FISSION PRODUCTS MEASUREMENTS IN THE SLOWPOKE 2 REACTOR AT THE UNIVERSITY OF TORONTO

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1. INTRODUCTION

During 1995 and 1996 new measurements on fission and activation products in the water and air of the SLOWPOKE 2 nuclear reactor at the University of Toronto were performed. Due to the age of the fuel, small quantities of fission products are released during normal operation of the reactor. Low concentrations of fission and activation products can be measured in the water and air of the reactor. Studying the behaviour of fission and activation products in their natural conditions inside the reactor is very useful, both for improving the understanding of their real chemical and physical properties, and for the development and evaluation of analytical methodologies.

By careful analysis of γ spectra measured at different delay times after sampling, fifty-two radioisotopes were identified in water samples and twenty-two in air samples. A methodology for correct evaluation of the initial activity of fission products belonging to a nuclear chain was developed for the second and third members of the disintegration chain using measurements made at different times after sampling.

Adsorption and desorption of noble gases on charcoal can play an important role during any release of fission and activation products from containment to the atmosphere following a reactor accident. However previous measurements of the adsorption and desorption coefficients for Xe and Kr were not performed using reactor air as the carrier gas. Experiments conducted at the SLOWPOKE 2 Reactor permitted the study of these phenomena.

Measurements of the ratios of fission products from the same radioactive chain can give information about the time of an accident. Because of their half-lives, and of their similarity in chemical and physical properties, the measurement of any desequilibrium between ^{99}Mo ($T_{1/2}$ = 2.75 d) and ^{99m}Tc ($T_{1/2}$ =6.02 h) can give an indication of the reactor status. The first determinations of the concentrations of ^{99m}Tc at the SLOWPOKE 2 Reactor were done during these experiments.

2. EXPERIMENTAL PROCEDURE

Fission and activation product concentrations in the air and water of the U of T SLOWPOKE 2 Reactor were determinate using APTEC gamma spectrometric facilities. Three HPGe detectors with resolution between 1.8 and 2.6 keV, at 1332 keV, and relative efficiencies between 10% and 20% were used. The spectra's analyses were performed using a 486 AMPAQ computer and the APTEC spectrum's analysis program (version 5.30[1]). For efficiency calibrations ^{131}I, ^{109}Cd, ^{133}Ba and ^{65}Zn solutions were used. The IAEA-375 intercomparison sample was used to check the efficiency calibration. Our measured value for ^{137}Cs was well within the confidence interval (less than 2% difference from the recommended value).

2.1. Water measurements

The reactor core, beryllium reflector, central control rod and the irradiation systems are immersed in water (water height above the core 3.3m [2]). The circulation of water from the bottom of the container, where the reactor core produces heat, to the top is determined by normal thermal convection. In addition there is a pump system that carries the water from the container through a demineralizer system to reduce corrosion of the components and minimize the level of radioactive contamination. Every week the container water is tested including routine measurement of fission products[3]. Vials containing 20 mL of this water were analysed by repeated gamma spectrometry analysis.

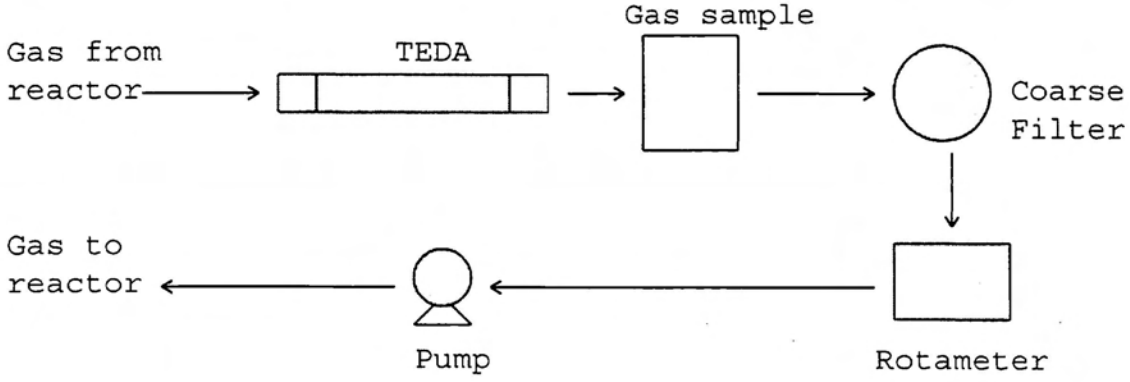
2.2. Air measurements

On the top of the reactor container there is 0.5 m^3 of air. The reactor container is purged every week with a small flow of air to remove hydrogen formed by radiolytic decomposition of the water[2,3]. Air samples can be taken with a small pump connected to the container.

The gas sampling system consisted of a filter tube filled with approximately 3cm^3 of triethyllenediamine (TEDA) impregnated charcoal, an 0.5 L erlenmeyer flask, a coarse filter (to prevent solids entering the pump), a rotameter and a pump. The charcoal was fixed in the tube using glass-wool. The glass-wool was removed before the measurements of the charcoal. A diagram of the air sampling system is presented in Fig. 2.1. The total volume of air that passed through the system during the sampling period was obtained by multiplying the average gas flow during the experiment by the aspiration time. The aspiration time was between 1 minute and one hour and the total volume of air was between 5 and 300L.

Repeated gamma measurements of air samples collected with the facility described above were performed. Both the gamma activities on the charcoal and in the sealed erlenmeyer flask were determined. During the measurements the charcoal samples were kept in an open vial. A high escape rate of the radioisotopes from the charcoal was observed

Fig. 2.1
Gas sampling system



3. INTERPRETATION OF THE SPECTRA

3.1. Identification Of The Peaks

3.1.1. Water Samples. The samples were measured at different times after collection. The gamma spectra were stored and carefully analysed later. The peak search algorithm included in the APTEC 5.30[1] spectra's analysis program was initially used. Also a manual analysis of each spectrum was performed.

One hundred and seventy-four peaks were present in the energy range between 30 keV and 1800 keV. One hundred and thirty-four peaks were attributed to individual radionuclides, to the x-ray peak and to single and double escaped peaks. Twenty-eight peaks had contributions from two radioisotopes and eleven peaks had contributions from three different radioisotopes.

Fifty-two radioisotopes were identified. They are presented in table 3.1.

Table 3.1.
Radionuclides identified in water samples

Noble gases	^{85m}Kr, ^{87}Kr, ^{88}Kr, ^{89}Kr, ^{131m} Xe, ^{133}Xe, ^{133m} Xe, ^{135}Xe ^{135m}Xe, ^{137}Xe, ^{138}Xe
Halogens	^{84}Br, ^{84m}Br, ^{131}I, ^{132}I, ^{133}I, ^{134}I, ^{135}I
Rare earths	^{140}La, ^{141}Ce, ^{143}Ce, ^{144}Ce
Alkali metals	^{88}Rb, ^{89}Rb, ^{137}Cs, ^{138}Cs, ^{138m}Cs
Metals	^{91m}Y, ^{95}Nb, ^{95}Zr, ^{97}Nb, ^{97m}Nb, ^{97}Zr, ^{99}Mo, ^{99m}Tc, ^{101}Tc, ^{101}Mo ^{123m}Sn, ^{103}Ru, ^{105}Ru, ^{105}Rh, ^{115}Cd, ^{132}Te, ^{134}Te
Alkaline earths	^{91}Sr, ^{92}Sr, ^{140}Ba, ^{141}Ba
Activation products	^{41}Ar, ^{56}Mn, ^{61}Cu, ^{239}U.

Fission products (FPs) are members of radioactive decay chains. Each member has its fission (production) yield and except for the first one, a cumulative fission yield. FP half-lives can vary between fraction of a second to many years. If in the chain there is more than one FP with a relatively long half-live, these can be identified in the sample. This fact (that the FPs are members of a radioactive chain) has important consequences for the measurement of FPs.

A typical procedure for isotope identification is the use of gamma energy tables and repetitive counts for half-life determination. If the identified FP is not the first one in the radioactive chain, the repetitive counts can not be used for the half-life determination. Some of the problems encountered during the identification procedure are presented.

In a large number of decay chains, the half-lives of the daughter products are longer than that of the parents. However for a few FP pairs, such as ^{132}I ($T_{1/2}$ = 20.8h) which comes from ^{132}Te (3.26 d), ^{91}Y (49.7 min.) which comes from ^{91}Sr (9.5h), and ^{140}La (1.67 d) which comes from ^{140}Ba (12.7 d) it is the opposite. This reversal can result in an increase in the activity of the daughter fission product over time after sampling. For example, repetitive measurements at 555.6 keV revealed the presence of a radioisotope with a slow increase in activity during the first two hours followed by a decrease in activity with a half-life of approximately ten hours. This is typical behaviour for the second or third member of a radioactive chain. By checking the energy in the library[5] this peak was identified as being due to ^{91m}Y. ^{91m}Y is a member of the ^{91}Sr, ^{91m}Y, ^{91}Y radioactive chain; its half-life is 49.71 min. It should be noted that the radionuclide library originally used [6] incorrectly put the ^{91m}Y gamma line at 557.6 keV.

A potential interference with the measurement of ^{131}I was also observed. The 362.2 keV peak of ^{88}Kr may interfere with the main peak of ^{131}I (364.5 keV) as may the 363.9 keV and 365.3 keV peaks of ^{138}Cs. Since normally the peaks of ^{88}Kr (2.84 hours half-live) and of ^{138}Cs (32.2 minutes half-life) are very intense during the first hours after sampling, when the activities of ^{131}I are relatively low (so is difficult to see other ^{131}I peaks), it is difficult to resolve the ^{131}I peak unless repetitive measurements with a good gamma spectrometric system is used (this difficulty was previously noted by Bekeris and Evans[7]). For example the water collected on October 12, 1995 from the SLOWPOKE was found to contain (4.1±0.1) Bq/L of ^{131}I, and (307±3) kBq/L of ^{88}Kr, and (515±5) kBq/L of ^{138}Cs.

<u>3.1.2 Air Samples</u>. In the gamma spectra, 103 peaks were present in the energy range between 30 keV and 2400 keV. Seventy-five peaks were attributed to individual radioisotopes, X rays, annihilation of radiation and to single and double escape peaks. Twelve were attributed to the superposition between two different contributions, nine to the superposition between three different contributions and seven were not identified. Twenty-two fission

products were identified. They are presented in Table 3.2. It cannot yet be resolved if all these isotopes were sparingly present in the reactor air as opposed to being produced through decay following sampling as is described in section 3.2.

Table 3.2.
Radionuclides identified in air samples

Noble gases	^{85m}Kr, ^{88}Kr, ^{89}Kr, ^{131m} Xe, ^{133}Xe, ^{133m}Xe, ^{135}Xe, ^{135m}Xe, ^{137}Xe, ^{138}Xe
Halogens	^{131}I, ^{133}I, ^{135}I
Metals	^{91m}Y
Alkali metals	^{88}Rb, ^{89}Rb, ^{137}Cs, ^{138}Cs, ^{138m}Cs
Rare earths	^{140}La
Alkaline earths	^{91}Sr, ^{140}Ba

All these radioisotopes were also present in the water samples.

Harnden and colab.[8] indicated that iodine isotopes were present in the reactor water but not detectable in the air from the top of the reactor container. By concentrating the air sample using adsorption on charcoal, radioiodine was detected in the present study, despite the elevated activity of noble gases that were also present. The detection of ^{131}I was confirmed by analysing the ratio of the 364 keV and 196 keV peaks in repetitive measurements. Other peaks of ^{131}I (at 284 keV and 637 keV) were also identified. For example, the charcoal used to sample air on August 9, 1996 contained (3.8±0.6)Bq which corresponds to an airborne concentration of (22±4)Bq/m^3.

Repeated measurements were done to study the time behaviour of the sample. The decrease in activity over time for the charcoal sample was greater than that due to radioactive decay. The desorption of noble gases from the sample is analysed in section 4.

3.2. Quantitative Analysis

The activity of an isotope at the time of sampling is often calculated based on the net area determined through counting based on the following equation:

$$\Lambda_0 = \frac{\lambda t_2}{1 - e^{-\lambda t_2}} * \frac{NA}{t_3} * \frac{e^{\lambda t_1}}{\varepsilon y} \qquad \text{(Eq.3.1)}$$

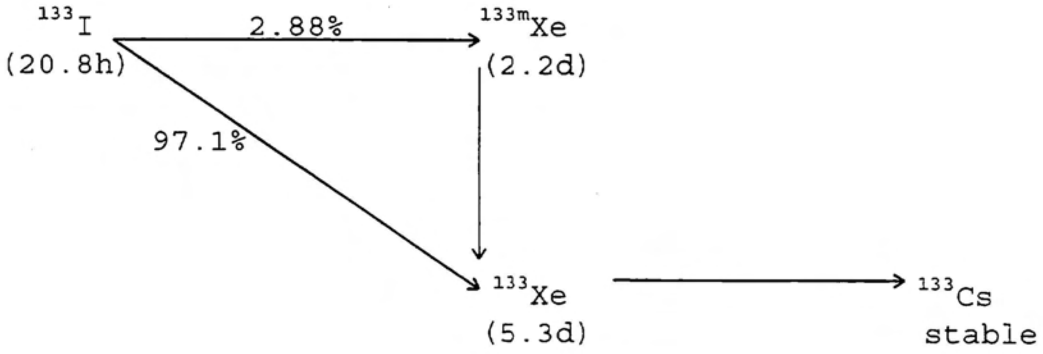
where:

Λ_0 is the initial activity, λ is the radioactive constant, t_1 is the time between sampling and the beginning of a measurement, t_2 is the true measurement time, t_3 ($t_3 < t_2$) is the live measurement time, ε is the photopeak efficiency of the detector, y is the yield of the gamma line, NA is the net area of the peak.

In the derivation of equation 3.1 a "$\Lambda = \Lambda_0 \exp(-\lambda t)$" time dependence of the activity is assumed. This time dependence of activity is valid only when the measured radionuclide is not a member of a radioactive chain. However, the fission products identified in our experiment were all members of radioactive chains. So, this formula (used also in the APTEC procedure[1]) was no longer valid, except for radioisotopes at the start of a radioactive chain. A proper procedure for time decay correction of second and third members of a radioactive chain was derived.

A typical radioactive chain that involves three measurable FPs is drawn here:

Fig. 3.1
Disintegration scheme of ^{133}I, ^{133}Xe and ^{133m}Xe



If N_i are the numbers of ^{133}I (i=1), ^{133m}Xe (i=2) and ^{133}Xe (i=3) atoms, λ_i the respective radioactive constants and f represents the decay fraction (0.0288) of the ^{133}I, the corresponding differential equation system is:

$$
\begin{aligned}
dN_1/dt &= -\lambda_1 N_1 \\
dN_2/dt &= -\lambda_2 N_2 + f\lambda_1 N1 \\
dN_3/dt &= -\lambda_3 N_3 + (1-f)\lambda_1 N_1 + \lambda_2 N_2
\end{aligned}
\qquad \text{(Eq. 3.2)}
$$

The analytical solution of the system was obtained and an QUATRO PRO computer code was build with the solution of equation 3.2. The determination of ^{91m}Y concentrations in a water sample was selected as an example for this procedure. ^{91m}Y is the second measurable member in the radioactive chain which starts with ^{91}Sr. The ^{91m}Y activities using the normal decay correction procedure (Eq. 3.1) and the correct activities, calculated using the solution of Eq. 3.2, are presented in Table 3.3.

Table 3.3

Activities of ^{91m}Y for water collected on June 28, 1996

Waiting time t_1 (s)	Counting time t_2 (s)	^{91m}Y activity (Bq/g) Eq. 3.1.	Correct ^{91m}Y activity (Bq/g)
600	810	116±5	81±15
8100	695	974±50	80±14
9600	3600	1852±90	78±14
24000	3600	44913±250	82±16

4. DETERMINATION OF THE ADSORPTION AND DESORPTION COEFFICIENTS OF XE AND KR RADIONUCLIDES ON ACTIVATED CHARCOAL

Measurements of charcoal used to sample air from the SLOWPOKE revealed the presence of ^{138}Cs, ^{138m}Cs, ^{137}Cs, ^{88}Rb and ^{89}Rb. The concentrations on the charcoal of these radionuclides are the result of their retention from the reactor air and of the disintegration of their parents adsorbed on the charcoal. The adsorption coefficient, for noble gases, K_D, ($dm^3\ kg^{-1}$), also called the distribution coefficient, was defined as [9]:

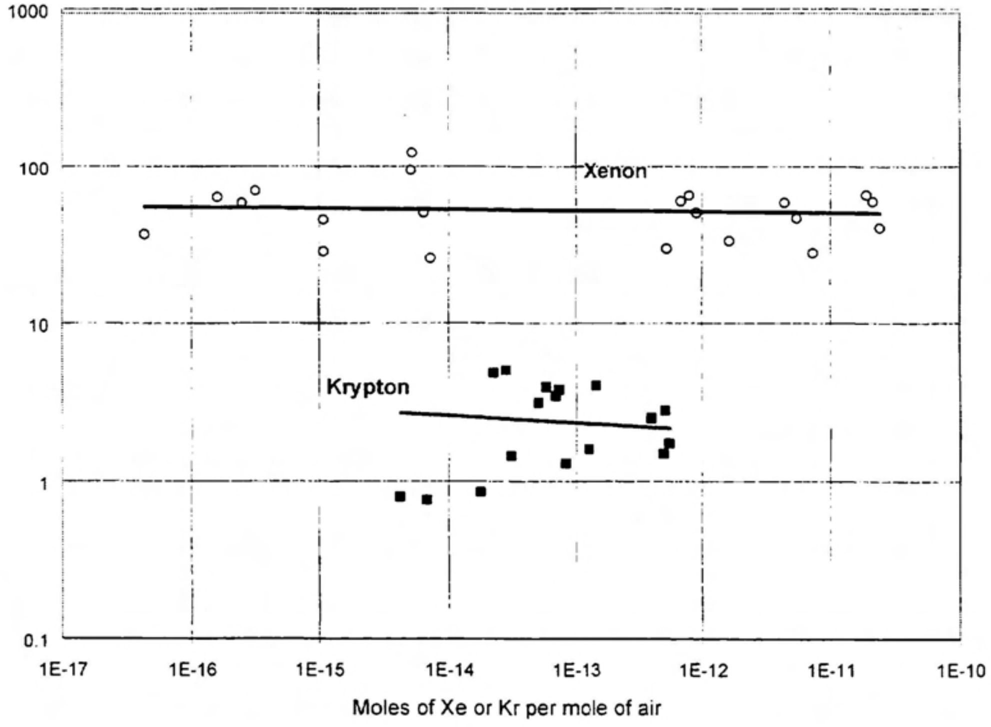
$$K_D = M/C_0 \qquad (4.1)$$

where: M = kilograms of adsorbate per kilogram of adsorbent, dimensionless, and C_0 *= airborne concentration of adsorbate, kg* dm^{-3}*.*

The values of this coefficient may be of use in safety analysis as noble gas isotopes, and their decay products, may contribute to the heating of charcoal used to prevent the release of fission products from CANDU containment structures.

The content of Xe and Kr fission products in the air of the reactor and the concentrations of these radionuclides retained by charcoal, were simultaneously determined. The humidity of the reactor air was 100% and the temperature was 20^0C. The activated charcoal was in a plastic cylindrical tube with the diameter d = 0.6 cm and the length l = 10.5 cm. The concentrations of Xe and Kr during the experiments varied from 10^{-10} to 10^{-17} moles of Xe and Kr per moles of air. The values of the retention coefficients for Xe and Kr radioisotopes are presented in Fig 4.1.

Fig. 4.1.
Adsorption coefficient for Xe and Kr [dm^3 (NTP) kg^{-1}]
Reactor air (humidity 100%, temperature 20^0C) was used as the carrier gas



A difference of one order of magnitude can be observed between the adsorption coefficients of Xe and of Kr. The retention coefficient values are in good agreement with the values reported by Underhill[9], even though there were for much higher concentrations of Xe and Kr mole fraction(between $4*10^{-4}$ and 10^{-2}) and He as the carrier gas. A dependence of the retention coefficient for Kr on sampling time was observed. That produce much of the scattered in fig.4.1. Further measurements are required in order to clarify this phenomenon. Other measurements are also necessary to determine the dependence of the retention coefficient on: the volume and geometry of the charcoal, and the humidity and temperature of the air.

Desorption of Xe and Kr on the charcoal can be described by the following equation [10]:

$$A(t) = A(0)*\exp(-k'*t) \qquad (4.2)$$

where: A(t) = activity of the radionuclide on the charcoal at time t, A(0) = maximum concentration of radionuclide at time = 0 after sampling, t = time (s), and k'= desorption coefficient (s^{-1}).

After sampling the plastic tube with the charcoal was kept in open atmospheric air and measured repeatedly. The activities of ^{133}Xe and ^{88}Kr were corrected back to the time of sampling. First determinations indicated values of k', the desorption coefficient, of $6*10^{-5}$ (s^{-1}) for ^{133}Xe, and of $7*10^{-4}$ (s^{-1}) for ^{88}Kr. Similar measurements after one day after sampling showed no presence of ^{88}Kr and a decrease in the desorption coefficient for ^{133}Xe to a value of $1*10^{-5}$ (s^{-1}). The desorption coefficient for Kr was one order of magnitude higher that for Xe. This experimental result is in accordance with the results for distribution coefficients (K_D) presented in Fig. 4.1.

As we already mentioned measurement of the charcoal used to sample air from the SLOWPOKE 2 Reactor revealed the presence of Cs and Rb isotopes. It was initially assumed that these isotopes were produced through the decay of Xe and Kr adsorbed by the charcoal. However, some ^{138}Cs and ^{88}Rb was also detected through direct collection of the reactor air in the erlenmeyer flask, suggesting that these isotopes were present as suspended aerosols produced by the decay of Xe and Kr within the reactor. A simple model was constructed in an attempt to resolve the dominant source of these isotopes, using ^{138}Cs as an example.

The equations for accumulation of Xe and Cs on the charcoal are:

$$d N_1(char)/dt = k N_1(g) - \lambda_1 N_1(char) - k' N_1(char) \quad (4.3)$$

$$dN_2(char)/dt = a f/m N_2(g) + \lambda_1 N_1(char) - \lambda_2 N_2(char) \quad (4.4)$$

where: $N_1(char)$ is the concentration of Xe on charcoal (atomskg^{-1}), $N_1(g)$ is the concentration of Xe in the reactor air (atoms*m^{-3}), k is the adsorption coefficient of Xe on charcoal ($m^3 * kg^{-1} * s^{-1}$), k' is the desorption coefficient (s^{-1}), $N_2(char)$, $N_2(g)$, and λ_2 have the same meaning and units for Cs, a is the fraction of the Cs in the reactor air to be adsorbed by the charcoal (dimensionless)*[*] *, f is the flow rate (m^3*s^{-1}) and m is the mass of the charcoal (kg).*

At steady state $dN_1(char)/dt = 0$ and hence the distribution coefficient K_D (defined according to Eq (4.1) and having units of $m^3 * kg^{-1}$) can be solved for:

$$K_D = N_1(char)/N_1(g) = k/(\lambda_1+k') \quad (4.5)$$

Using equation 4.5 to eliminate $N_1(char)$ from equation 4.4 gives:

$$dN_2(char)/dt = a*f/m*N_2(g)+\lambda_1 K_D N_1(g)-\lambda_2 N_2(char) \quad (4.6)$$

This equation allows that rate of accumulation of ^{138}Cs on the charcoal due to the decay of ^{138}Xe to be resolved from that due to ^{138}Cs in the reactor air. Preliminary evaluations showed that the majority of the ^{138}Cs on the charcoal came from the reactor air itself rather than from the decay of ^{138}Xe on the charcoal. For a value for "a" close to 1% their contributions are comparable.

5. DETERMINATION OF ^{99M}TC AND ^{99}MO CONCENTRATIONS

In a previous work [11] it was reported that the activity of ^{99m}Tc could not be measured in the SLOWPOKE water using γ spectrometry. ^{99m}Tc (half-time 6.02 hours) is the daughter of ^{99}Mo (half-time 2.75 days) and can only be determinate by γ spectrometry using its 140.5 keV peak where ^{99}Mo also has a γ peak. Because of their half-lives, these isotopes can be used to determine the time of an incident in a nuclear reactor, if their escape rate from the nuclear fuel is different.

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[*] In our experiments "a" was close to 1. If an aerosol filter were used to clean reactor air before passing through the charcoal, the value of "a" would be lower.

^{99}Mo and ^{99m}Tc are usually considered to be in equilibrium and their concentrations are reported as equal. Their concentrations were determined by using the information from the 140.5 keV peak (reported[4] as 3.8% for ^{99}Mo and 89.07% for ^{99m}Tc and in [5] as 90.7% for ^{99}Mo and 87.7% for ^{99m}Tc when equilibrium is assumed). A sample with pure ^{99}Mo and ^{99m}Tc was obtained from Shedoke McMaster Hospital. By doing repeated measurements of this sample using the 140.5 keV γ peak, the concentrations of ^{99}Mo and ^{99m}Tc at a specific time were determined. The concentrations were equal (to within the limit of the measurement's precision) indicating the isotopes were in equilibrium, as expected. The ^{99}Mo concentration for this sample was confirmed using other γ peaks.

In the SLOWPOKE 2 reactor water, the concentration of ^{99}Mo were not high enough to allow the other γ peaks (at 181.1 keV with 6.1% γ yield or at 739.4 keV with 12.1% γ yield [5]) to be determined precisely. By a careful analysis of available gamma ray catalogs [4, 5, 12] the possibility of any interference of other fission or activation products at the 140.5 keV peak was eliminated. Repeated measurements of the same sample in the same geometry with the same detector allowed us to determine from the time behaviour equation of ^{99}Mo and ^{99m}Tc, the concentrations of both radionuclides at the time of sampling. In Table 5.1, ^{99}Mo and ^{99m}Tc concentrations in different water samples are presented.

Table 5.1
Concentrations of ^{99}Mo and ^{99m}Tc in water samples from the SLOWPOKE 2 U of T Reactor

Sampling time	^{99}Mo (Bq/mL)	^{99m}Tc (Bq/mL)
28 Aug. 1995,10:07 AM	0.08±0.01	10.5±1.1
12 Oct. 1995, 03:30 PM	0.71±0.07	32.0±3.2
28 June 1996, 09:15 AM*	1.86±0.19	190.6±19.1
4 Nov. 1996, 09:58 AM	0.18±0.02	13.6±1.4
8 Nov. 1996, 09:20 AM	2.02±0.20	198.5±19.9

*$T_{1/2}$ *of the radionuclide from the counting rate at 140.5 keV pick was determined for this sample. A value of (7±1) hours was found for the measurements done on the first day after sampling and of (2.8±0.5) days for*

the measurements done a week after sampling. Also a value of (4.3±2.5) Bq/mL of ^{99}Mo was determined using the 181.1 keV peak for the same sample.

The non-equilibrium between these two radionuclides, evident from the values presented in table 5.1, cannot be explained at this time. Analysing their fission production yields indicates no important differences between them. Both have [13] independent fission yields close to 0 and their cumulative yields are 6.13 and 6.14.

6. CONCLUSIONS

The methodology developed at the University of Toronto permits an improved accuracy in measurements of the concentrations of fission and activation products in reactor water and air. Using this methodology, the adsorption and desorption coefficients on charcoal for the radioisotopes of Xe and Kr in reactor air, were determined. For the first time the concentration of ^{99m}Tc in the reactor water was evaluated. An unexpected non-equilibrium between ^{99m}Tc and its parent (^{99}Mo) was observed. The methodology and the results obtained at the SLOWPOKE 2 reactor can be easily extended to the study of the behaviour of fission products under normal and accident conditions at power reactors.

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