Experimental Measurements Of Volatility Of Fission-Product Iodine Released From Irradiated CANDU Fuel In High-Temperature Steam Environment: Tests I1 And I2 In The HCE5 Experiment

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ABSTRACT

Sheathed samples of irradiated UO_2 were heated in flowing steam to 1930 K and 2010 K, in order to investigate the volatility of iodine released from CANDU® fuel. Iodine volatility was measured in these two tests by γ-scanning of short-lived iodine isotopes on particulate and charcoal filters within a few hours after exposure to air. Final fractional releases of I, Cs and Mo isotopes were about 70% in both tests. No significant quantities of volatile iodine were found (probably less than 0.1% of the iodine that reached the particulate filters), which supports the approach used to assess iodine volatility in containment for CANDU 6 licensing.

INTRODUCTION

The fraction of iodine released from fuel in volatile forms (*e.g.*, I_2, HI) is of interest in reactor safety analysis, because volatile forms of iodine can be released from containment more easily than solid and liquid aerosol forms. In the ORNL (Oak Ridge National Laboratory) VI (Vertical Induction-heated) test series [1,2], the reported fraction of iodine released in volatile forms from light-water reactor fuel was typically 1%. In two tests (VI-5 and VI-6), the reported volatile iodine fraction was about 3%. For both of these tests, there was a waiting period of five to six months in which the charcoal cartridges remained sealed up together with the particulate filters before iodine volatility was measured. The ORNL researchers acknowledged that air ingress into the VI apparatus in conjunction with the radiation fields associated with the apparatus might have decomposed some of the particulate iodine species and allowed the iodine to migrate to the charcoal filters, artificially increasing the measured volatile iodine fractions [2]. Typical waiting periods before analysis in other tests in the ORNL VI and HI (Horizontal Induction-heated) series were a few weeks, so some iodine decomposition and migration may have occurred in these tests as well.

Tests I1 and I2 in Hot-Cell Experiment #5 (HCE5) conducted at Chalk River Laboratories were intended to measure iodine volatility while avoiding long delays after exposure to air. Short-lived iodine isotopes were generated in the fuel by trace-reirradiation, then released from the fuel at high temperature in a steam environment in a zirconia furnace tube and trapped on particulate and charcoal filters. The filter modules were γ-scanned to measure ^{131}I on the particulate and charcoal filters within four hours after disassembly of the apparatus and exposure of the deposited fission products to air. Two tests were planned with different peak sample temperatures, but with the same fuel environment (steam) and identical iodine volatility filter apparatus, to ensure successful experimental measurements of iodine volatility. The trace-reirradiation techniques also allowed measurement of releases of Mo, which is expected to have a significant effect on the volatility of iodine [3,4].

EXPERIMENTAL APPARATUS

The 15 kW tube furnace (manufactured by Central Iron and Steel Research Institute, Beijing) used to perform these tests had zirconia main heating elements, molybdenum disilicide preheating elements and a single-piece vertical yttria-stabilized zirconia guide tube (Figure 1). Two two-colour optical pyrometers were used to measure the temperatures of opposite sides of the outside of the guide tube. The yttria-stabilized zirconia furnace tube assemblies for the tests were inserted through the top of the furnace, and were suspended from the top of the furnace casing. The annulus between the guide tube and the furnace tube was purged with Ar/2%H_2 flowing at 2 L/min [(1)] for sample temperatures above 1520 K, to minimize oxygen permeation through the

[1] Flow rates of Ar and Ar/2% H_2 are given in units of L/min at a temperature of 273 K and a pressure of 101.3 kPa.

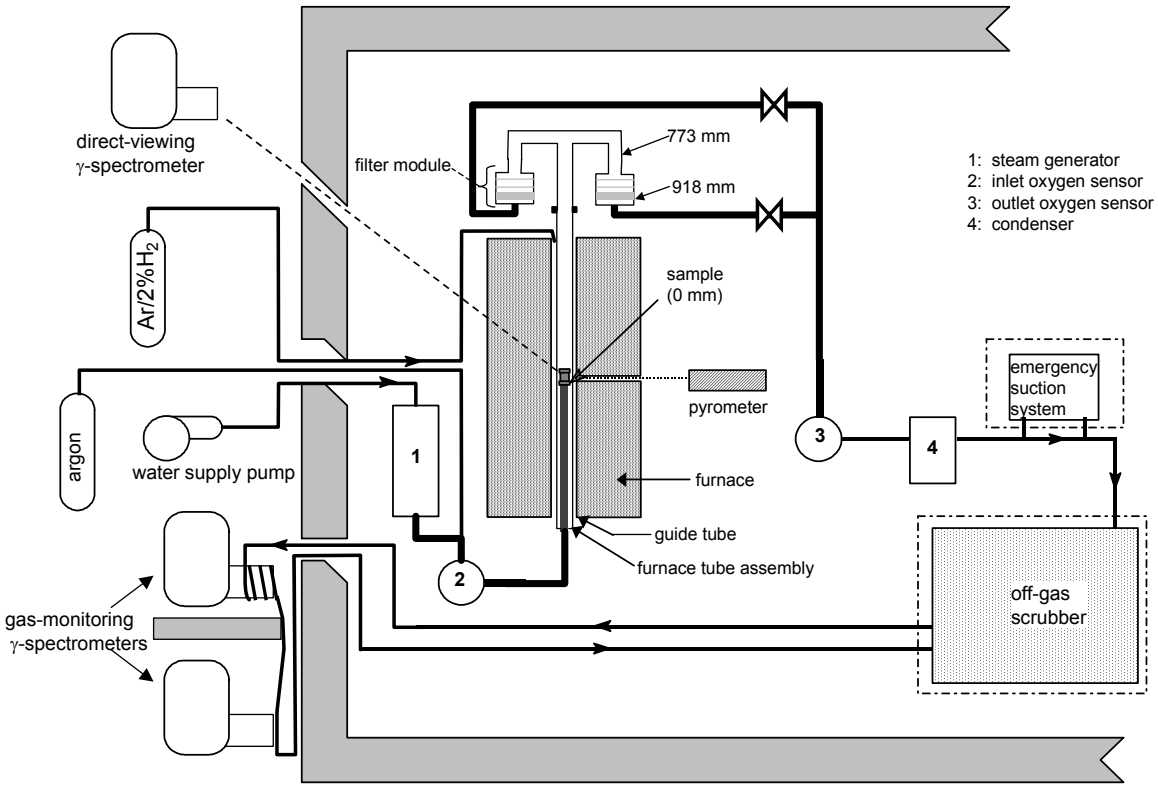

FIGURE 1: SCHEMATIC OF THE HCE5 EXPERIMENTAL APPARATUS.

furnace tube. The carrier gas inlet at the bottom of the furnace tube assembly employed an O-ring fitting that also supported a yttria sample boat on a yttria-stabilized zirconia support post. A two-leg manifold and two filter modules in a parallel configuration were supported on a 25 mm Swagelok fitting at the top of the furnace tube. The internal step in the Swagelok fitting was removed so that the ceramic and glass tubes could be butted together to minimize gas contact with metals and the Teflon ferrule. The outlet of each filter module was connected to stainless steel tubing leading to a valve that could be opened to allow the gas to pass through the filter. Only one filter module valve was open at a time, except for a period of about 60 s while switching between filter modules. The manifold and filter assemblies were trace-heated to between 390 K and 450 K to prevent steam condensation; the iodine filter modules were heated to 390 K.

The manifold, filter body, filter spacers and particulate filters were made of silica glass, based on its minimal sorption of iodine compounds [5]. A schematic of one filter module is given in Figure 2. The particulate filter section consisted of two layers of binder-free silica-glass-fibre filter material (Whatman QM-A, nominal filtration efficiency of 99.999% for particles 0.6 μm in diameter), separated by a silica glass spacer 20 mm long. The particulate filter section was followed by another silica glass spacer 20 mm long, then a charcoal filter section (a 15 mm bed of TEDA-impregnated charcoal) supported using a stainless steel type 304 screen and a dual layer of particulate filter material. The filter spacers were incorporated to assist the γ-scanner in resolving the regions in which

fission products were located. All internal components were held in place by a bottom lid, Viton O-ring and silica glass spacer downstream of the charcoal filter.

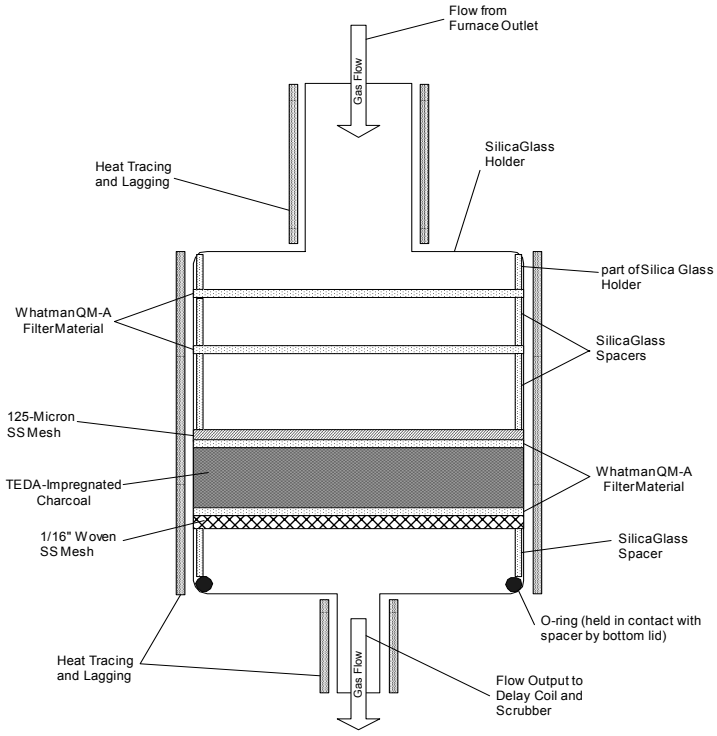

FIGURE 2: SCHEMATIC OF THE HCE5 IODINE FILTER.

The inlet flow rate of Ar was measured using a mass flow meter. Distilled deionized water was fed at a controlled rate of 60 g/h from outside the hot cell to a small steam generator on the furnace chassis. The steam and argon were mixed and fed through heat-traced lines into a zirconia oxygen sensor [6] upstream of the furnace tube; a similar oxygen sensor monitored the oxygen partial pressure downstream of the filter assemblies. The oxygen sensors were operated at temperatures in the range 930 K to 1040 K, and were used to determine sample oxidation rates. Downstream of the second oxygen sensor, the steam was condensed and the non-condensable gas was passed through high-efficiency filters before flowing into the delay coil for γ-spectrometric monitoring.

Three γ-spectrometers were used to measure fission-product release during the tests (Figure 1). A direct-viewing γ-spectrometer was located outside the hot-cell and sighted through a collimated port to view the fuel sample through the wall of the furnace. Two γ-spectrometers monitored the ^{133}Xe, ^{133m}Xe and ^{85}Kr count rates in the carrier gas in two shielded delay coils of different γ-ray counting efficiency connected serially outside of the hot cell [7-11]. The counting time for each γ-spectrum was 300 s. For post-test measurements, a scanning table inside the hot cell [10,11] was placed in front of a hot-cell port with a lead collimator with a slit width of about 10 mm. A fourth γ-spectrometer

was set at different distances from the other end of the collimator outside the hot-cell to adjust spectrometer efficiency. Spectra were taken at intervals of 6 mm for the furnace tube and 5 mm for the filter modules.

FUEL SAMPLES

The samples were segments 20 mm long cut from a Gentilly-2 CANDU power reactor fuel element (57 kW/m peak linear power, 193 MW·h/kg U, discharged 25 months before the experiment). The cut ends were covered with press-fitted Zircaloy end-caps. The fuel samples were trace-reirradiated in the NRU reactor at low power to build up inventories of short-lived fission products (*e.g.*, ^{133}Xe and ^{131}I). After the trace-reirradiation, the short-lived fission products were primarily resident in the UO_2 grains. The first iodine volatility test was conducted seven days after the end of the trace-reirradiation.

CONDITIONS OF TEST I1

The apparatus was purged with argon before heating. The sample was heated to 1620 K at a rate of 5 K/min in Ar flowing at 0.2 L/min, at which point a steam flow (1 g/min) was added to the Ar flow while heating continued to 1930 K. The flow was switched from the first iodine filter module to the second filter module 1480 s after the steam flow was started. The sample was held at 1930 K for 1 h, then the temperature was decreased at a rate of 6 K/s. The steam flow was turned off when the sample temperature decreased below 1660 K, and the sample was cooled to room temperature in flowing Ar.

CONDITIONS OF TEST I2

The apparatus was purged with argon before heating. The sample was heated to 1590 K at a rate of 5 K/min in Ar flowing at 200 mL/min, at which point a steam flow (1 g/min) was added to the Ar flow while heating continued to 2020 K. The flow was switched from the first iodine filter module to the second filter module 1000 s after the steam flow was started. A loss of flow at the outlet was detected when the sample temperature reached 2020 K, so the furnace cooling cycle was started, the steam flow was stopped, and the inert gas flow rates inside and outside the furnace tube were increased. On removing the furnace tube assembly the following day, the furnace tube proved to be broken.

DATA ANALYSIS

Fission-product release percentages (R) were calculated from changes in count rates of selected peaks in the direct-viewing γ-spectra [10,11] with respect to the count rates during a standard period (sample temperature < 1120 K) near the beginning of the test (no significant fission-product releases are expected from sheathed samples of UO_2 at this temperature in argon environment [8-10]):

$$R = \left[1 - \frac{I_t - B_t}{kc(I_s - B_s)} \right] \times 100\% \qquad \textbf{(1)}$$

where I_s is the initial count rate from the sample and background during the standard period,
I_t is the count rate from the sample and background at time t during the test,
B_s is the background count rate during the standard period,
B_t is the background count rate during the test (assumed to be constant),
c is the decay correction factor, and
k is the normalization factor obtained from the 487 keV or 1596 keV count rate of ^{140}La or the 757 keV count rate of ^{95}Zr.

Normalization accounts for changes in sample geometry and attenuation by using the count rate behaviour of isotopes that are known not to release significantly under the test conditions [8-11]. Neither ^{140}La nor ^{95}Zr was significantly released in test I1 or I2 (based on post-test scanning of the furnace tube, and on experience from previous tests conducted under similar conditions [10]). The k values were in the range 0.8 to 1.3 in test I1 and 1.0 to 1.55 in test I2.

The delay-coil γ-spectrometry data on Kr and Xe isotope releases will not be discussed in this paper, except to note that the timing of noble-gas releases was very similar to the timing of Cs and I release, as found in previous hot-cell experiments under these conditions [8-11].

The γ-scanning count rates were converted to linear activities (Bq/cm) of the observed isotopes using Aptec-NRC Version 7.00.01 γ-spectrometry software (from Aptec Instruments Ltd.) and the known efficiency function of the spectrometer (determined from out-cell calibrations using the same collimator and spectrometer to observe a source of known linear activity). The linear activities were decay-corrected to the date of the test, then multiplied by the scan interval between γ-spectra (6 mm or 5 mm) and summed to yield the total activities observed in each region of the apparatus.

RESULTS

Rapid release of Cs, I, Mo and noble gases occurred about 1600 s after the introduction of steam in test I1 (Figure 3). The Cs and I releases observed in both tests started at temperatures from 1740 to 1790 K and began to level off near 70% (Figures 3 and 4). The final releases correlated well with the final Cs releases in steam environment observed in the HCE2 [8,9] tests on LWR fuel (~77%) and the HCE3 tests on sheathed samples of CANDU fuel [10]. The releases of Cs reported for HCE4 tests conducted in steam atmosphere at 1920 K were somewhat higher (~85%) [11], but the HCE4 tests were held at the peak temperature in steam environment for about 6300 s, instead of 3600 s as in test I1.

The differences between ^{134}Cs final fractional releases measured using the 604 keV and 796 keV count rates in the two tests (Table 1) were slightly larger than the combined 2σ uncertainties. Similarly, differences between the final fractional releases of ^{131}I and ^{132}I in test I1 were larger than the combined 2σ uncertainty. The results may have disagreed because the uncertainty due to normalization of the count rates using a line at a different energy was not accounted for; the uncertainties in Table 1 may therefore have been underestimated.

Releases of ^{132}Te in test I1 began at about the same time as the rapid release of Cs and I, but the poor counting statistics and interfering peaks from other fission products obscured more detailed information. The ^{132}Te counting statistics for test I2 were not good enough to show any release kinetics information.

The ^{99}Mo release percentage in test I1 was 84% (Table 1). The ^{99}Mo counting statistics for test I2 were not good enough to show release kinetics, but the overall fractional release of 76% was similar to the overall cesium and iodine fractional releases.

The ^{140}Ba release in test I1 was about 5%, and the ^{140}Ba release in test I2 was not statistically significant. The isotopes ^{95}Zr, ^{95}Nb, ^{103}Ru, ^{106}Rh, ^{140}La, ^{156}Eu, ^{144}Pr, ^{148}Pm and ^{239}Np showed no statistically significant releases in these tests (Table 1).

Post-test γ-scanning of the furnace tubes and filters, followed by leaching and inductively coupled plasma mass spectrometry analysis of the leachate showed deposited Rb, Tc, Ag, Sn, Sb, Ba and U, in addition to the Mo, Te, I, noble gas and Cs releases measured by direct-viewing and delay-coil γ-spectrometry. The Sn originated

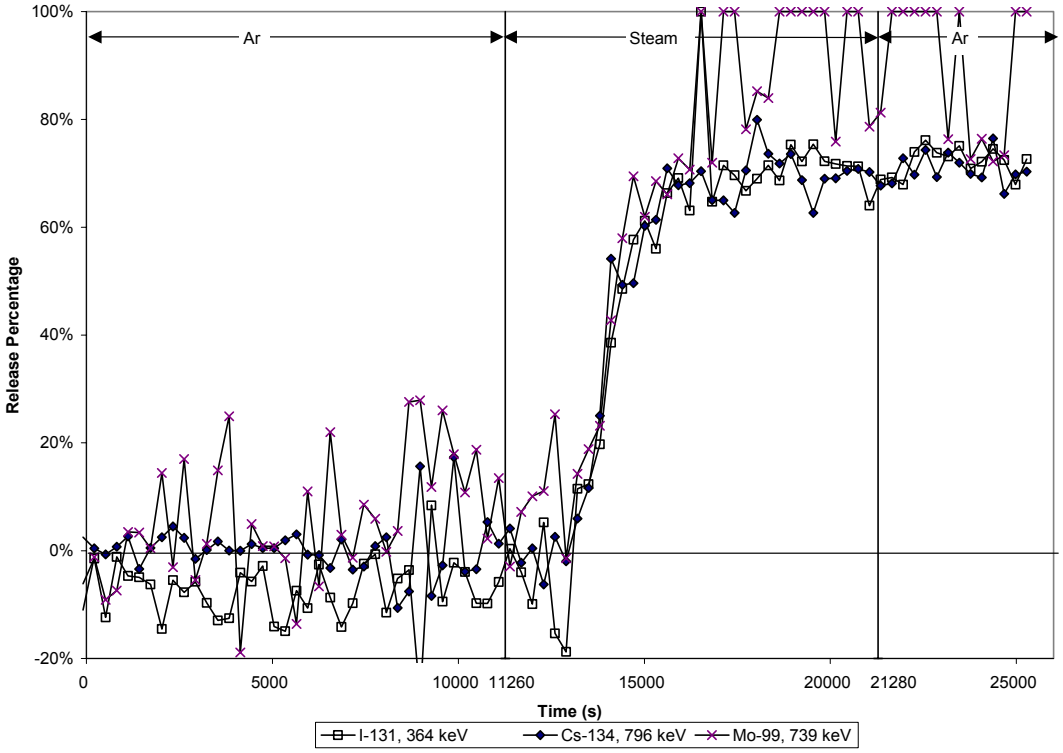

FIGURE 3: RELEASE KINETICS OF ^{131}I, ^{134}Cs AND ^{99}Mo FOR HCE5 TEST I1.

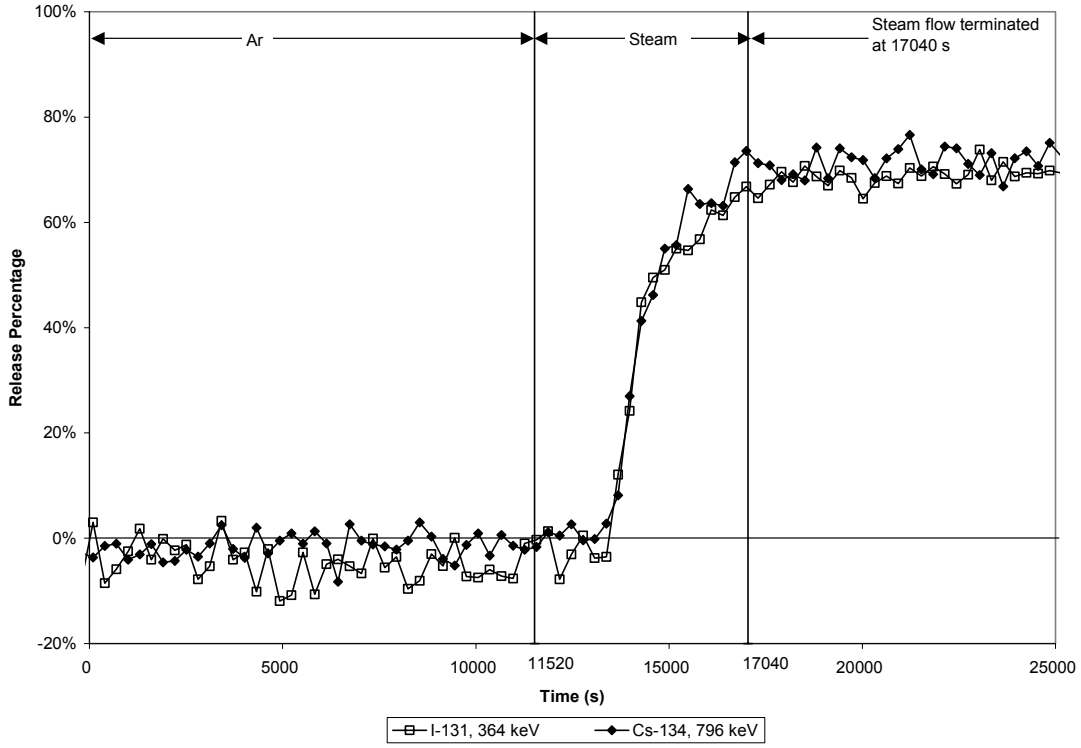

FIGURE 4: RELEASE KINETICS OF ^{131}I AND ^{134}Cs FOR HCE5 TEST I2.

from the oxidized Zircaloy sheath and end-caps [1,10] and the U was from volatilization of the UO_2 fuel material [12].

The filter modules (described under Experimental Apparatus) were γ-scanned in order to resolve the locations of fission products. The combined positional uncertainty[2] of deposition peaks and the location of the filter on the scanning table was 10 mm. One filter module was used early in each test in order to check indications from the ORNL VI

TABLE 1: PERCENTAGE RELEASES OF FISSION PRODUCTS IN HCE5 TESTS I1 AND I2

Isotope	Peak Energy (keV)	Test I1		Test I2	
		Normalized Release (%)	2σ Uncertainty (%)	Normalized Release (%)	2σ Uncertainty (%)
^{95}Nb	765.82	0.7 [3]	1.9	≤ 0.7 [3, 4]	2.0

[2] Uncertainties in this paper are reported at the 2σ or 95% confidence level.
[3] Not observed by γ-scanning
[4] Non-physical negative release percentages (R) that were not statistically significant were denoted as ≤(R+2σ)

^{99}Mo	739.47	84	4	76	12
^{103}Ru	497.08	2.2 [3]	1.7	0.039 [5]	0.008
^{106}Rh	621.80	2.1 [3]	2.1	0 [3, 6]	2.8
^{110m}Ag		12.8 [5]	2.7	17 [5]	4
^{125}Sb		35 [5]	8	40 [5]	9
^{132}Te [7]	228.16	32 [7]	4	10 [7]	19
^{131}I	364.48	74.9	1.5	71	8
^{132}I	772.61	71.9	0.6	74.3	1.0
^{134}Cs	604.66	68.7	1.0	71.5	1.1
	795.76	75.5	0.8	68.8	0.9
^{137}Cs	661.62	69.6	0.5	68.9	0.6
^{140}Ba	537.38	1.7 [5]	0.3	1.6 [5]	0.3
^{140}La	328.75	≤3 [4]	5	≤2 [4]	7
^{156}Eu	2097.68	≤13 [3, 4]	23	3 [3]	28
^{144}Pr	2185.61	0 [3, 8]	2.9	0.3 [3]	3.0
^{148}Pm	1465.10	1 [3]	24	≤34 [3]	34
^{239}Np	277.60	≤4 [3]	4	≤8 [3, 4]	13

[5] Value from γ-scanning
[6] The fractional release for Rh-106 calculated at the end of test I2 was (-9.5 ± 2.8)%. Comparison of ^{106}Rh count rates and their uncertainties determined using different peak search methods in the Aptec-NRC software indicates that the count rate uncertainty for this peak may have been underestimated. The release was reassigned a value of zero.
[7] The γ-peak for ^{132}Te is interfered with by the peak for ^{239}Np. These values should be treated as lower limits for the fractional release of ^{132}Te.
[8] The fractional release for ^{144}Pr calculated at the end of test I1 was (-4.0 ± 2.9)%. Inspection of the ^{144}Pr count rates and spectral regions did not indicate any abnormalities, so the negative result may have been due to statistical fluctuations. The release was reassigned a value of zero.

tests that iodine released early in the test tended to have a higher volatile fraction. The first filter module in test I1 contained 1.7% of the released ^{131}I and 4.4% of the released Cs (Figure 5), and the first filter module in test I2 contained 2.1% of the released ^{131}I and 4.4% of the released Cs (Figure 6). No statistically significant amounts of iodine were observed in the charcoal filter regions of either of these modules. The maximum fraction of iodine that could have remained unobserved in the charcoal filter region was between 0.2% and 0.3% of the amount in each filter module.

A second filter module was used for the balance of each test. In the second module for test I1, (0.59 ± 0.06)% of the ^{131}I that was in the module was observed in the charcoal filter region (Figure 7), but (0.43 ± 0.08)% of the ^{134}Cs, (0.43 ± 0.10)% of the ^{132}Te and (0.68 ± 0.14)% of the ^{99}Mo were also observed in the charcoal filter region, indicating aerosol material bypass of the upstream particulate filters. The scanned fractions in the charcoal region of the filter module corresponded to 19 µg of Cs and 2.6 µg of I (a Cs/I mole ratio of 7). In the second module for test I2, aerosol material bypass of the first aerosol filter was very evident; the fraction of the Cs that was in the charcoal filter region was higher than the corresponding fraction of I (Figure 8).

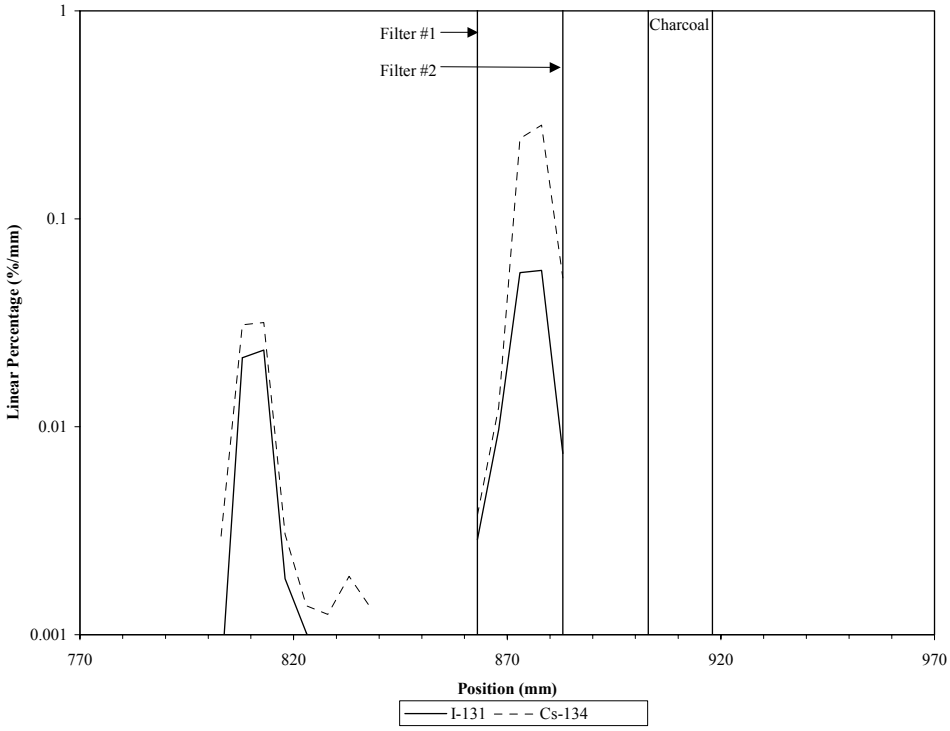

FIGURE 5: LINEAR ACTIVITY PERCENTAGES OF ^{131}I AND ^{134}Cs ON FILTER MODULE USED IN FIRST PART OF TEST I1 (LOGARITHMIC VERTICAL SCALE). POSITIONAL UNCERTAINTY IS ±10 MM.

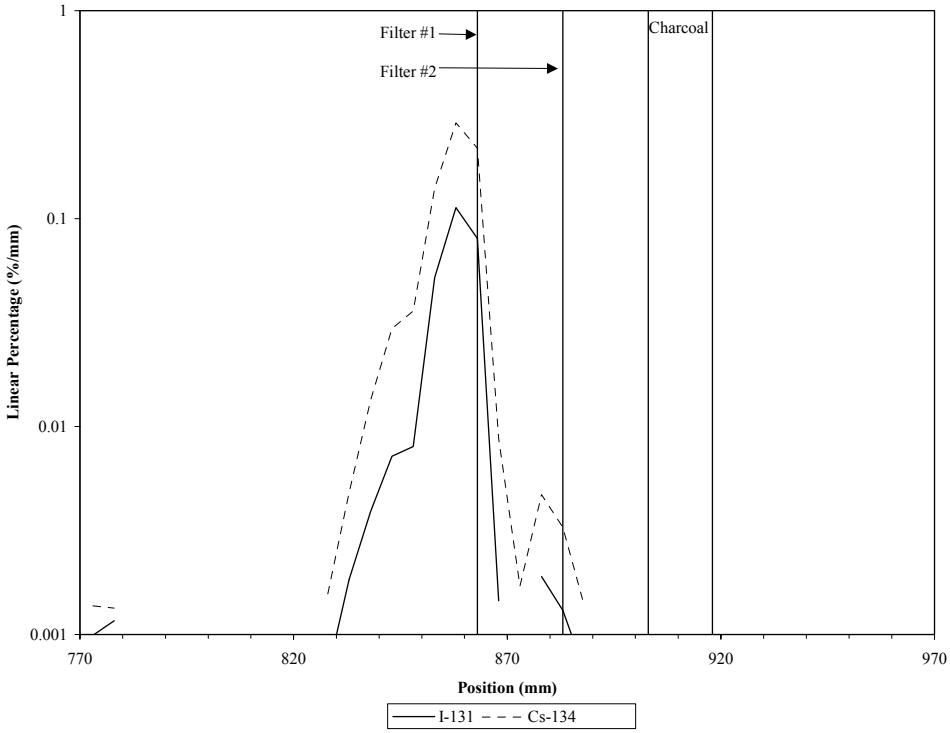

FIGURE 6: LINEAR ACTIVITY PERCENTAGES OF ^{131}I AND ^{134}Cs ON FILTER MODULE USED IN FIRST PART OF TEST I2 (LOGARITHMIC VERTICAL SCALE). POSITIONAL UNCERTAINTY IS ±10 MM.

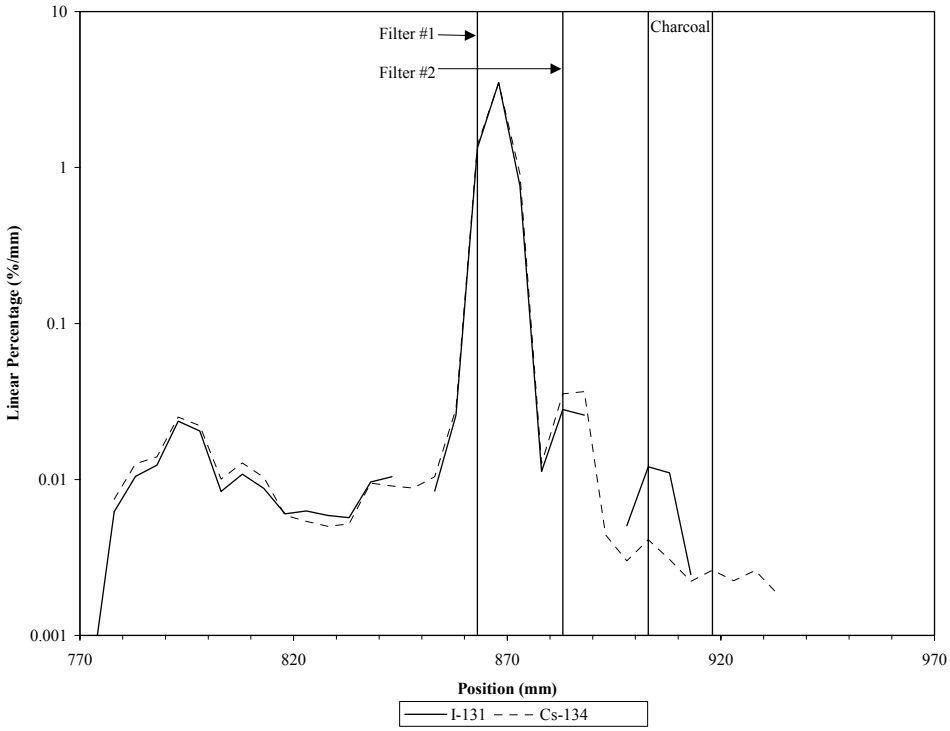

FIGURE 7: LINEAR ACTIVITY PERCENTAGES OF ^{131}I AND ^{134}Cs ON FILTER MODULE USED IN SECOND PART OF TEST I1 (LOGARITHMIC VERTICAL SCALE). POSITIONAL UNCERTAINTY IS ±10 MM.

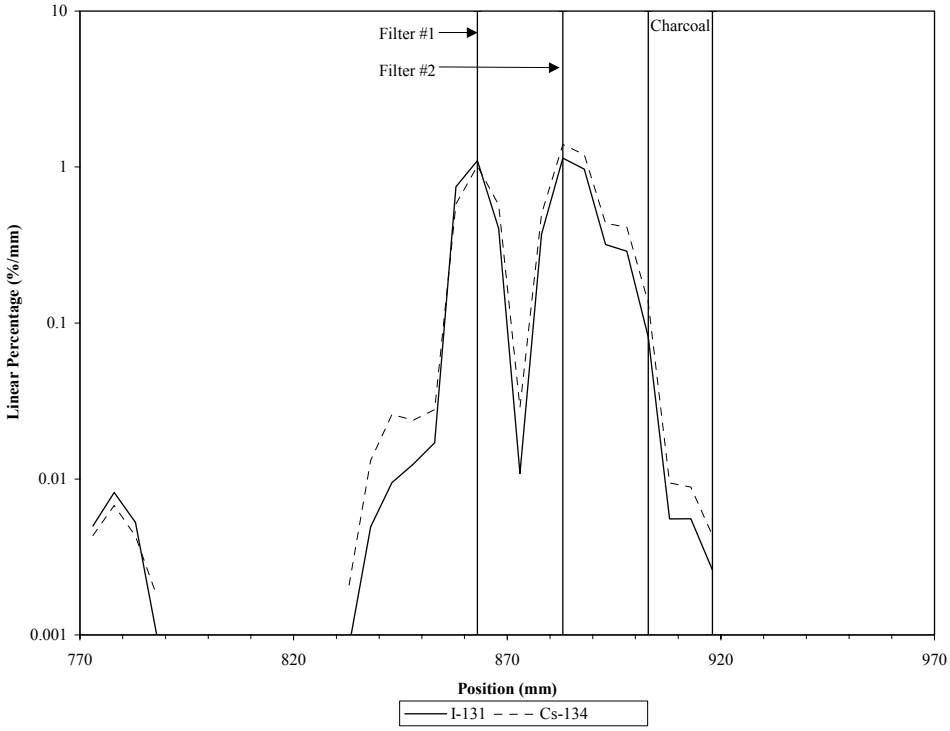

FIGURE 8: LINEAR ACTIVITY PERCENTAGES OF ^{131}I AND ^{134}Cs ON FILTER MODULE USED IN SECOND PART OF TEST I2 (LOGARITHMIC VERTICAL SCALE). POSITIONAL UNCERTAINTY IS ±10 MM.

DISCUSSION

The samples in the I1 and I2 tests exhibited the normal pattern of fission-product release for sheathed samples in steam environment at 1700 K and higher: low releases prior to through-wall oxidation of the sheath, and rapid release during the oxidation of the UO_2 [8-11]. The released fission products and structural materials (as well as the composition of the gas stream in which they were transported) were therefore reasonably typical of a steam-environment accident scenario. Most of the released I, Cs and Mo entered the gaseous environment surrounding the sample within a 1500 s interval after through-wall sheath oxidation.

For the early part of tests I1 and I2, the detection limit of 0.2 to 0.3% of the ^{131}I in the filter modules was the upper limit on the amount of iodine that could have been present in volatile form. In the later parts of the tests, some ^{131}I was observed in the charcoal filter regions, but was probably mostly due to aerosol bypass of the particulate filters. Based on the fractions of Cs and I in the charcoal filter region, the test I1 results indicated a maximum credible quantity of volatile iodine of (0.16 ± 0.10)%, while the test

I2 results indicated no significant amounts of volatile iodine. In both tests, the number of moles of Cs in the charcoal filter region was much greater than the number of moles of I, so that all of the I in the charcoal filter could have been CsI (the usual assumed form of fission-product iodine after release from fuel) rather than volatile iodine compounds. The iodine was predominantly in non-volatile forms, and the fraction of iodine in volatile forms may well have been less than 0.1% in all cases. This is significantly lower than most of the observations in the ORNL VI tests, but is consistent with some earlier measurements made in ORNL tests HI-3, HI-4 and HI-5 [13] [(9)].

At all locations in the furnace tubes and the filters used for the high-release phases of tests I1 and I2, the Cs/Mo mole ratio was low enough that all of the Cs could have been in the form of Cs_2MoO_4. If all the Cs were in the form of Cs_2MoO_4, the iodine volatility would have been expected to be very high because CsI (the most stable iodine compound in the absence of Mo) would have been decomposed and volatile iodine species such as HI and I_2 would have been formed [3,4]. In fact, the iodine volatility remained low. The contaminant levels indicated by analysis of the leachates were significantly lower than the levels of I, so that unexpected iodine compounds are not likely to have affected the results. Several possible reasons may account for this disagreement with thermodynamic calculations. For instance:

- despite its thermodynamic stability, the formation of Cs_2MoO_4 may be slow on the timescale of fission-product transport in this experiment, or
- other Mo compounds more stable than Cs_2MoO_4 may have been present in the experiment but not accounted for in the thermodynamic calculations.

CONCLUSIONS

Sheathed samples of irradiated UO_2 were heated in flowing steam to 1930 K and 2010 K, in order to investigate the volatility of iodine released from CANDU® fuel. The released fission products and structural materials were trapped on particulate and charcoal filters heated to 390 K during these two tests. The iodine volatility was measured by γ-scanning of ^{131}I within a few hours after exposure of the deposited fission products to air. No measurable quantities of volatile iodine (probably less than 0.1% of the iodine that reached the particulate filters) were found in these two tests. These results support the approach used to assess iodine volatility in containment for CANDU 6 licensing. The volatile fraction of iodine was low, despite considerable releases of Mo,

[9] Iodine volatility measurements in tests HI-1 and HI-2 should not be considered, because tungsten volatilized from the induction susceptor used for tests HI-1 and HI-2 reacted with cesium and affected iodine chemistry significantly. The tungsten susceptor was replaced with a graphite susceptor for tests HI-3, HI-4 and HI-5. Inspection of the text of Section 3.2.2 in the experimental data report for test HI-3 [14] and the amount of ^{129}I on the charcoal in Table 8 of the same report [14] indicates that the "Presumably I_2" value for HI-3 in Table III of Reference [13] should in fact be 0.003%, rather than the tabulated value of 0.4%.

which reacts with CsI and other Cs compounds and would therefore be expected to increase iodine volatility.

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