessel. The frit served to diffuse the incoming gas and support the fuel material during combustion. A flange at the top of the burner was designed with annular recesses to hold a sintered Hastelloy X filter (with 10-μm-diam pores) and gold-plated gaskets. Thermocouples were positioned in thermowells attached inside and outside the burner to monitor the temperatures during the reaction.

A stainless steel tube connected to the top of the burner was employed to carry the exhaust gases from the reaction chamber through two activated-charcoal sorbers in series, a silver-exchanged zeolite sorber, an absolute filter, a heated CuO bed, and finally into a gas collection container. The first charcoal sorber was located at that point downstream of the burner at which the temperature was below 150°C. This was done to avoid ignition of the sorber bed. (A temperature profile of the burner assembly during operation at 850°C is shown in Fig. 4.) The activated-charcoal and silver-exchanged zeolite sorber beds were fabricated from stainless steel pipe; the zeolite assemblies were smaller versions of that shown in Fig. 5. The charcoal employed was type G618 (5% triethylenediamine impregnated), which was obtained from North American Carbon, Inc., Columbus, Ohio. The silver-exchanged zeolite sorber beds were fabricated using 99% AgX on 1.6-mm pellets; these were obtained from Union Carbide Corporation, Tarrytown, New York. The absolute filters employed in the study were pretested, disposable, polypropylene-encased HEPA filters (No. MBY 3001 URA) made by Pall, Trinity, Inc.

A third subassembly consisted of a glass dissolution vessel with an inlet gas line and a reflux condenser. The reflux condenser was connected to a "scrubber" vessel containing NaOH solution to remove any radioactivity entrained in the off-gas leaving the reflux condenser.

2.3 Operation

2.3.1 Grind

The fuel rod selected for each experiment was handled with care to avoid contamination with nuclides from the cell environment. The blender container and the off-gas line connectors were replaced after each test. The blender was conditioned by grinding 1 g of unirradiated graphite before adding the fuel rod. The graphite effectively plugged pits and crevices inside the container, thereby reducing fuel losses to these areas when transferring the blended fuel to the burner.

ORNL DWG 78-17814

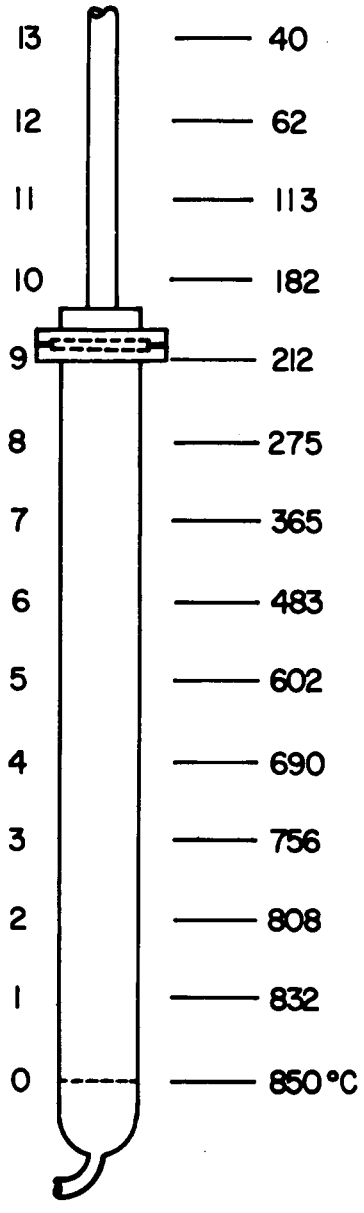

Fig. 4. Burner assembly temperature profile.

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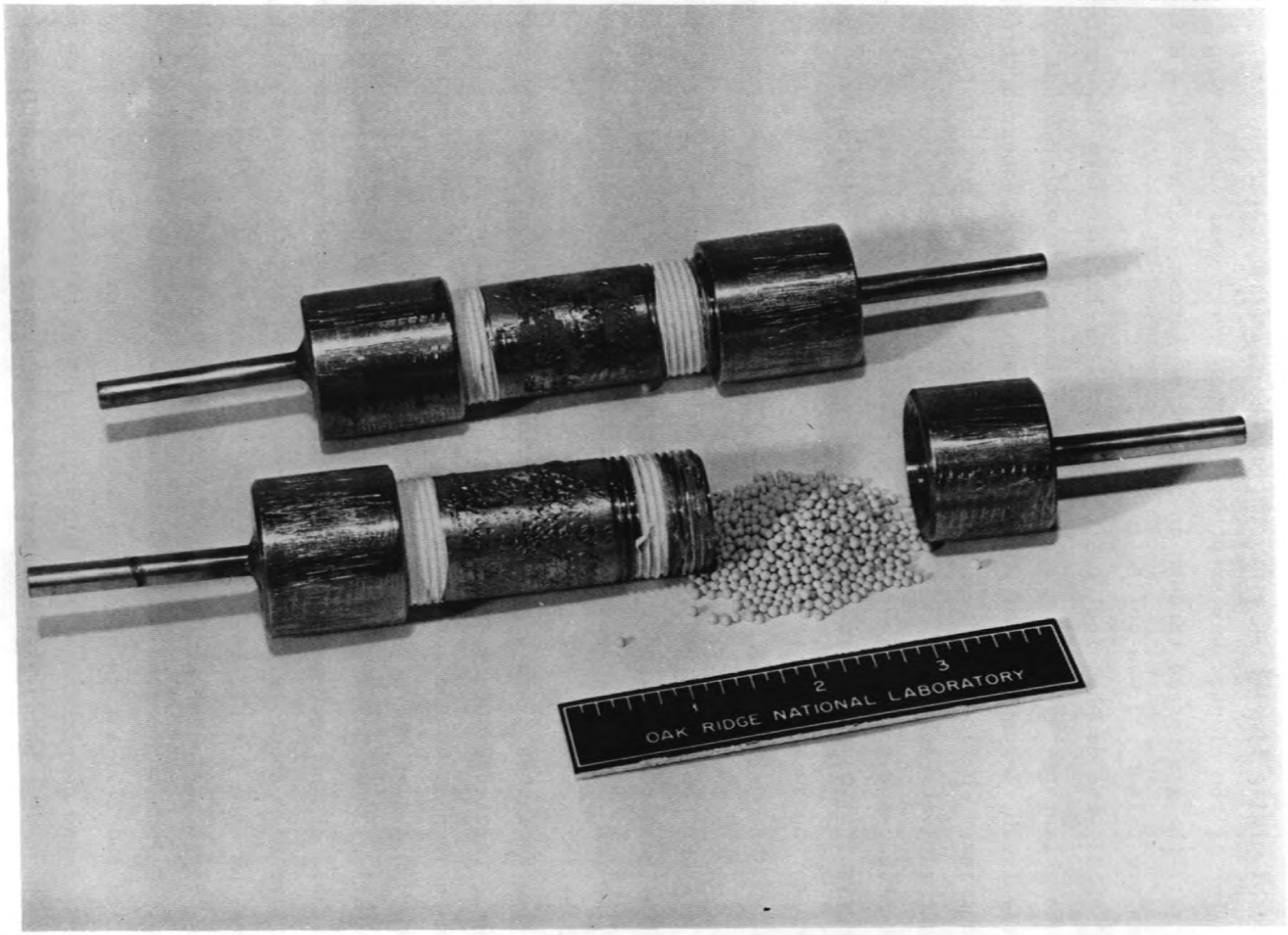

Fig. 5. Silver-exchanged zeolite sorber.

The fuel was ground for 2 min, followed by a 2-min period with the motor off to prevent overheating the bearings. The cycle was repeated for a total mixing time of 30 min. During the grinding operation, the inner chamber of the blender was swept by an argon purge gas. On leaving the vessel, the gas passed through an activated-charcoal sorber and a HEPA absolute filter and was collected in a plastic bag.

While the crushed fuel was poured from the blender into the burner, it was blanketed by flowing argon to minimize contact with air and moisture. Three fresh, 1-g charges of unirradiated graphite were processed through the grinder to flush any remaining fuel into the burner for a more complete quantitative transfer.

Figures 6 and 7 illustrate the work flow for experiments FTE-16-1-3 and FTE-16-1-4, respectively. Sampling points are identified by the codes used for those samples removed from each location.

2.3.2 Burn

After the system was tested and found to be leak proof, the temperature in the burner was raised to 850°C and the oxygen flow rate was adjusted to 100 cm^3/min. The burn was normal in both runs and lasted approximately 3.5 hr in experiment FTE-16-1-3 and approximately 4 hr in experiment FTE-16-1-4. Figures 8 and 9 show the burner performance in each experiment. A well-regulated burn is depicted by Fig. 8; the burner temperature remained constant and the released CO_2 reached a peak concentration of 96% after 50 min of operation, then evenly decreased to the end of the operation. The decrease of the CO_2 concentration in the off-gas indicated that all of the carbon present had been burned, and the experiment was terminated. About 10.3 g of carbon were burned and 27.5 liters of oxygen were consumed for FTE-16-1-3. A more irregular burn is illustrated by Fig. 9; the temperature fluctuated between 800 and 900°C and the CO/CO_2 evolved in a correspondingly erratic manner. About 11.4 g of carbon were burned and 27.1 liters of oxygen were consumed for FTE-16-1-4.

At the completion of the burning operation, the burner ash was removed and the sorber beds, HEPA and Hastelloy X filters, and gas samples were sent for radiochemical analysis.

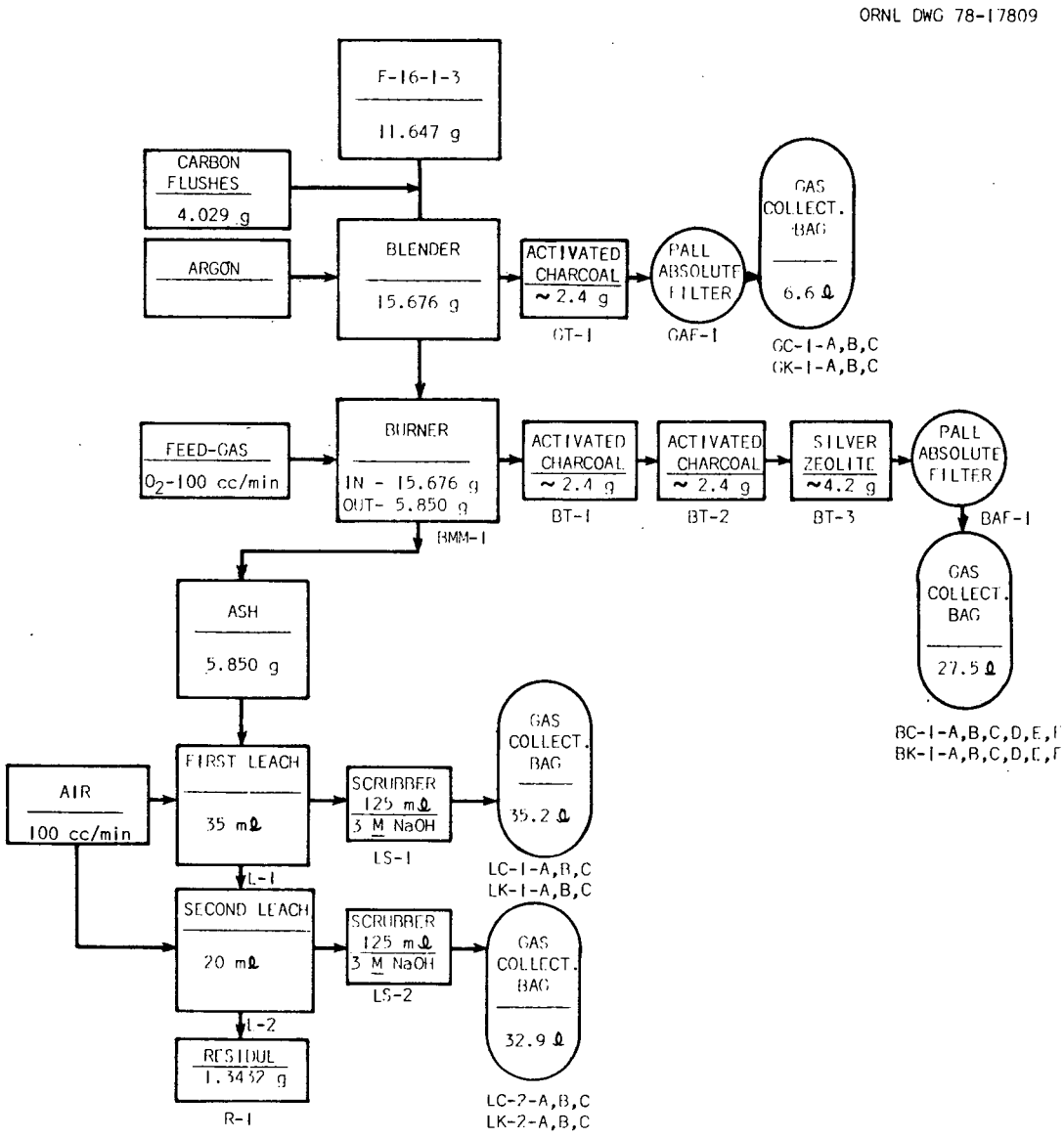

Fig. 6. Flowsheet for experiment FTE-16-1-3.

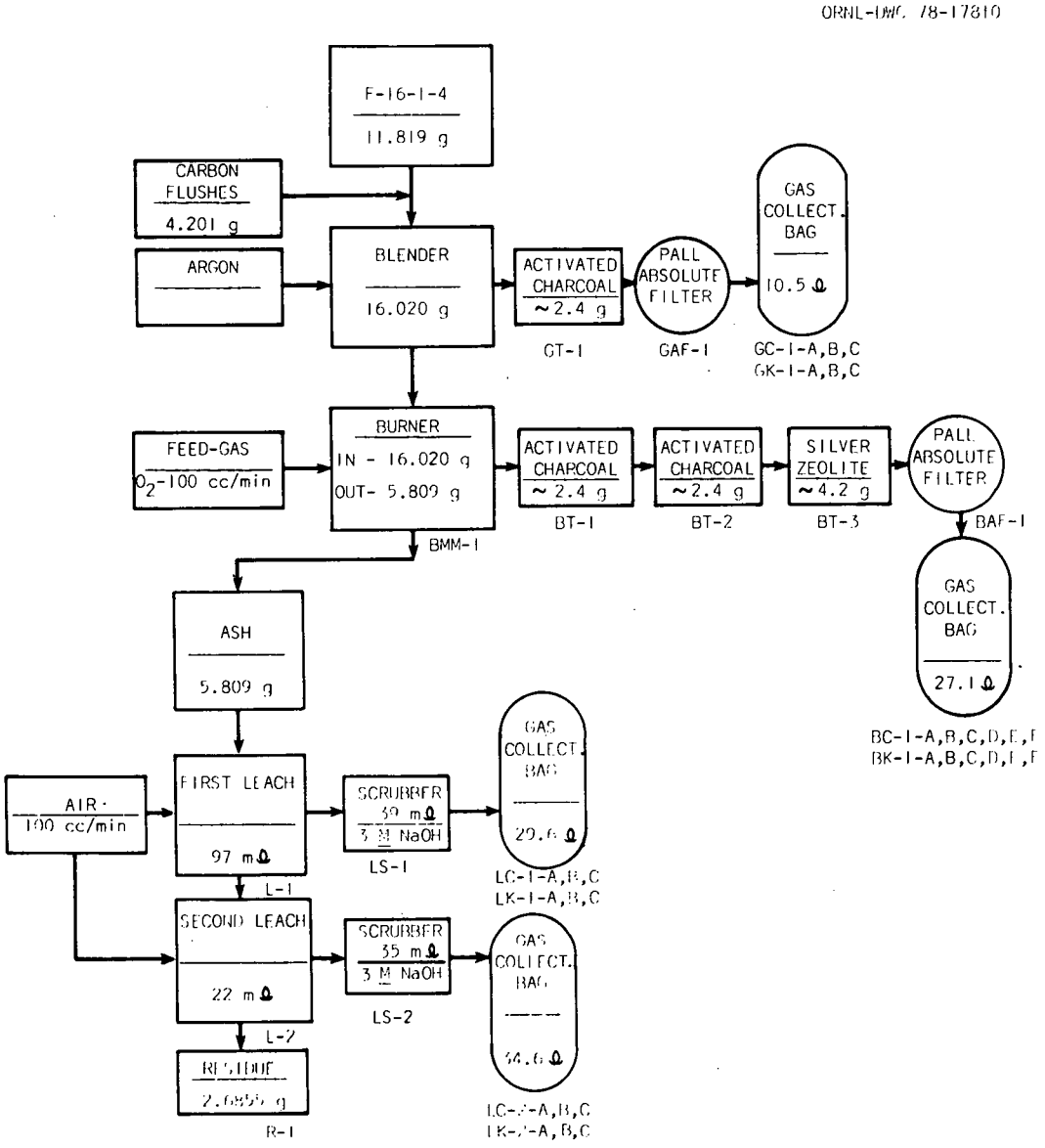

Fig. 7. Flowsheet for experiment FTE-16-1-4.

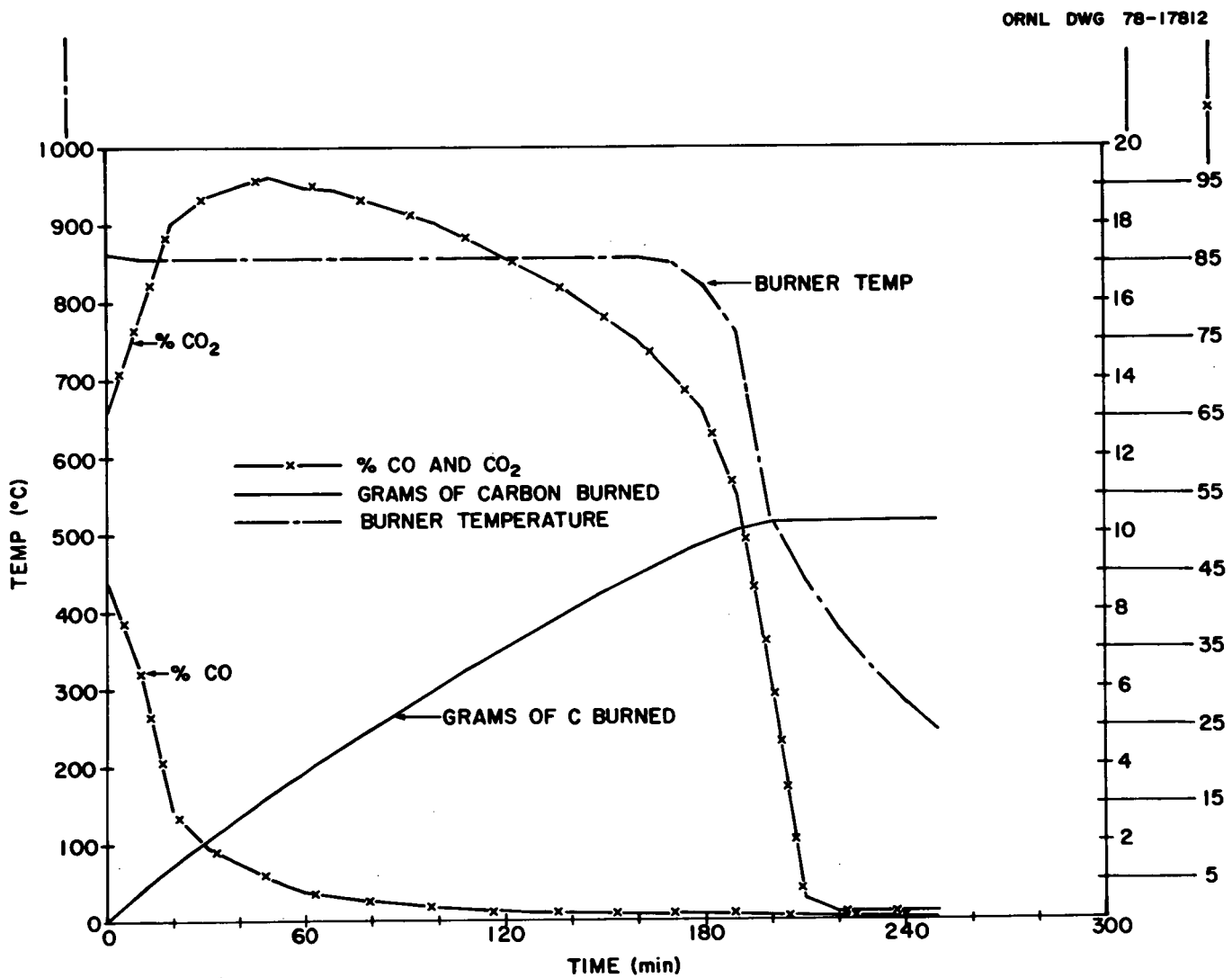

Fig. 8. Burner performance in experiment FTE-16-1-3.

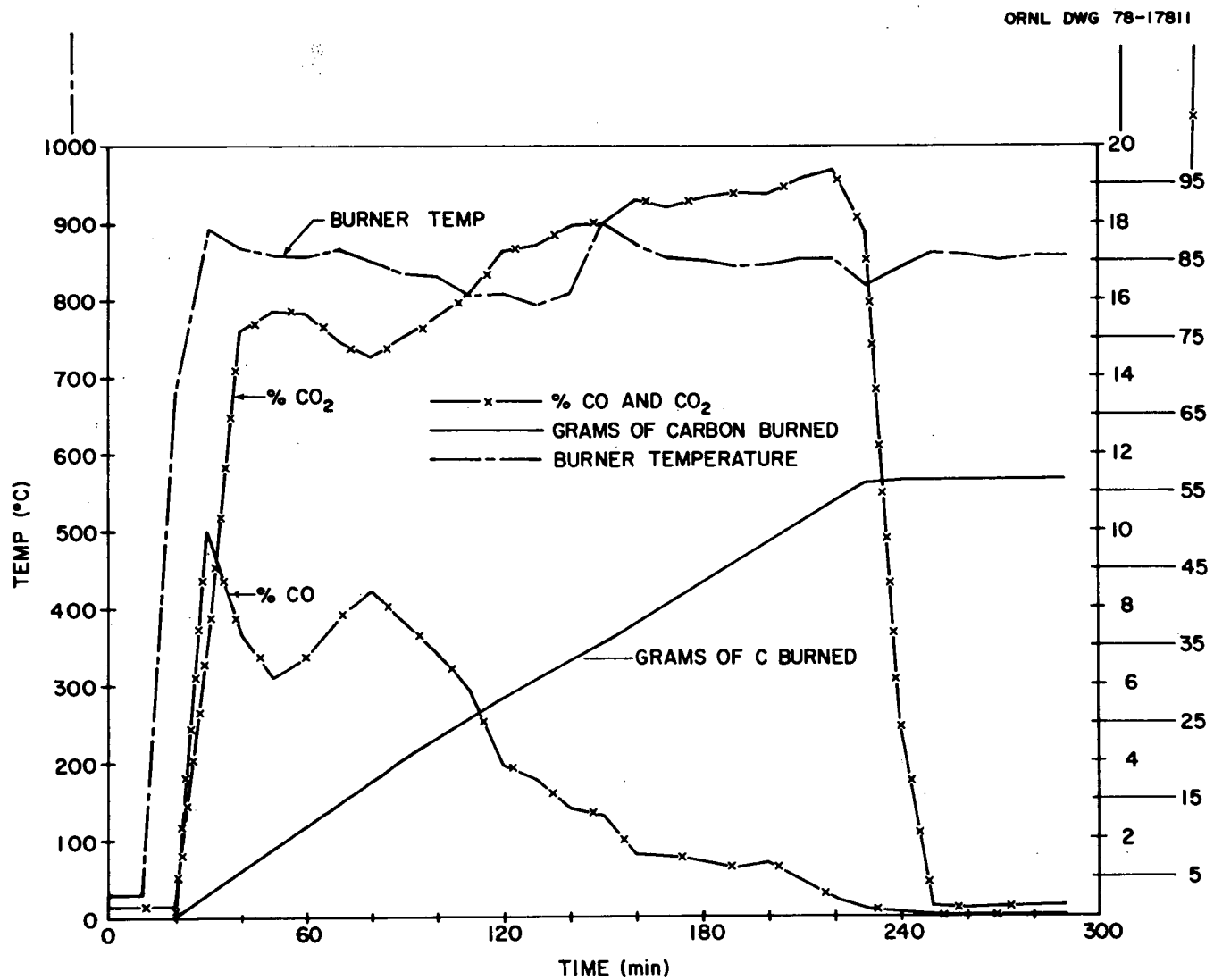

Fig. 9. Burner performance in experiment FTE-16-1-4.

2.3.3 Leach

The ash from the burner contained the metal oxides, SiC hulls, and nonvolatile fission products. After it was weighed and transferred into the dissolver vessel, a reflux condenser was inserted with a line attached to carry the off-gas to a scrubber and gas collector. A line connecting an air supply to the dissolver was attached and the assembly was checked for leaks. The air supply was then adjusted to a 100-cm^3/min flow to purge the ^{85}Kr. An electric heating mantle encasing the dissolver vessel was adjusted to heat the solution to a gentle boil. After a 6-hr contact, the heat to the dissolution was terminated; the air purge was continued for an additional hour.

The leach assembly was dismantled and the solution was filtered to separate the undissolved material for further treatment. Samples of the filtrate and collected gas were removed for analysis.

The remaining undissolved material was returned to the dissolver and a second contact was made using the same set of conditions. At the end of the second dissolution, additional samples were removed for analysis. They were submitted along with the final undissolved material and the previously separated samples to the Analytical Chemistry Division laboratories for analysis.

3. RESULTS AND DISCUSSION

3.1 Heavy Metals

The distributions of the uranium and thorium concentrations in the two experiments are listed in Table 1. The results indicate that 80% of the uranium and 84% of the thorium concentrations predicted by ORIGEN were identified in experiment FTE-16-1-3, while only 65% of the uranium and 74% of the thorium were found in FTE-16-1-4. These ratios may indicate the uncertainty of the fuel loading. The burner ash contained the majority of the heavy metals found in both experiments: 87.7% of the uranium and 89.2% of the thorium in experiment FTE-16-1-3 and 93.4% of the uranium and 96.6% of the thorium in experiment FTE-16-1-4. The Hastelloy X filter held <0.1% of the uranium and thorium in experiment FTE-16-1-3 and only 0.1% of the uranium and 0.3% of the thorium in FTE-16-1-4.

Table 1. Distribution of heavy metals

	FTE-16-1-3				FTE-16-1-4			
	Uranium		Thorium		Uranium		Thorium	
	mg	%	mg	%	mg	%	mg	%
Hastelloy X filter	<0.1	<0.1	1.8	<0.1	0.3	0.1	5.6	0.3
First leach solution	290.2	87.7	2209	89.2	253.2	93.4	2112	96.6
Off-gas scrubber	<0.1	<0.1	<0.2	<0.1	<0.1	<0.1	<0.1	<0.1
Second leach solution	<0.1[a]	<0.1[a]	266	10.7	15.8	5.8	38.9	1.8
Off-gas scrubber	37.3[a]	11.3[a]	<0.2	<0.1	<0.1	<0.1	<0.1	<0.1
Insolubles	3.4	1.0	0.3	<0.1	1.8	0.7	29.4	1.3
Total (CA)	330.9	100	2477	100	271.1	100	2186	100
ORIGEN (O)	415		2940		415		2940	
CA/O[b]	0.80		0.84		0.65		0.74	

[a]Suspect results; scrubber and leach results are probably reversed.
[b]Chemical analysis/ORIGEN ratio.

With 10.7% of the total thorium in FTE-16-1-3 and 5.8% of the total uranium in FTE-16-1-4 found in the second leach, a more concerted effort to improve the first leach efficiency seems in order. However, the combination of the two leaches in each experiment removed all of the heavy metals except 0.1-1% in FTE-16-1-3 and 0.7-1.3% in FTE-16-1-4. Less than 0.1% of the heavy metals was carried in the off-gas to both the first and second leach off-gas scrub solutions. (However, see Sect. 3.3.)

Based on the heavy metal values in the leached residues and assuming that all of the uranium and thorium in these residues came from unbroken microspheres, only 1.3% or less of the microspheres could have remained intact through the crush and burn operations. (However, see Sect. 3.3.)

The data in Table 2 compare the quantity of fuel ash removed from the burner in each experiment with the amount recovered from the dissolution step. One expects all values to be proportionate to the quantities of burner ash. While there are variations between the two sets of data, the largest discrepancy is the weight of SiC hulls. A 1-g weighing error could have occurred while weighing the SiC hulls in experiment FTE-16-1-3, thus accounting for most of the difference in the material balances.

The isotopic uranium values and those predicted by ORIGEN are compared in Table 3. The chemical analysis/ORIGEN (CA/O) ratios were 0.80 for experiment FTE-16-1-3 and 0.65 for experiment FTE-16-1-4, with the greatest disparity occurring in the concentration of ^{235}U: 67% of ORIGEN's prediction for FTE-16-1-3 and 55% for FTE-16-1-4. No explanation is available at this time.

3.2 Iodine Behavior

The radiochemical determination of ^{129}I involved irradiating a representative fraction of the sample in a reactor and measuring the resulting ^{130}I (ref. 5). For safe handling, therefore, it was first necessary to treat the highly radioactive sample in a hot cell to separate the ^{129}I from the other fission products. Four types of samples were analyzed for ^{129}I content: (1) solids (SiC hulls), (2) metals (Hastelloy X filter), (3) liquids (leach and scrub solutions), and (4) granules and pellets (activated-charcoal and silver-exchanged zeolite sorbers). Each type required a different treatment.

Table 2. Material balances

	FTE-16-1-3 (g)	FTE-16-1-4 (g)	Theoretical[a] (g)
Burner ash (weight before dissolution)	(5.850)	(5.809)	
Uranium	0.331	0.271	0.50
Thorium	2.477	2.186	3.00
Oxide oxygen	0.401	0.345	0.50[b]
Insolubles (SiC)	1.343	2.686	1.55
Fission products	0.140[c]	0.140[c]	———
	4.682	5.628	5.550
Material balance, %	80	97	

[a]Preirradiation.
[b]Calculated as postburn oxide.
[c]Calculated by ORIGEN code.

Table 3. Comparison of isotopic uranium values by ORIGEN and chemical analysis[a]

Isotope	ORIGEN[b] (g)	FTE-16-1-3		FTE-16-1-4	
		Chemical analysis[c] (g)	CA/O ratio[d]	Chemical analysis[c] (g)	CA/O ratio[d]
^{233}U	0.047	0.054	1.15	0.044	0.94
^{234}U	0.005	0.006	1.20	0.005	1.00
^{235}U	0.303	0.203	0.67	0.167	0.55
^{236}U	0.033	0.042	1.27	0.034	1.03
^{238}U	0.027	0.026	0.96	0.021	0.78
	0.415	0.331	0.80	0.271	0.65

[a]TRISO-coated $(Th,U)C_2$--TRISO-coated ThC_2 fuel.
[b]ORIGEN (1066-day cooled), October 1, 1977.
[c]Mass spectrometry.
[d]Chemical analysis/ORIGEN ratio.

The hulls were slowly dried at ambient temperature. They were then weighed into a platinum dish and fused in a flux of Na_2CO_3. Any ^{129}I in microspheres remaining intact would probably escape to the cell environment during this fusion treatment, along with the undissolved SiC hulls. After cooling, the salt was dissolved in dilute HCl solution. The Hastelloy X filter was dissolved in aqua regia. The activated-charcoal adsorbers were leached with a solution of NH_4OH and H_2O_2 to remove the ^{129}I. The silver-exchanged zeolite sorber pellets were crushed and leached with a slightly heated solution of NaOH and Na_2S. Each leach solution was then treated as described below.

To effect the ^{129}I separation, a portion of each solution was spiked with a known amount of ^{127}I. It was then treated in sequential steps with the addition of solutions containing NaOCl, HNO_3, and $NH_2OH \cdot HCl$ to prepare the iodine for extraction into CCl_4. Most of the fission products remained in the aqueous phase. The iodine was then stripped from the CCl_4 with a solution of $NaHSO_3$. The technique usually resulted in only a partial separation of the iodine from the fission products. It was routinely necessary to repeat the steps several times.

An aliquot of the prepared solution was then irradiated in the Bulk Shielding Reactor (BSR) to activate the ^{129}I present to ^{130}I. A gamma spectrometer with a high-resolution Ge(Li) detector was then used to measure the ^{130}I produced. A correction based on the original addition of ^{127}I was made and the result for ^{129}I was reported.

The analytical data showing the distribution of ^{129}I in experiments FTE-16-1-3 and FTE-16-1-4 are listed in Table 4. Examination of the results revealed that most of the ^{129}I was found in the first activated-charcoal adsorber, Hastelloy X filter, and burner ash (represented by the values corresponding to the first and second leach). About 18-28% of the ^{129}I predicted by ORIGEN was found in these two experiments. This compares with about 2-7% of the predicted quantities found in two previously completed experiments using FTE-4 HTGR fuels.[1] The low material balance suggests that the iodine is deposited in an unidentified location (e.g., one such location may be the inner surfaces of the burner).

In experiment FTE-16-1-3, about 5% of the total iodine was determined to be in the burner ash. Approximately 14% of the iodine was in the sintered Hastelloy X filter and about 80% was on the first activated-

Table 4. Distribution of ^{129}I found after each experiment

Operation	Sampling point	Iodine-129 distribution			
		FTE-16-1-3		FTE-16-1-4	
		μg	%	μg	%
Grind	Activated-charcoal adsorber	<0.1	<0.1	<0.1	<0.1
	Absolute filter	a	a	a	a
Burn	Hastelloy X filter	23.0	14.2	54.5	52.0
	Off-gas scrubber (from filter dissolution)	<0.1	<0.1	<0.1	<0.1
	Activated-charcoal adsorber 1	130.0	80.3	30.0	28.7
	Activated-charcoal adsorber 2	0.5	0.3	<0.1	<0.1
	Silver zeolite adsorber	0.3	0.2	<0.1	<0.1
	Absolute filter	a	a	a	a
Leach	First solution	1.8	1.1	18.5	17.7
	Off-gas scrubber solution	3.5	2.2	0.7	0.7
Leach	Second solution	1.5	0.9	0.8	0.8
	Off-gas scrubber solution	1.4	0.8	0.1	0.1
Insolubles		≤0.1	<0.1	≤0.1	<0.1
Total (CA)		162.0	100	105.0	100
ORIGEN[b] (O)		584		584	
CA/O[c]		0.28		0.18	

[a]Not analyzed.
[b]ORIGEN (1066-day cooled), October 1, 1977.
[c]Chemical analysis/ORIGEN ratio.

charcoal sorber. In the second experiment, about 19% of the total iodine in FTE-16-1-4 remained with the burned fuel, 52% was trapped by the Hastelloy X filter, and about 29% was found on the first activated-charcoal sorber. These variations may be attributed partly to small variations in the burner operation and partly to the difficulty of reproducing the lengthy separation and irradiation procedure in the analysis for ^{129}I in highly radioactive materials.

Both the Hastelloy X filter and the first charcoal trap were effective in capturing the released iodine. Less than 0.5% of the total ^{129}I found was in the activated-charcoal and silver-exchanged zeolite sorbers downstream of the first activated-charcoal sorber. Less than 0.1% of the total ^{129}I was evolved during the grinding operation and <0.1%, if any, remained with the undissolved SiC hulls.

3.3 Fission Products Other than Iodine-129

The samples submitted for analysis were first gamma scanned to identify the nuclides present in measurable quantities. In addition to ^{129}I, the most abundant fission products measured were ^{106}Ru, ^{125}Sb, ^{134}Cs, ^{137}Cs, ^{144}Ce, and ^{154}Eu (Tables 5 and 6). Ruthenium-106 was concentrated in the SiC hulls in both runs (91.9 and 87.4% respectively). Approximately 89% of the ^{125}Sb was dissolved from the ash in experiment FTE-16-1-3, but over half of the ^{125}Sb remained undissolved in the hulls in FTE-16-1-4.

The larger part of the remaining fission products was dissolved during the reflux of the burner ash with Thorex reagent. About 90% of the ^{144}Ce and 90-95% of the ^{154}Eu were dissolved by the first leach solution in each experiment. The remaining cerium and europium in FTE-16-1-3 were dissolved by the second leach solution. However, the ^{144}Ce and ^{154}Eu remaining in FTE-16-1-4 were apparently unaffected by a second leach and stayed with the undissolved SiC hulls. Similar large fractions of the ^{134}Cs and ^{137}Cs were found in the hulls. A possible explanation is that the residue of FTE-16-1-4 contained more unbroken particles than the heavy metal analysis indicated (Sect. 3.1). The residue weights also indicated that unbroken particles were present; however, the ^{85}Kr results did not substantiate the possibility. Cesium was the only nuclide other than

Table 5. Composite of results for experiment FTE-16-1-3

FTE-16-1-3	^{129}I µg	^{129}I %	Uranium mg	Uranium %	Thorium mg	Thorium %	^{106}Ru dps[a]	^{106}Ru %	^{125}Sb dps	^{125}Sb %	^{134}Cs dps	^{134}Cs %	^{137}Cs dps	^{137}Cs %	^{144}Ce dps	^{144}Ce %	^{154}Eu dps	^{154}Eu %	^{14}C dps	^{14}C %	^{85}Kr dps	^{85}Kr %	Gross gamma cps[b]	Gross gamma %
Grind, off-gas																			2.53E03	0.2	7.00E08	32.1		
Activated-charcoal adsorber	<0.1	0.1					1.45E03[c]	<0.1	d	d	4.74E03	<0.1	1.12E04	<0.1	1.04E04	<0.1	2.55E02	0.1						
Absolute filter							1.83E02	<0.1	d	d	3.46E02	<0.1	8.77E02	<0.1	8.53E02	<0.1	d	d						
Burn, off-gas																			1.06E06	99.5	1.47E09	67.5		
Activated-charcoal adsorber 1	130	80.3					1.52E04	<0.1	d	d	9.45E03	<0.1	2.31E04	<0.1	2.94E03	<0.1	e	e						
Activated-charcoal adsorber 2	0.5	0.3					1.96E04	<0.1	d	d	2.27E05	<0.1	5.14E05	<0.1	2.86E04	<0.1	e	e						
Silver zeolite adsorber	0.3	0.2					5.99E02	<0.1	d	d	8.47E02	<0.1	2.28E03	<0.1	3.51E02	<0.1	e	e						
Absolute filter							≤2.30E01	<0.1	d	d	8.60E01	<0.1	3.40E02	<0.1	5.90E01	<0.1	e	e						
Hastelloy X filter	23.0	14.2	<0.1	<0.1	1.8	<0.1	d	d	d	d	5.37E08	10.0	1.21E09	9.7	d	d	d	d					4.23E08	8.7
Off-gas scrubber	<0.1	<0.1					2.20E02	<0.1	d	d	1.68E02	<0.1	6.90E02	<0.1	6.20E01	<0.1	d	d					1.88E02	<0.1
Leach, first	1.8	1.1	290.2	87.7	2209	89.2	≤7.00E07	6.9	≤2.45E08	65.8	3.71E09	69.5	8.82E09	70.3	2.12E10	89.5	3.42E08	91.0					3.42E09	70.4
Off-gas scrubber	3.5	2.2	<0.1	<0.1	<0.2	<0.1	≤5.00E04	<0.1	≤5.00E03	<0.1	1.60E05	<0.1	3.70E05	<0.1	≤3.75E05	<0.1	≤1.25E03	<0.1					1.26E05	<0.1
Off-gas																			2.61E03	0.2	≤8.08E06	≤0.4		
Leach, second	1.5	0.9	<0.1[f]	<0.1[f]	266	10.7	≤1.20E07	1.2	8.72E07	23.4	6.92E08	13.0	1.60E09	12.7	2.44E09	10.3	3.40E07	9.0					6.24E08	12.9
Off-gas scrubber	1.4	0.8	37.3[f]	11.3[f]	0.2	<0.1	≤2.50E04	<0.1	≤1.25E03	<0.1	3.13E03	<0.1	1.01E04	<0.1	≤7.50E03	0.1	≤2.50E02	<0.1					4.25E03	<0.1
Off-gas																			≤4.81E02	<0.1	≤1.50E06	<0.1		
Insolubles	<0.1	<0.1	3.4	1.0	0.3	<0.1	9.36E08	91.9	4.03E07	10.8	4.02E08	7.5	9.12E08	7.3	4.57E07	0.2	d	d					3.90E08	8.0
Total (CA)	162	100	330.9	100	2477	100	1.02E09	100	3.73E08	100	5.34E09	100	1.25E10	100	2.37E10	100	3.76E08	100	1.07E06	100	2.18E09	100	4.86E09	100
ORIGEN[g] (O)	584		415		2940		2.16E09		3.70E08		5.72E09		1.50E10		1.75E10		4.62E08		h		1.72E09			
CA/O[i]	0.28		0.8		0.84		0.47		1.01		0.93		0.83		1.35		0.81				1.27			

[a] dps = disintegration per second.
[b] cps = counts per second.
[c] A shorthand notation for showing exponents (also used in subsequent tables); this example is read as 1.45×10^3.
[d] Undetected.
[e] Limits of detection.
[f] Suspect results; scrubber and leach results are probably reversed.
[g] ORIGEN (1066-day cooled) and radioactivity measurements were corrected to October 1, 1977.
[h] Insufficient historical data for an ORIGEN prediction.
[i] Chemical analysis/ORIGEN ratio.

Table 6. Composite of results for experiment FTE-16-1-4

FTE-16-1-4	^{129}I		Uranium		Thorium		^{106}Ru		^{125}Sb		^{134}Cs		^{137}Cs		^{144}Ce		^{154}Eu		^{14}C		^{85}Kr		Gross gamma	
	μg	%	mg	%	mg	%	dps[a]	%	dps	%	dps	%	dps	%	dps	%	dps	%	dps	%	dps	%	cps[b]	%
Grind, off-gas																			3.84E03	0.4	6.12E08	28.8		
Activated-charcoal adsorber	<0.1	<0.1					1.00E04	<0.1	c	c	2.57E06	<0.1	5.89E06	<0.1	c	c	c	c						
Absolute filter							c	c	c	c	2.60E02	<0.1	7.70E02	<0.1	c	c	c	c						
Burn, off-gas																			1.03E06	99.1	1.51E09	71.1		
Activated-charcoal adsorber 1	30.0	28.7					c	c	c	c	6.42E04	<0.1	1.55E05	<0.1	c	c	c	c						
Activated-charcoal adsorber 2	<0.1	<0.1					c	c	c	c	1.96E04	<0.1	5.07E04	<0.1	6.85E05	<0.1	≤2.00E03	<0.1						
Silver zeolite adsorber	<0.1	<0.1					c	c	c	c	1.69E03	<0.1	4.12E03	<0.1	3.50E02	<0.1	c	c						
Absolute filter							c	c	c	c	1.10E01	<0.1	3.50E01	<0.1	c	c	c	c						
Hastelloy X filter	54.5	52.0	0.3	0.1	5.6	0.3	≤5.00E06	<0.2	<2.50E06	≤0.7	1.04E08	1.6	2.44E08	1.7	9.88E06	0.4	2.50E05	<0.1					8.55E07	1.7
Off-gas scrubber	<0.1	<0.1					1.35E04	<0.1	<5.00E02	<0.1	2.34E03	<0.1	6.06E03	<0.1	1.20E03	<0.1	<5.00E01	<0.1					2.72E03	<0.1
Leach, first	18.5	17.7	253.2	93.4	2112	96.6	<2.91E08	11.7	≤4.85E07	≤14.1	4.13E09	65.0	9.37E09	64.5	2.18E09	89.7	3.36E08	95.4					3.65E09	73.4
Off-gas scrubber	0.7	0.7	<0.1	<0.1	<0.1	<0.1	5.23E05	<0.1	≤3.51E03	<0.1	3.64E03	<0.1	9.01E03	<0.1	≤4.29E03	<0.1	≤2.34E02	<0.1					3.28E04	<0.1
Off-gas																			≤1.64E03	≤0.2	≤2.32E06	≤0.1		
Leach, second	0.8	0.8	15.8	5.8	38.9	1.8	1.76E07	0.7	1.04E08	30.3	7.99E03	<0.1	1.83E04	<0.1	2.84E04	<0.1	6.14E02	<0.1					2.75E08	5.6
Off-gas scrubber	0.1	0.1	<0.1	<0.1	<0.1	<0.1	≤7.00E04	0.1	≤7.00E03	<0.1	2.93E05	0.1	7.18E05	<0.1	5.57E05	<0.1	8.40E03	<0.1					1.06E05	0.1
Off-gas																			3.40E03	0.3	≤7.30E05	<0.1		
Insolubles	<0.1	<0.1	1.8	0.7	29.4	1.3	2.17E09	87.4	1.88E08	54.9	2.12E09	33.4	4.91E09	33.8	2.42E08	9.9	1.61E07	4.6					9.61E08	19.3
Total (CA)	105	100	271.1	100	2186	100	2.48E09	100	3.43E08	100	6.35E09	100	1.45E10	100	2.43E09	100	3.52E08	100	1.04E06	100	2.13E09	100	4.97E09	100
ORIGEN[d] (O)	584		415		2940		2.16E09		3.70E08		5.72E09		1.50E10		1.75E10		4.62E08		e		1.72E09			
CA/O[f]	0.18		0.65		0.74		1.15		0.93		1.11		0.97		0.14		0.76				1.24			

[a] dps = disintegration per second.
[b] cps = counts per second.
[c] Undetected.
[d] ORIGEN (1066-day cooled) and radioactivity measurements were corrected to October 1, 1977.
[e] Insufficient historical data for an ORIGEN prediction.
[f] Chemical analysis/ORIGEN ratio.

iodine found in appreciable quantity (∿20%) on the Hastelloy X filter during experiment FTE-16-1-3.

3.4 Carbon-14 and Krypton-85

Although the two experiments were initiated to study the behavior of ^{129}I, they provided an opportunity to determine the release of ^{14}C and ^{85}Kr during a modified grind-burn-leach process using HTGR fuel and small burner equipment. To avoid upsetting the movement of any iodine in the off-gas stream, the only samples of gas removed were taken from the gas collection bag at the end of each operation. Samples of approximately 275 cm^3 for ^{14}C and 6.8 cm^3 for ^{85}Kr were removed in calibrated gas bottles. Triplicate samples were taken from each gas collection for increased reliability.

The distributions of ^{14}C in experiments FTE-16-1-3 and FTE-16-1-4 are shown in Tables 7 and 8. Less than 1% of the ^{14}C was released from the grind and leach operations. Almost all of the total released ^{14}C resulted from the burn operation (99.5% in experiment FTE-16-1-3 to 99.1% in FTE-16-1-4). Agreement between averaged values for total concentration of ^{14}C was within 3% (1.07E06 dps for FTE-16-1-3 and 1.04E06 dps for FTE-16-104). This quantity of ^{14}C corresponds to a nitrogen content in the starting fuel of about 1000 ppm (based on uranium plus thorium metal), as calculated using the ORIGEN code. The distribution of ^{85}Kr in experiments FTE-16-1-3 and FTE-16-1-4 is shown in Tables 9 and 10. Nearly one-third of the total ^{85}Kr found in each experiment was released during the grind operation. Less than 0.5% of the ^{85}Kr was released during leaching. The largest quantity of ^{85}Kr was released during the burn operation in each experiment (67.4% in FTE-16-1-3 and 71.1% in FTE-16-1-4). The total quantity of ^{85}Kr determined by radiochemical analysis was about 25% greater than predicted by ORIGEN (27% more in FTE-16-1-3 and 24% more in FTE-16-1-4).

4. CONCLUSIONS AND FUTURE WORK

The history for the fuel rods used in the two experiments was the same, yet about one-third less iodine was identified in experiment FTE-16-1-4 than in FTE-16-1-3. The iodine found in each experiment was

Table 7. Distribution of ^{14}C in the off-gas system (FTE-16-1-3 fuel)[a]

Operation	Off-gas volume at STP (liter)	Sample code	Carbon-14		
			Σ dps[b,c]	Average (dps)	Percent of total
Grind	6.6	GC-1	5.72E03		
		GC-2	1.11E03	2.53E03	0.2
		GC-3	7.54E02		
Burn	27.5	BC-1	1.07E06		
		BC-2	1.05E06		
		BC-3	1.21E06	1.06E06	99.5
		BC-4	9.98E05		
		BC-5	1.01E06		
		BC-6	9.85E05		
First leach	35.2	LC-1	3.21E03		
		LC-2	3.59E03	2.61E03	0.2
		LC-3	1.03E03		
Second leach	32.9	LC-4	≤4.81E02		
		LC-5	≤3.60E02	≤4.81E02	<0.1
		LC-6	≤6.01E02		
	102.2			1.07E06	100

[a]TRISO-coated $(Th,U)C_2$--TRISO-coated ThC_2.
[b]All radioactivity measurements were corrected to October 1, 1977.
[c]dps = disintegration per second.

Table 8. Distribution of ^{14}C in the off-gas system (FTE-16-1-4 fuel)[a]

Operation	Off-gas volume at STP (liter)	Sample code	Carbon-14		
			Σ dps[b,c]	Average (dps)	Percent of total
Grind	10.5	GC-1	3.60E03		
		GC-2	3.33E03	3.84E03	0.4
		GC-3	4.60E03		
Burn	27.1	BC-1	1.02E06		
		BC-2	1.14E06		
		BC-3	1.18E06	1.03E06	99.1
		BC-4	9.39E05		
		BC-5	9.14E05		
		BC-6	9.46E05		
First leach	29.6	LC-1	2.82E03		
		LC-2	≤7.59E02	≤1.64E03	≤0.2
		LC-3	1.35E03		
Second leach	34.6	LC-4	≤4.81E02		
		LC-5	≤8.75E02	3.40E03	0.3
		LC-6	8.85E03		
	101.8			1.04E06	100

[a]TRISO-coated $(Th,U)C_2$--TRISO-coated ThC_2.
[b]All radioactivity measurements were corrected to October 1, 1977.
[c]dps = disintegration per second.

Table 9. Distribution of ^{85}Kr in the off-gas system (FTE-16-1-3 fuel)[a]

Operation	Off-gas volume at STP (liter)	Sample code	Krypton-85		
			Σ dps[b,c]	Average (dps)	Percent of total
Grind	6.6	GK-1	7.00E08		
		GK-2	6.95E08	7.00E08	32.1
		GK-3	7.06E08		
Burn	27.5	BK-1	1.40E09		
		BK-2	1.43E09		
		BK-3	1.42E09	1.47E09	67.4
		BK-4	1.55E09		
		BK-5	1.50E09		
		BK-6	1.53E09		
First leach	35.2	LK-1	≤8.12E05		
		LK-2	≤1.62E06	≤8.80E06	≤0.4
		LK-3	≤2.18E07		
Second leach	32.9	LK-4	≤1.52E06		
		LK-5	≤2.51E06	≤1.50E06	<0.1
		LK-6	4.84E05		
	102.2			2.18E09	100

[a]TRISO-coated $(Th,U)C_2$--TRISO-coated ThC_2.
[b]All radioactivity measurements were corrected to October 1, 1977.
[c]dps = disintegration per second.

Table 10. Distribution of ^{85}Kr in the off-gas system (FTE-16-1-4 fuel)[a]

Operation	Off-gas volume at STP (liter)	Sample code	Krypton-85		
			Σ dps[b,c]	Average (dps)	Percent of total
Grind	10.5	GK-1	6.10E08		
		GK-2	6.04E08	6.12E08	28.8
		GK-3	6.24E08		
Burn	27.1	BK-1	1.38E09		
		BK-2	1.38E09		
		BK-3	1.39E09	1.51E09	71.1
		BK-4	1.65E09		
		BK-5	1.62E09		
		BK-6	1.64E09		
First leach	29.6	LK-1	2.59E06		
		LK-2	≤1.74E06	≤2.32E06	≤0.1
		LK-3	2.63E06		
Second leach	34.6	LK-4	≤5.09E05		
		LK-5	≤1.02E06	≤7.30E05	<0.1
		LK-6	≤6.62E05		
	101.8			2.13E09	100

[a]TRISO-coated $(Th,U)C_2$--TRISO-coated ThC_2.
[b]All radioactivity measurements were corrected to October 1, 1977
[c]dps = disintegration per second.

much less than predicted by ORIGEN. Approximately 14.2% of the total ^{129}I found in experiment FTE-16-1-3 and 52.0% of the total ^{129}I found in experiment FTE-16-1-4 were on the Hastelloy X filter. The small percentages of uranium and thorium found on the Hastelloy X filters indicate that the iodine found resulted from deposition and not from fuel fines entrained in the off-gas.

Future experiments must incorporate methods to locate and measure the iodine deposits that are not now being sampled (e.g., a system to determine the iodine and fission-product distribution on the inner walls of the burner must be devised). In addition, the time between sampling and analysis must be shortened to avoid sample deterioration and adulteration, as well as the consequent loss of the sample as being representative of the parent source.

A new method developed since this experiment by L. C. Bate et al.[6] for determining ^{129}I should be tested. The method utilizes an anion exchange resin to concentrate the ^{129}I for direct neutron activation analysis in the HFIR facility. The authors reported a capability of detecting approximately 2×10^{-10} g of ^{129}I and an agreement between duplicate samples of better than 5% for microgram quantities. The method shows great promise to meet the need for improved sensitivity, increased precision and accuracy, and more rapid measurement.

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