

ANALYSIS OF URANIUM ORE SAMPLES

6.1 Introduction

Naturally occurring uranium consists of three isotopes, namely, ^{234}U ($\tau_{1/2} = 2.46 \times 10^5$ years), ^{235}U ($\tau_{1/2} = 7 \times 10^8$ years) and ^{238}U ($\tau_{1/2} = 4.47 \times 10^9$ years) with relative abundance of 0.0056%, 0.7205% and 99.2739% respectively (Krane, 1987). Uranium ore normally contains a few hundredths of percent uranium, though extremely high-grade ore in Saskatchewan, Canada contains up to 60% uranium. Depending on the size of the ore body, uranium concentrations of greater than 100 ppm are of potential economic interest. (IAEA Tec. Doc. Series No. 186). However, before reaching any conclusion to start mining of any ore deposit, it is necessary to perform an accurate and reliable quantification of the uranium contents in it. The amount of ^{238}U present in ore samples will play a key role in deciding the feasibility of mining of the ore body. ^{238}U contents are sometime determined using CR-39 detector by measuring the ^{222}Rn concentration in the ore samples, which is formed by the decay of ^{226}Ra . As ^{226}Ra is one of the nuclides formed in the disintegration series of ^{238}U , the amount of ^{226}Ra and hence radon formed in rocks and soils depends on its ^{238}U contents.

CR-39 nuclear track detector has been intensively used in radon dosimetry, neutron dosimetry, etc. (see, for example, Matiullah et al., 1988a, b, 1990, 1991, 2001; Matiullah, 2000, Matiullah et al., 2005a, b). Therefore, in the present study we will use CR-39 based NRPB radon dosimeter, which, we believe, will yield promising, results. It is perhaps the simplest and cheapest method of determining the ^{238}U contents in the ore samples. Here ^{238}U contents in the ore samples are determined by registering the tracks due to the alpha activity of ^{222}Rn , which is the immediate decay product of ^{226}Ra . After measuring the track density, it is then related to radon concentration (and hence ^{226}Ra) using appropriate calibration factor. From ^{226}Ra concentration, ^{238}U contents are determined assuming secular equilibrium. However, this assumption may not be valid for certain ore-deposits because the progeny may or may not be in equilibrium. One of the main reasons of disequilibrium is the leachability of uranium. Under oxidizing conditions U(IV) is oxidized to U(VI) which in turn is converted into uranyl ion complexes that are highly soluble in water. This

may result in transport of the Uranium away from its location, which is then re-deposited from ground water if and when geologically favorable circumstances are met (Edsfeldt, 2001). To attain equilibrium again, another 10000 years would be required. Therefore to verify the existence of equilibrium, we have to analyse the ore samples using either high-resolution Inductively Coupled Plasma Atomic Emission Spectrometer (ICP-AES) or HPGe based gamma spectrometry technique.

6.2 Theory

^{226}Ra decays to ^{222}Rn through α emission, which then is emanated from grain to the pore spaces through recoil effect (Semkow, 1990). The amount of radon emanated from grain to the pore spaces depends on the ^{226}Ra distribution in grain, ^{226}Ra concentration, grain size, water contents in pore spaces, porosity, etc. (Sasaki et al., 2004; Duenas et al., 1997). The ratio between the amount of radon emanated from grain to the pore spaces, and amount of radon produced in the grain, is called emanation coefficient. The reported emanation coefficient ranges from 0.05 to 0.7 for different materials. For example, for sand its value ranges from 0.15 to 0.3 whereas for clay it ranges from 0.3 to 0.7 (Durrani and Ilic, 1997). Once radon is emanated to the pore spaces, it then starts diffusing and some of the radon is exhaled from ore surface to the air gas volume of the container. With the passage of time the radon build up take place in the container volume and in about $4\tau_{1/2}$ of radon, equilibrium is established. It may however be noted here that for larger value of growth time, back-diffusion may also take place which has to be taken into account. The concentration of radon in void space of the container is given by the following relation (Jang et al., 2004)

$$F_0 = \frac{C_{Rn}(\alpha A + \lambda V)}{A \left(1 - \exp\left(-\frac{\alpha A}{V} + \lambda\right)t \right)} \quad (6.1)$$

Where

F_0 = Exhalation rate ($\text{Bq}\cdot\text{m}^{-2}\cdot\text{s}^{-1}$)

C_{Rn} = ^{222}Rn concentration in void space of the container ($\text{Bq}\cdot\text{m}^{-3}$)

α = Correction term for back diffusion and is equal to λx ($\text{m}\cdot\text{s}^{-1}$)

A = Surface area of the sample (m^2)

- V = Volume of the air space of the container (m^3)
 λ = ^{222}Rn decay constant (s^{-1})
 t = Exposure time (s)

From the exhalation rate, specific activity of ^{226}Ra is calculated using the following relation.

$$C_{Ra} = \frac{F_0}{\rho \lambda E x} \quad (6.2)$$

Where

- C_{Ra} = Specific activity of ^{226}Ra (Bq.kg^{-1})
 ρ = Bulk density of the ore sample (kg.m^{-3})
 E = Emanation coefficient of the sample
 x = Thickness of the ore sample (m)

The specific activity of ^{226}Ra calculated using above equation was then used to determine the ^{238}U contents in ore samples.

6.3 Experimental Procedures

6.3.1 Setup for CR-39 Detector

Eight ore samples, collected from different sites, were ground and then dried in oven at 110°C for about 24 h till the weight of the samples became constant. The samples were then weighed and bulk densities were determined. The data obtained is given in Table 6.1. The uncertainty in, each, thickness and weight of the samples and volume of the void space listed in Table 6.1 is less than 1%. These dried samples were then placed in plastic containers of volume $5.4 \times 10^3 \text{ cm}^3$ as shown in Fig. 6.1 given below

These containers were made of polyethylene terephthalate, which is very effective barrier for radon and delays its permeation for 170 days (permeability = $8.4 \times 10^{-10} \text{ cm}^2 \cdot \text{s}^{-1}$) (Durrani and Ilic, 1997 and references quoted therein). The sample surface area was 201.06 cm^2 . Having prepared the samples setup, the next step was to install NRPB radon dosimeters within the containers. In this context, large sheets of CR-39 having $500 \mu\text{m}$ thicknesses and supplied by Page Mouldings, Ltd., UK, were cut into small pieces of size $2 \text{ cm} \times 2 \text{ cm}$. These detectors were then pasted in NRPB radon dosimeter holders. The NRPB radon dosimeters were placed in the above-mentioned ore-containing plastic containers at a distance of 25 cm (in order to avoid

the ^{220}Rn contribution to track density) from the surface of the ore sample and the containers were then hermetically sealed. It may please be noted here that NRPB radon dosimeters assure the discrimination of radon/thoron progenies (particulate daughters) by allowing only the gases to enter the dosimeter (Howarth and Miles, 2002).

Table 6.1: Characteristics of the ore samples used in present study

Sample	Type	weight (10^{-3} kg)	E	$x(\text{m})$ 10^{-3}	ρ (kg.m^{-3}) $\times 10^3$	Volume 10^{-3} m^3	T_{eff} (h)
PIEAS-1	sand	180	0.30	5.66	1.58	5.286	364.896
PIEAS-2	sand	189.5	0.30	5.97	1.58	5.280	373.632
PIEAS-3	sand	261.5	0.30	9.23	1.40	5.214	375.84
PIEAS-4	sand	271.5	0.30	9.00	1.50	5.219	367.848
PIEAS-5	clay	200	0.5	7.58	1.30	5.248	377.376
PIEAS-6	clay	283	0.5	9.96	1.40	5.200	376.632
PIEAS-7	sand	212.5	0.30	7.35	1.43	5.252	376.44
PIEAS-8	sand	220.5	0.30	7.11	1.54	5.257	376.536



Figure 6.1: Experimental setup for ore analysis using CR-39 detector.

All the dosimeters were exposed to radon for three weeks. During this time period, about 98% of equilibrium level is reached between ^{226}Ra and ^{222}Rn . This resulted in exposure of the dosimeters to variable levels of radon concentration (i.e. starting from zero concentration level to equilibrium concentration level). Therefore

effective exposure time of the NRPB dosimeters to radon was calculated using the following relation.

$$T_{effective} = t - \tau(1 - e^{-\lambda t}) \quad (6.3)$$

Where

τ = Mean life of radon (5.5 days)

t = total exposure length (days)

λ = ^{222}Rn decay constant

It may please be noted here that this type of correction is needed only for closed system (Durrani and Ilic, 1997). The effective time calculated from Eq. (6.4) is also shown in Table 6.1. After the exposure, CR-39 detectors were then etched in 25% NaOH at 80 °C for 16 h and counted under an optical microscope. The track densities were related to the radon concentration level using calibration factor of 2.7 Tracks.cm⁻².h⁻¹/kBq.m⁻³ (Miles, 2005).

6.3.2 Set-up for HPGe based Gamma spectrometry

Oven dried ore samples, weighing 200 g each, were packed in plastic bottles and then hermetically sealed. In order to establish equilibrium between ^{222}Rn and ^{226}Ra , the samples were stored for ~30 days (~8 $\tau_{1/2}$ of ^{222}Rn). Each sample was then counted for 10,000 seconds using coaxial HPGe detector having active volume of 180 cm³. The detector's relative efficiency and energy resolution at 1.33 MeV (^{60}Co) was 30% and 2.0 keV respectively. The software Canberra Genie-2000 was used for the evaluation of the spectra. The detector was calibrated with reference material, namely, soil-6 and soil-375 which were provided by the International Atomic Energy Agency (IAEA). The ^{238}U contents were calculated from the measured specific activities of ^{226}Ra (assuming secular equilibrium) and ^{235}U (using relative abundance). The specific activity of ^{226}Ra was calculated using the gamma energy 609.3 keV (yield 46.1%) of ^{214}Bi (assuming secular equilibrium) from the relation given below.

$$A = \frac{C}{t \times \% \text{ yield} \times \eta} \quad (6.4)$$

Where

A = Total Activity in sample used

C = Net Counts

t = Data collection time

η = Efficiency of the detector for the corresponding peak.

The specific activity of ^{235}U was calculated using its own gamma energy 143.76 keV (yield 10.5%). The Lower Level of Detection (LLD) at 143.76 keV and at 609.3 keV was 20 Bq.kg⁻¹ and 8 Bq.kg⁻¹, respectively.

The sensitivity of a particular system is usually expressed as a lower limit of detection (LLD). A frequently used criterion is to express two standard deviations of the background in terms of activity as the LLD.

$$LLD = \frac{\sqrt{\text{background} + \text{continuum}}}{t \times \% \text{yield} \times \text{efficiency}} \quad (6.5)$$

6.3.3 Set-up for ICP atomic emission spectrometry

The use of plasmas as excitation sources for atomic emission has become very important in recent years, and ICP spectrometers are used for multi elements determinations. The principle of this technique is given below:

Argon gas flows upward through a quartz tube, which is surrounded by a solenoid. The ICP discharge is produced by energizing the coil through the radio frequency generator operating at 5–75 MHz and 1–2 kW power, creating a changing magnetic field in the flowing gas inside the coil. This induces a circulating eddy current in a conductor (the gas), which, in turn, heats it. Argon is not a conductor at room temperature, but it can be made electrically conducting by heating it. To initiate the ICP discharge, a discharge from a Tesla coil or pilot spark is applied to the flowing argon. The argon is quickly heated, with stable plasma being produced having a core temperature of about 9000 to 10,000 K.

The sample to be analyzed, if solid, is normally first dissolved in water before being fed into the plasma. Atoms in the plasma emit light (photons) with characteristic wavelengths for each element. This light is recorded by one or more optical spectrometers and when calibrated against standards the technique provides a quantitative analysis of the original sample (Christian, 1994).

In order to prepare samples for ICP-AES, 0.5 g of each ore sample was mixed with 5 ml of concentrated HNO_3 and 20 ml of 40% HF (Hydro Fluoric acid) in a Teflon beaker which was then kept in a furnace to completely dry the sample. After drying, 2 ml of concentrated H_2SO_4 was added to the mixture and was once again kept in the furnace to remove any HF left in the sample. Finally the samples were dissolved in 4% (V/V) HNO_3 and were filled in a 50 ml volumetric flask. For background reading, a blank sample was also prepared using the above-mentioned procedure. Each sample was then analyzed for ^{238}U contents using the ARL 3580 ICP-AES of Applied Research Laboratories. This instrument is equipped with Fassel Torch and Meinhard Nebulizer having $2.0 \text{ ml}\cdot\text{min}^{-1}$ sample flow rate. The incident power of the plasma was set at 1.25 kW. The analytical line of uranium at 385.96 nm, available in the simultaneous spectrometer of the instrument, was used for the determination of the uranium. IAEA standards of uranium ore (pitchblende); S-8, S-12 and S-13 containing 0.14%, 0.014% and 0.039% of U_3O_8 , respectively, were used for the calibration of the instrument.

6.4 Results and discussion

6.4.1 Determination of ^{238}U contents using CR-39 detector

As mentioned earlier, radon exposed CR-39 detectors were etched in 25% NaOH at 80°C for 16 h and counted under an optical microscope. From the measured track densities, ^{222}Rn concentrations (C_{Rn}) in the plastic containers were determined using the calibration factor $2.7 \text{ Tracks}\cdot\text{cm}^{-2}\cdot\text{h}^{-1}/\text{kBq}\cdot\text{m}^{-3}$ (Miles, 2005). Having determined the ^{222}Rn concentrations, specific activity of ^{226}Ra (C_{Ra}) in each ore sample was calculated using Eqs. (6.1) and (6.2). ^{238}U contents in each sample were then calculated from the specific activity of ^{226}Ra assuming secular equilibrium. The results obtained are shown in Table 6.2.

In order to check the validity of the assumption of secular equilibrium, the above ore samples were re-analyzed for ^{238}U contents using ICP-AES technique. The data obtained using ICP-AES technique is shown in Table 6.3. Comparing the ^{238}U contents in the same samples using two different techniques (see Table 6.2 and Table 6.3); the ICP-AES technique gave very high ^{238}U contents. This clearly indicates that there is disequilibrium between ^{226}Ra and ^{238}U . Hence our assumption of equilibrium is invalid. Therefore, to overcome this problem equilibrium factor has to be

determined. Due to the unavailability of the ^{226}Ra standard for ICP-AES, the equilibrium factor was determined using HPGe based gamma spectrometry technique. This matter is deal with in the following section.

Table 6.2: C_{Rn} , C_{Ra} and ^{238}U contents in ore samples assuming secular equilibrium

Sample	Tracks.cm ⁻²	C_{Rn} (Bq.m ⁻³)	C_{Ra} (Bq.kg ⁻¹)	^{238}U (ppm) (CR-39)
PIEAS-1	2931±54	2975±55	317±16	26±1
PIEAS-2	3685±61	3653±60	368±17	30±1
PIEAS-3	4020±63	3961±62	290±14	24±1
PIEAS-4	3685±61	3710±61	261±12	21±1
PIEAS-5	8040±90	7891±88	454±19	37±2
PIEAS-6	7538±87	7413±85	301±12	24±1
PIEAS-7	3518±59	3461±58	311±14	25±1
PIEAS-8	1843±43	1813±42	157±09	13±1

Table 6.3: Comparison of ^{238}U contents in ore samples

Sample No	^{238}U (ppm)	^{238}U (ppm)
	CR-39 Detector	ICP-AES
PIEAS-1	26±1	743±17
PIEAS-2	30±1	431±12
PIEAS-3	24±1	465±12
PIEAS-4	21±1	393±09
PIEAS-5	37±2	2061±39
PIEAS-6	24±1	711±20
PIEAS-7	25±1	369±10
PIEAS-8	13±1	356±10

6.4.2 Determination of ^{238}U contents using HPGe detector

Here specific activity of ^{226}Ra (C_{Ra}) in each sample was determined using the gamma ray peak of ^{214}Bi (609.3 keV, yield 46.1%). From the measured specific activity of

^{226}Ra , ^{238}U contents were calculated assuming secular equilibrium between ^{226}Ra and ^{238}U . The results obtained are shown in Table 6.4.

Table 6.4: The ^{238}U contents in the sample determined using the measured specific activity of ^{226}Ra

Sample No.	C_{Ra} (Bq.kg ⁻¹)	^{238}U (ppm) (HPGe)
PIEAS-1	292.50±11.7	25±1
PIEAS-2	342.84±10.3	29±1
PIEAS-3	266.10±13.3	22±1
PIEAS-4	244.28±12.2	21±1
PIEAS-5	415.35±12.5	35±1
PIEAS-6	280.18±11.2	24±1
PIEAS-7	285.11±11.4	24±1
PIEAS-8	149.95±8.9	13±1

To avoid the assumption of secular equilibrium, an alternative and accurate method of ^{238}U contents may be the use of gamma peak, at 143.76 keV (10.5%), of ^{235}U . Due to the identical chemistry of ^{238}U and ^{235}U , they are always present in the same ratio i.e. ~ 1:138 irrespective of equilibrium or disequilibrium. Therefore ^{238}U contents were determined using the gamma peak, at 143.76 keV (10.5%), of ^{235}U . The results are shown in Table 6.5.

Table 6.5: ^{238}U contents and its specific activity determined using the measured specific activity of ^{235}U (S.A. = Specific Activity)

Sample	S.A. of ^{235}U (Bq.kg ⁻¹)	^{235}U (ppm)	^{238}U (ppm) (HPGe)	S.A. of ^{238}U (Bq.kg ⁻¹)
PIEAS-1	442.71±22.13	5.59	771±39	9514±476
PIEAS-2	246.78±14.8	3.12	429±26	5294±318
PIEAS-3	327.38±16.37	4.14	570±29	7034±352
PIEAS-4	199.65±11.98	2.52	348±21	4294±258
PIEAS-5	1063.99±31.92	13.44	1852±56	22854±686
PIEAS-6	324.90±16.25	4.10	566±28	6984±349
PIEAS-7	190.97±11.46	2.41	332±20	4097±246
PIEAS-8	143.85±8.63	1.82	250±15	3085±185

As may be seen from Table 6.4 and Table 6.5, the ^{238}U contents determined from the gamma peak of ^{235}U (143.76 keV, yield 10.5%) are higher than that determined by using the specific activity of ^{226}Ra . Moreover the specific activities of ^{226}Ra and ^{238}U shown in Table 6.4 and 6.5 differ greatly. This confirms that there is disequilibrium between ^{238}U and ^{226}Ra . The results shown in Table 6.5 are also similar to those shown in Table 6.3 with in experimental errors.

After verifying the existence of disequilibrium, we next determined the equilibrium factor by taking the ratio of the specific activities of ^{226}Ra and ^{238}U from Table 6.4 and 6.5. The results obtained for equilibrium factor are shown in Table 6.6. In order to determine the ^{238}U contents in the present ore samples (disequilibrium case) using CR-39 detectors, the specific activity of ^{226}Ra has to be corrected for equilibrium factor. Therefore the specific activity of ^{226}Ra given in Table 6.2 was simply multiplied by the respective equilibrium factor. The equilibrium factor corrected data is also shown in Table 6.6.

Table 6.6: The ^{238}U contents based on the equilibrium corrected specific activity of ^{226}Ra

Sample	C_{Ra} (Bq.kg ⁻¹) (CR-39)	Equilibrium Factor	C_{Ra} (Bq.kg ⁻¹) corrected for equilibrium factor	^{238}U (ppm) (CR-39)
PIEAS-1	317 ± 16	34	10778 ± 544	873 ± 44
PIEAS-2	368 ± 17	14	5152 ± 238	418 ± 19
PIEAS-3	290 ± 14	24	6960 ± 336	564 ± 27
PIEAS-4	261 ± 12	14.5	3784.5 ± 174	307 ± 14
PIEAS-5	454 ± 19	53.5	24289 ± 1017	1968 ± 82
PIEAS-6	301 ± 12	24	7224 ± 288	585 ± 23
PIEAS-7	311 ± 14	12	3732 ± 168	302 ± 14
PIEAS-8	157 ± 09	18	2826 ± 162	229 ± 13

For the sake of comparison, ^{238}U contents determined using CR-39 detector, ICP-AES technique and HPGe based gamma spectroscopy are shown in Table 6.7. It can be seen from Table 6.7 that the results of CR-39 and HPGe detectors are relatively in close agreement with each other. The %age difference between these two results ranges from 1–13%. However the difference between the results of CR-39 and ICP-AES is relatively on higher side (i.e. from 3–35%). The deviation amongst the

results of the above mentioned techniques may be attributed to the various factors including statistical errors, grain size, ^{226}Ra distribution in grain, etc.

To summarize, results of all the above-mentioned three techniques, namely, CR-39 detector, HPGe detector and ICP-AES are reasonably in good agreement within experimental error. To decide which of the above-mentioned methods is suitable for large scale analysis of the ore samples, several other factors have to be considered. These include the capital, running, maintenance cost and sample processing time. Taking all these factors in to consideration, CR-39 detector seems to be the best choice for determination of ^{238}U contents in the ore samples on large scale basis. This is due to the fact that it does not require any sophisticated electronics and is particularly more convenient in rugged and remote areas. However there will always remain a need of ICP-AES technique or HPGe detector to determine the equilibrium factor in case if there is disequilibrium in ore samples.

Table 6.7: Comparison of ^{238}U contents obtained from thee different techniques

Sample	^{238}U (ppm) (CR-39)	^{238}U (ppm) (HPGe)	^{238}U (ppm) ICP-AES
PIEAS-1	873 ± 44	771±39	743±17
PIEAS-2	418 ± 19	429±26	431±12
PIEAS-3	564 ± 27	570±29	465±12
PIEAS-4	307 ± 14	348±21	393±09
PIEAS-5	1968 ± 82	1852±56	2061±39
PIEAS-6	585 ± 23	566±28	711±20
PIEAS-7	302 ± 14	332±20	369±10
PIEAS-8	229 ± 13	250±15	356±10

6.5 Conclusion

To conclude, all the three techniques studied, namely, CR-39 based radon dosimeter, HPGe detector and ICP-AES technique, yield similar results within experimental errors. Keeping simplicity, cost and potential in consideration, CR-39 detector seems to be the best choice for determination of ^{238}U contents in ore samples on large scale basis. Besides laboratory use, CR-39 based dosimeter has greater potential for field studies. With such a dosimeter, a large area can be explored and analyzed for uranium ore deposits at relatively lower cost. It may however be borne in mind that equilibrium of the ore deposit has to be confirmed. In case if there is disequilibrium then equilibrium factor has to be determined using either HPGe detector or ICP-AES technique and ^{238}U contents obtained using CR-39 detector has to be corrected for equilibrium factor.

6.6 References

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