AN OVERVIEW OF RECENT WASTE CHARACTERIZATION ACTIVITIES FOR ONTARIO POWER GENERATION'S LOW & INTERMEDIATE LEVEL WASTE DEEP GEOLOGIC REPOSITORY

A. Husain[1], R. Burany[1] and K. Liberda[2]
[1] Kinectrics Inc., Toronto, Ontario, Canada
[2] Nuclear Waste Management Organization, Toronto, Ontario, Canada
aamir.husain@kinectrics.com, ruth.burany@kinectrics.com, kliberda@nwmo.ca

Abstract

Low and Intermediate Level Wastes (LLW and ILW) generated from the operations and refurbishment of Ontario Power Generation's (OPG's) owned or operated nuclear reactors are planned for disposal in a Deep Geologic Repository (DGR). Characterization data are required to support the safety case and the licensing of the DGR. Kinectrics has been involved in the ongoing characterization activities since 1999; Nuclear Waste Management Organization (NWMO) currently manages this work on behalf of OPG.

This paper presents an overview of the recent waste characterization work being performed. While increasingly focused on ILW streams such as pressure tube wastes and spent resins, additional data for a range of LLW streams such as non-processible wastes and incinerator ash are also being developed. Typically, characterization activities involve the radiochemical characterization of gamma, alpha and beta activities for each waste stream and measurement of elemental compositions. The radiochemical data are used to develop scaling factors for various alpha/beta emitting radionuclides.

1. Introduction and Background

Low and Intermediate Level radioactive wastes from Ontario Power Generation (OPG)'s owned or operated stations are presently stored at the Western Waste Management Facility (WWMF) in Kincardine, Ontario, Canada. Low Level Wastes (LLW) consists largely of bulk non-processible, compacted and incinerator wastes, low activity resin and filter wastes, and other miscellaneous wastes. Intermediate Level Waste (ILW) consists principally of spent resins and filters originating from the Moderator and Primary Heat Transport System (PHTS), and irradiated core components. The stored wastes are planned to be disposed of in OPG's Deep Geologic Repository (DGR) for L&ILW. Detailed characterization information is required to perform safety assessments in support of the licensing process for the DGR.

In general, wastes contain alpha- (α), beta- (β) and gamma- (γ) emitting radionuclides. Gamma-emitting radionuclides (e.g., Co-60 and Cs-137) are easy-to-measure (ETM), while α- emitting radionuclides (e.g., Pu-238, Cm-244) and β- emitting radionuclides (e.g., C-14, Ni-63) are difficult-to-measure (DTM). Scaling factors are, therefore, employed to estimate the concentrations of DTM radionuclides based on the concentrations of ETM radionuclides.

Ongoing waste characterization activities are focused on the need to improve statistics for the waste data, address gaps in the database and ensure that characteristics of newly generated wastes are consistent with those measured previously. Development of the database also takes advantage of opportunistic data or samples which become available from other station activities. In general, waste characterization activities include both radiochemical and chemical characterization of the various waste streams. In recent years, attention has increasingly focused on the characterization of ILW streams. Characterization method development is concurrently undertaken for new DTM radionuclide/ matrix combinations to support characterization needs for the program.

This paper focuses on some recent activities pertaining to radiochemical characterization of reactor waste. The topics listed below are covered; before presenting them, a capsule summary of the overall waste characterization work performed to date is presented in the next section.

1. Characterization of a Shield Plug
2. Characterization of Deposits in a Pressure Breakdown Device, and
3. Characterization of Pressure Tubes

2. Historical Overview of the Overall Waste Characterization Work at Kinectrics

Waste characterization activities at Kinectrics have been ongoing since the mid-1980s. However, since 1999, a more focused program geared towards the needs of the planned DGR has been in place. It was recognized that several of the radionuclides of importance to the DGR are α and β emitters, whose low concentrations often challenged then existing analytical capabilities. Additionally, because these radionuclides cannot be detected non-intrusively, quantifying their activities in large waste packages required the development of scaling factors (SF). Therefore, the focus since 1999 has been on the development of scaling factors for various DTM radionuclides, some of which are waste specific (e.g., Zr-93 in pressure tube waste), while others (H-3, C-14, Cl-36, Fe-55, Ni-63, Sr-90, Tc-99, I-129 and actinides) are typically present in several waste streams. Because of analytical limitations arising from very low concentrations in waste (e.g., Tc-99 and I-129), 'first principles' models based on fission product release from defective and tramp fuel were developed to predict scaling factors. More recently, to avoid over-reliance on empirical scaling factor relationships, a more general predictive capability was developed for radionuclides associated with in-core components, where the primary formation mechanism is via neutron activation (e.g., C-14, Co-60, Ni-63, Zr-93), although a secondary mechanism involving deposition from coolant may also exist (e.g., actinides). This capability, assessed using pressure tube data, is deemed to be equally applicable to other in-core components.

Ongoing characterization work is prioritized based on the overall assessment by OPG of data needs for various waste streams. As a result, greater focus is now being directed to characterization of core component wastes such as pressure and calandria tubes. Such materials typically become available from component life assessment programs, whereby integral pressure and calandria tubes removed from operating stations are periodically sent to Chalk River Laboratory (CRL) for examinations. Recent work at Kinectrics was based on samples cut or

scraped from small pressure tube sections received from CRL at the McMaster Hot Cell facility. This work demonstrated the usefulness and cost-effectiveness of utilizing routinely generated scrape samples to enhance the characterization database for pressure tubes.

Other ILW samples of significant interest are Primary Heat Transport System (PHTS) and moderator spent resins and filters. In 2002, several resin samples were directly sampled from 3 m^3 liners located within in-ground storage containers at the WWMF. Subsequently, resin samples were directly sampled from Darlington resin storage tanks using an extended probe; a similar technique will be employed to sample resins from Pickering storage tanks in 2016. A separate approach has also been developed to obtain resin gamma activity data when spent resins are slurry-transferred to station storage tanks; for this purpose, a gamma spectrometer is trained on the transfer line over the resin transfer duration.

For characterization of filters, the approach is different because of the difficulty of obtaining samples of filter material. Crud samples can, however, be relatively easy to obtain for characterization. The radionuclide activity distribution obtained from characterization of crud can be combined with results from in-situ gamma spectrometry of filter packages to complete the overall characterization. For this purpose, a technique was developed and implemented at the WWMF whereby spent resin filters received at the WWMF were slowly lowered through an instrumented metal donut (equipped with gamma detector) into a designated underground pipe storage liner. More recently, spent filters have been successfully gamma scanned when they are transferred from the service vessel into the transportation flask at the nuclear station.

In parallel with various ILW characterization initiatives, work is also ongoing on the characterization of bulk LLW such as non-processible waste bins and baled wastes. Individual waste packages are scanned to quantify the inventory of gamma emitting radionuclides present; the information obtained, along with detailed DTM radionuclide characterization data developed from collected samples, completes the overall radiochemical characterization of the wastes. Besides the characterization of bulk LLW, removed feeder pipe sections (also LLW) are also being characterized to develop inventories for feeder pipe wastes arising from past and future reactor retubing projects.

Table 1 presents a summary of various waste characterization activities undertaken at Kinectrics in support of the DGR. The last column provides pertinent references (available in the open literature) for the activities undertaken (proprietary reports are not referenced).

3. Characterization of Shield Plug

Figure 1 shows an overall view of the Shield Plug Assembly[1] (SPA); a cross-sectional view is shown in Figure 2. In November 2012, the outboard section of a Pickering B SPA, which included the latch mechanism, flow tube and a truncated length of the shield body, was shipped to Kinectrics for forensic examination. Whether the section is an inlet or an outlet shield plug is not known; it measured 30.7 cm in total.

[1] The SPA, housed within the liner tube of the inboard section of an end fitting, serves to shield against channel radiation fields.

Table 1: Summary of Waste Characterization Activities Undertaken at Kinectrics

Activity Undertaken	Description of Activity	Reference Documents
Chemical Composition	• Elemental composition of various LLW and ILW streams • Detailed elemental composition of pressure tubes and garter springs with an emphasis on trace element constituents	Proprietary report(s)
Analytical Method Development	• Cl-36, Pu-241 and I-129 activity in assorted waste samples • C-14, Cl-36 and Zr-93 activity in pressure tube waste • Be-10 activity in beryllium waste • Cl-36 and I-129 activity in ash	[1], [2]
Detailed Characterization and SF Development	• Assorted LLW samples including ash, non-processibles and compactables, resins and filters, feeders and steam generator tubes • Assorted ILW samples including pressure tubes, shield plug, PHTS resins and filters, and moderator resins	[3] – [10]
Theoretical SF development	• Theoretical SF estimates for C-14, Cl-36, Fe-55 and Ni-63 derived based on activation considerations • Theoretical estimates for Se-79, Tc-99 and I-129 derived from 'first principles' models based on fission product releases from defective and tramp fuel	[11] – [13]
Dose Rate to Activity Conversion Methodology	• Bulk LLW (non-processible, compacted), spent resin, bottom ash, baghouse ash and sludge, miscellaneous drums • Containers up to 2.5 m^3 in volume	[5], [14]
In-situ Gamma Spectrometry	• Low level waste containers of size 1-2.5 m^3 filled with Ash, Bales, Non-processible waste, Resin Totes, Sludge • Stored resin waste liners (3 m^3) • Refurbishment waste boxes containing pressure tubes, end fittings, shield plugs, calandria tubes, calandria tube inserts and feeders • In-station spent resin transfer lines • Filter packages during unloading into storage liner • In-station transfer from service vessel to transportation flask • Feeders, end fittings and steam generators at station	[15], [16]
Reactor Artefact Characterization	• Pressure tubes and shield plugs • Feeder pipes, steam generator tubes, and end fittings	[6], [17]
State-of-the-Art Report	• Pressure tube waste characteristics (based on an in-depth review of available CANDU literature on physical, chemical and radio-chemical characteristics of pressure tube waste	Proprietary report
ILW Resin Characterization	• PHTS and moderator resin samples have been successfully sampled from Darlington resin tanks and characterized • Plans are underway to sample resins from several Pickering tanks	[7], [8]
Assessment of ORIGEN data for core components and used fuel	• Detailed assessment of available radionuclide specific activity data to develop inventories for core component wastes such as pressure and calandria tubes • Assessments based on used fuel specific activity data to interpret measured radiochemical data for various waste streams	Proprietary report(s)

The dis-assembled section was swiped and then ultra-sonicated to remove the loose crud present before obtaining samples of various sub-component via drilling. The swipe, as well as, the drilled shavings were radio-chemically and chemically (elemental composition) characterized; some shavings were also characterized for α and β emitters.

The main constituents of the crud, consistent with PHTS materials were: iron (5.0E+05 µg/g), zirconium (6.2E+04 µg/g), chromium (5.2E+04 µg/g), and nickel (2.4E+04 µg/g). Niobium, a minor constituent, represented 2.3 % of the zirconium content of the crud, thus indicating that wear of pressure tube material likely dominated over that of Zircaloy 2 fuel cladding material. Trace constituents included cobalt (170 ppm), beryllium (9 ppm) and selenium (1.2 ppm), the activation precursors for Co-60, Be-10 and Se-79, respectively. Beryllium likely originates from the fuel-bundle bearing-pad brazing material. Co-60 was the dominant radionuclide present in the crud; significant levels of Am-241 in addition to fission products were also observed.

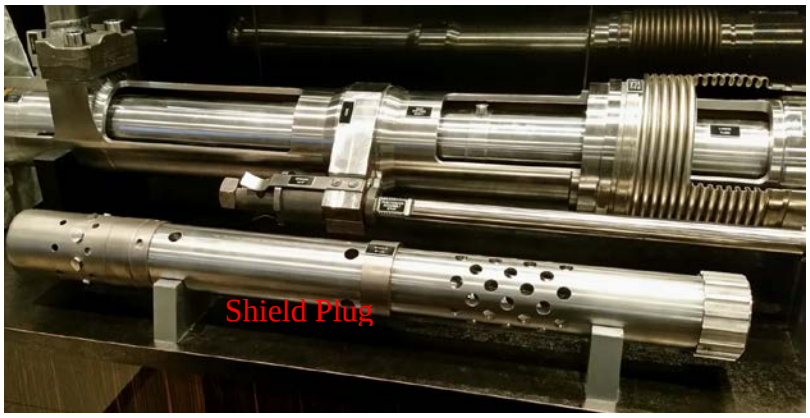

Figure 1: Overall View of a Shield Plug

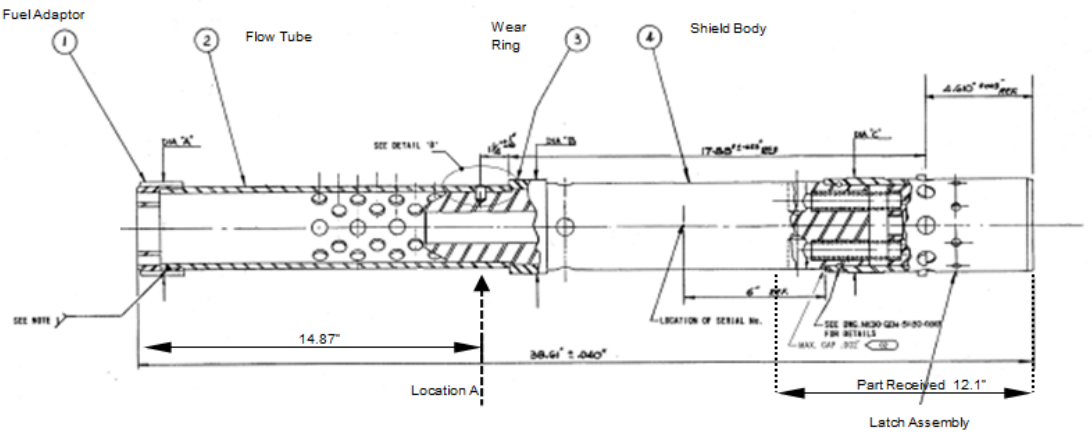

Figure 2: Cross-sectional View of the Pickering Shield Plug Assembly

The elemental composition data for the various SPA sub-components indicated SS-410 to be the principal material of construction with some sub-components being made of SS-630 and Inconel

X750. The measured cobalt content of these materials ranged approximately between 300 and 850 ppm.

Co-60 was the dominant radionuclide associated with all SPA sub-components; comparable levels of Fe-55 and Ni-63 were also present in some sub-components. Based on a comparison of the measured SPA scaling factor values for various DTM radionuclides with corresponding estimates based on ORIGEN code and also with scaling factors for other waste streams, it was concluded that:

- H-3 associated with the SPA is not indigenous but is likely picked up from the coolant,
- C-14, Co-60 and Ni-63 associated with the SPA are of indigenous origin, and
- Sr-90 and the actinides on SPA surfaces are directly deposited from fuel.

Measured specific activity results for various activation radionuclides were assessed by comparison with available ORIGIN-S code based data and found to be consistent. Based on the validated specific activity estimates and considering the axial variation of neutron flux, the overall specific activity and the total inventory of various radionuclides associated with the SPA at discharge were estimated as shown in Table 2; the latter also included the contribution from coolant-deposited activity.

Table 2: Estimated Specific Activity and Radionuclide Inventory of Shield Plug Assembly

Radionuclide	Specific Activity* (Bq/g)	Radionuclide Inventory* (Bq)
H-3	4.7E+05	1.6E+10
C-14	1.4E+03	4.8E+07
Mn-54	1.0E+06	3.5E+10
Fe-55	7.9E+07	2.7E+12
Co-60	3.4E+06	1.1E+11
Ni-63	8.1E+04	2.7E+09
Sr-90	2.5E+04	8.6E+08
Nb-94	1.3E+05	4.6E+09
Sb-125	8.1E+04	2.7E+09
Am-241	1.0E+03	3.4E+07
Pu-241	1.3E+04	4.3E+08
Pu-238	1.2E+02	4.0E+06
Pu-239/40	8.4E+02	2.9E+07
Cm-244	1.6E+02	5.5E+06

*At discharge

4. Characterization of a Pressure Breakdown Device Deposits

The Pressure Breakdown Device (PBD) is a SS-304 component of the mechanical cartridge which seals the shaft of the PHTS pump (see Figure 3); the PHTS water flows through the helical flow channels of the PBD. In order to investigate an increased pressure drop observed across several PBDs in some reactors, a PBD, which had already been removed from service in January 2010 was sent to Kinectrics for examination. The latter confirmed the presence of

deposits in the channels; this was shown to be largely beryllium hydroxide, which likely originated from the fuel bundle/bearing pad brazing material.

In 2014, radiochemical characterization of the deposits was undertaken. As the deposits were largely stable Be-9 hydroxide, the activation product Be-10, a long-lived beta emitter, was expected to be present in the deposits. Development of a Liquid Scintillation Counting method to quantify Be-10 in the deposits was, therefore, also undertaken.

Scanning Electron Microscopy (SEM) examination of a flake of the black deposit indicated that it was dual layered: its outer surface exposed to the PHTS water consisted of a highly facetted black, tetrahedral crystalline material, while its inner surface in contact with the metal had an amorphous appearance. The thickness of the deposit was found to be 137 ± 15 µm. An Energy Dispersive X-Ray Spectroscopy (EDS) analysis performed on the deposit flake (see Table 3), indicated very low levels of iron.

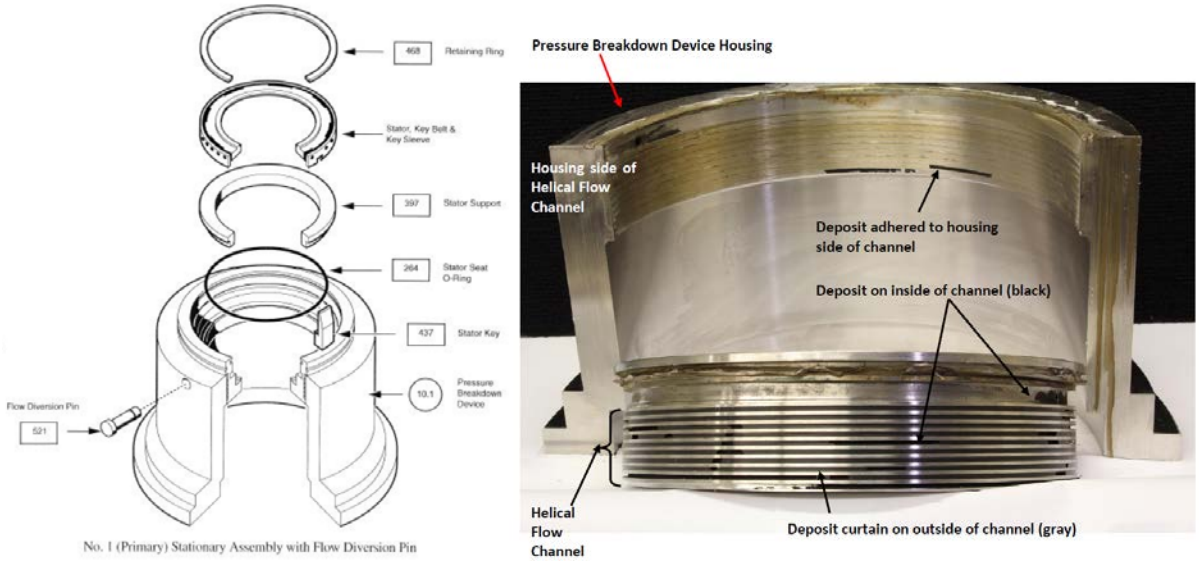

Figure 3: Pressure Breakdown Device - Exploded and Dismantled Views

Table 3: Deposit Composition Measured by EDS

	Carbon (wt. %)	Oxygen (wt. %)	Silicon (wt. %)	Iron (wt. %)
Outer Surface	5.60 ± 0.6	93.4 ± 0.6	0.3 ± 0.1	0.5 ± 0.1
Inner Surface	4.2 ± 0.5	94.4 ± 0.6	0.4 ± 0.1	0.9 ± 0.2

*Elements lighter than carbon (includes beryllium) are not detectable by EDS.

Chemical analysis of the deposit flake, measured using Inductively Coupled Plasma Mass Spectrometry (ICP-MS), confirmed that beryllium was the major (20 wt. %) elemental constituent of the deposit; other reported elements (oxygen, hydrogen and carbon cannot be measured using ICP-MS) accounted for only 1.1 wt. % of the sample. The ICP-MS elements detected along with the estimates for oxygen, hydrogen and carbon determined from stoichiometry and the data in Table 3 fully accounted for the weight of the deposit.

Divalent beryllium exists in a number of forms across the pH range of interest. As shown in Figure 4(a), it has a very low solubility between pH 6 and 12 and exists as insoluble $Be(OH)_2$ [18]. Figure 4(b) shows the predicted temperature - pH boundaries for various beryllium species based on two independent data sets [19]; despite some discrepancy, the two datasets present essentially the same picture of Be^{+2} hydrolysis, i.e., the pH range over which $Be(OH)_2$ is the prevalent species widens with increasing temperature. Clearly, the deposition of beryllium as $Be(OH)_2$ in the helical flow channels of the PBD is consistent with the observations in Figure 4.

A sample of the PBD deposits was subjected to detailed radiochemical characterization. Measured results, decay corrected to January 2010 are shown in Table 4. The equivalent activity of 7050 Bq Be-10/g beryllium agreed reasonably with the corresponding estimate of 8700 Bq/g based on neutron activation considerations. Scaling factors for DTM radionuclides based on the data in Table 4 were generally consistent with those for PHT resins and filters; the value for Be-10/Co-60 was 0.34.

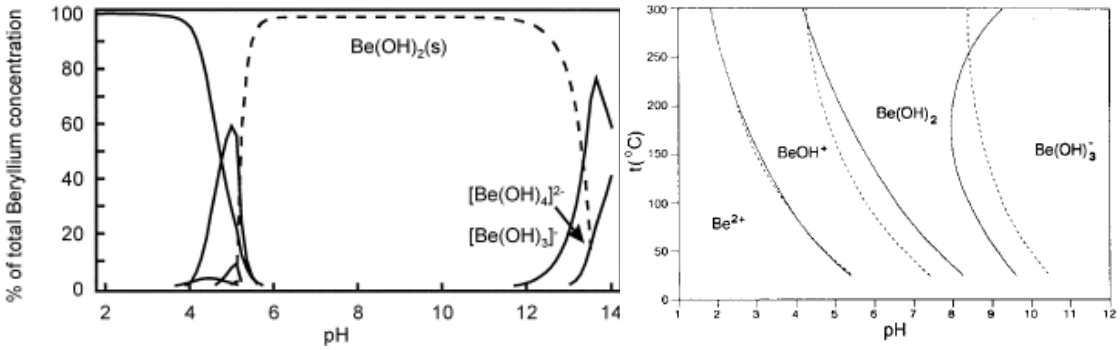

Figure 4: (a) Distribution Diagram of Beryllium Hydroxo Species (b) Stability Regions for Various Hydroxide Complexes of Be^{+2}

Table 4: Decay Corrected Activity of PBD Deposits

Radionuclide	Bq/g	Radionuclide	Bq/g	Radionuclide	Bq/g
Be-10	1410	Sr-90	9.2	Pu-239/240	3.37
C-14	5020	Nb-94	117	Pu-241	62.2
Fe-55	1330	Sb-125	701	Am-241	3.5
Co-60	4150	Cs-137	124	Cm-244	5.97
Ni-63	1200	Pu-238	0.37		

As shown in Table 4, the deposit contained Co-60 and other radionuclides that are typically associated with magnetite deposits in the PHTS. Based on the measured iron concentration (4710 µg/g), the equivalent Co-60 activity was estimated to be 0.64 Bq/g magnetite which is comparable to the value of 1.3 Bq/g magnetite based on characteristics of steam generator deposits [17]. This suggests that Co-60 and iron co-deposited in the PBD channels in like manner to their deposition on other PHTS surfaces. This is understandable as the PHTS Pump and associated PBD is located downstream of the steam generator, where the coolant is typically super-saturated and hence prone to iron precipitation. Despite this and given that the concentration of beryllium in the coolant is typically much lower than that of iron, the

preponderance of $Be(OH)_2$ in this PBD deposit suggests a period of increased wear of the fuel bundle brazing material leading to an increase in pressure drop across the PBDs.

Finally, based on the geometry of the PBD channels, average deposit thickness of 137 µm, $Be(OH)_2$ density of 1.92 g/cm^3 and considering the deposit porosity to be 50 %, the total weight of deposit associated with the PBD was estimated to be approximately 11 g. Based on a Be-10 activity of 1410 Bq/g, the total Be-10 inventory of the PBD was, therefore, estimated to be approximately 1.6E+04 Bq.

5. Characterization of Pressure Tubes

Recent work on the radiochemical characterization of pressure tubes is presented in this section.

- Pressure Tube A was removed from service after approximately 17.3 Effective Full Power Years (EFPY). Small circumferential samples, representing the full cross-section of the pressure tube wall, were cut from a mid-section of the pressure tube made available by CRL. This section had earlier been subjected to the following treatments: a) the outer surface was hydrided and then heated at 300 ˚C to homogenize the introduced hydrogen, b) the inside oxide layer was removed by honing, c) finally, the outer hydride layer was removed by immersion in an acid solution.
- Pressure Tube B was removed from service after approximately 17.1 EFPY. Scrapes obtained from its outlet ends were characterized. Two successive scrapes were obtained: the first scrape was a composite oxide/metal layer about 100 µm thick while the second was the underlying, somewhat thicker, metal layer. The first scrape had an oxide layer less than 5 µm thick. Note that the surface of the scrape samples has a saw tooth appearance; thus the two successive scrape samples are not uniformly layered but represent some inter-mixing.

Characterization results were decay corrected to the tube removal date. The data obtained represent the first set of Zr-93 and Pu-241 measurements for pressure tubes; the measured Am-241 activity was decay corrected considering the combined decay of the Pu-241/Am-241 parent-daughter pair.

Results for Pressure Tube A samples indicated a relatively high activity of Cm-244: the measured Cm-244/Pu-239/40 ratio was ~550 which is significantly greater than that measured for other waste streams (< 10); this is likely the result of prolonged pressure tube irradiation. Despite the absence of a surface oxide, sample activities, with the exception of H-3 and C-14 activities, were generally consistent with previous data for oxide-covered pressure tube samples. This is likely due to the small contribution of the relatively thin oxide to the overall pressure tube activity. Compared with previously published Pickering data [20], the C-14 and H-3 in these samples were factors of ~3.5 and ~100 lower, respectively. The significant discrepancy, particularly in H-3 activity, may be due to the aggressive sample pretreatment, which possibly caused depletion of both radionuclides.

One Pressure Tube A sample was depth profiled using a purpose-built milling jig. Three collected shavings, corresponding to cumulative thicknesses (reference plane: oxide-free inner surface) of 4.6 µm, 21.4 µm and 40.1 µm, respectively, were characterized. With the exception

of actinides, the activity data for all other radionuclides appeared to plateau out with increasing thickness. The activity of each actinide, however, consistently increased with thickness as shown in Figure 5.

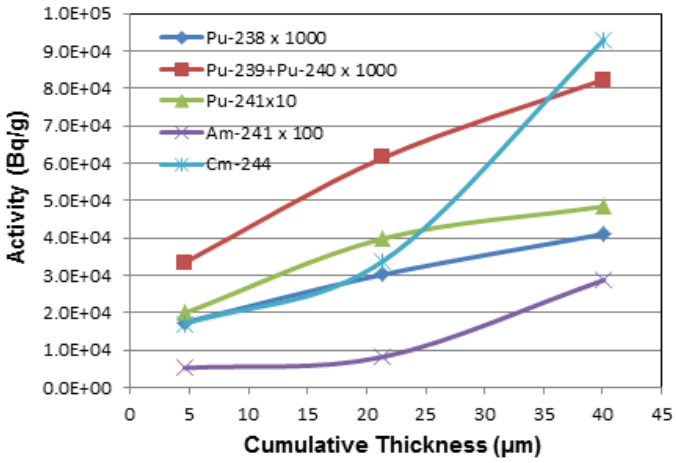

Figure 5: Profiles of Actinides across Inner Surface of a Pressure Tube A Sample

Results for the Pressure Tube B scrape samples are shown in Table 5. As in Pressure Tube A, note the relatively high Cm-244 activity. The activity results for the first and second scrapes were generally similar. The scrape sample results for Pressure Tube B in Table 5 are not inconsistent with the trends observed in Figure 5 for the Pressure Tube A sample. Those trends were observed over a distance of 40 µm from the oxide-free inner metal surface or equivalently over a distance of about 60 µm from the inner oxide surface. The increasing trends observed for Pressure Tube A were thus limited to a distance which was only about half the thickness of the first, Pressure Tube B, scrape sample.

Table 5: Characterization Results for Pressure Tube B Scrape Samples

Radionuclide	Activity (Bq/g)	
	Scrape 1	Scrape 2
H-3	1.5E+06	2.5E+06
C-14	1.8E+05	1.3E+05
Cl-36	3.8E+02	3.0E+02
Fe-55	5.9E+07	8.3E+07
Co-60	6.0E+06	6.1E+06
Ni-63	2.0E+05	3.1E+05
Sr-90	3.6E+04	1.8E+04
Zr-93	1.1E+05	1.2E+05
Nb-94	9.4E+06	9.5E+06
Cs-137	1.1E+05	7.9E+04
Pu-238	8.3E+01	6.0E+01
Pu-239/240	9.6E+01	7.2E+01
Pu-241	7.6E+03	5.4E+03
Am-241	8.3E+01	1.4E+02
Cm-244	4.2E+04	4.4E+04

6. Conclusions

A broad overview of the characterization work undertaken in support of OPG's DGR project has been presented in this paper. The characterization work has systematically addressed various L&ILW streams, taking advantage of opportunistic samples which periodically become available. Where applicable, results obtained are interpreted using a thorough understanding of the CANDU reactor operation, radiochemistry fundamentals and activation and fission product release models. The unique characterization requirements of challenging waste streams, e.g., pressure tubes, have been met by developing radiochemical analysis methods for new waste-specific radionuclides.

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