PART B

RADIOISOTOPE ACTIVITIES IN UTS-1 to UTS-4

INTERLABORATORY PROGRAM FOR RADIOISOTOPES

A fifth material, designated UTS-5, was incorporated in the interlaboratory program. This material was produced by diluting CCRMP compositional reference ore (DH-1a) that contains certified amounts of uranium, thorium, and radium-226 (3,B1) with precipitated silica powder. Secular equilibrium in both the uranium and thorium decay series is probable. Consequently, UTS-5 was incorporated as a 'yardstick' of the probable quality of the interlaboratory program, and also to test the feasibility of silica dilution or possible future control applications. Participants were not informed of the nature of UTS-5.

Eight Canadian laboratories contributed isotope activity measurements, seven of these under contract arrangements with Supply and Services Canada (Table B-1). All participants were experienced with radiochemical determinations in uranium tailings.

It was requested that isotope activities (becquerels per gram) be measured in four independently-treated subsamples of each material. Recognized reference standards having published total uncertainties (including a precision estimate at the 99% confidence level) of less than 5% were to be employed in determining instrumental calibration factors for the measurements. Sufficient count data were to be accumulated that the uncertainty component in each result due to counting statistics was less than 2% (at 1σ confidence). The expertise of the participants was otherwise relied upon to provide unbiased and precise results within their capabilities.

The radioactive isotopes measured in this project were thorium-230, radium-226, lead-210, and polonium-210 (uranium-238 decay series); and thorium-232, radium-228, and thorium-228 (thorium-232 decay series, measured in UTS-1, UTS-2 and UTS-5 only). In addition, one laboratory provided measurements of thorium-232 and thorium-228 in UTS-3 and UTS-4, and protactinium-231 in all materials.

The analytical results are presented in Tables B-2 to B-11. The calibration standards are identified in Table B-14. Radiochemical methodologies are summarized in Table B-12; contractor reports and references cited should be consulted for more detailed descriptions.

CONSENSUS ACTIVITY VALUES: STATISTICAL TREATMENT OF RESULTS

The grand mean of all individual determinations exclusive of statistical outlier sets is defined as the consensus value for the activity of an isotope for purposes of this program. Dixon's 'r' statistic for the extreme laboratory mean values was used to identify laboratory sets having a low probability of belonging to the assumed consensus universe (B2). A level of significance $\alpha = 0.05$ ($r_{.05}$) was used for this purpose. The suspected sets were excluded from the calculation of the consensus value and its uncertainty. Four instances of such apparent outliers at this confidence level were detected in the 29 consensus determinations herein (Table B-17). However, the positive result in one case (Ra-228 in UTS-1) was deemed to arise from a fortuitously small variance in the small set of peer means, and was consequently not declared as an outlier set.

Results of analysis of variance (Tables B-15 and B-16) indicate that the between-laboratory component is generally significant relative to the within-laboratory component as has been observed to be the norm in chemical analysis consensus programs.

The consensus activity values for UTS-1 to UTS-4, their uncertainty estimates, and associated statistical parameters are compiled in Table B-18. The confidence limits (CL) estimate the range of reproducibility of the mean in 95% of cases were the identical program to be repeated many times. The average within-laboratory relative standard deviation (CV, %) and the relative standard deviation of the individual determinations from the grand mean (RSD, %) provide an indication of how closely an individual determination approaches respectively, the laboratory estimate and consensus value on the average for general comparative purposes.

The corresponding results for UTS-5 are compiled separately in Table B-19; a 'bias estimate' comparing the consensus and predicted activity values is included for this material.

DISCUSSION

Results for UTS-5

UTS-5 was incorporated in the consensus program as a 'blind control' material to indicate potential biases in laboratory methods and consequently in the consensus values. Gamma-ray and uranium concentration measurements indicated that a homogeneous dilution of DH-1a (49.4 ± .6%, 2σ) in the silica was accomplished in the blending of this material (B3). From the certified uranium and thorium concentrations in DH-1a and published half-lives, the activities of uranium-238, uranium-235 and thorium-232 in UTS-5 are 15.86 ± .32, 0.744 ± .018, and 1.82 ± .10 Bq g^{-1} respectively, with uncertainty estimates propagated at the 99% confidence level (B1,B4,B5,B6). The consensus value for radium-226 in DH-1a in a thirteen-laboratory consensus program was 31.5 ± 1.1 Bq g^{-1} essentially confirming uranium-radium secular equilibrium (3). It is inferred that there is a high probability that thorium-230 is also in secular equilibrium. Radon emanation rate measurements of DH-1a suggest that lead-210 and polonium-210 may be 3 to 5% or more below secular equilibrium values if these rates existed in the natural ore state (B7,B8,B9). Polonium-210 in DH-1a was measured as 31.3 ± 1.1 Bq g^{-1} (95% CL), however, suggesting equilibrium (B9). In view of the relatively short half-lives of radium-228 and thorium-228 and the nature of DH-1a, it is highly probable that these isotopes are in secular equilibrium with thorium-232.

The consensus program results for UTS-5 are compared with the predicted (secular equilibrium) conditions in Table B-19. In all cases, the consensus means are in agreement with the predicted values, within the 95% confidence limits. The 'bias estimate', i.e., the relative departure of the consensus mean from the predicted value, is well within the 95% confidence interval estimate for each isotope. The bias estimate approaches the 95% confidence limit only for polonium-210. The single laboratory result for protactinium-231 suggests either a slightly low methodological bias or a lower than equilibrium activity for this isotope in UTS-5.

It is inferred that the consensus means, within the statistically determined confidence intervals, are likely to provide reliable estimates of the activities present in the tailing materials, provided that very different matrix or spectral interference effects are not encountered.

Results for UTS-1 to UTS-4

The recommended values for isotope activities for the tailings reference materials UTS-1 to UTS-4 are compiled in Table B-18.

The recommended activities of uranium-238 decay series isotopes in UTS-1 are not significantly different, i.e., their 95% confidence intervals overlap at about 3.5 Bq g^{-1}. Thus thorium-230, radium-226, lead-210, and polonium-210 are close to, if not in, secular equilibrium in UTS-1. Thorium may be slightly depleted relative to radium in UTS-2 ($^{230}Th/^{226}Ra$ = 0.8 ± 0.2), as may be lead-210 and polonium-210.

Thorium-230 is found to be depleted relative to radium-226 in UTS-3 and UTS-4 with activity ratios of .85 ± .07 and .59 ± .08 (2σ) respectively. Thorium-230 is similarly depleted relative to lead-210 in UTS-4 (Th/Pb .71 ± .14). Within the measurement uncertainties, polonium-210 is completely supported by lead-210 in both materials (activity ratios of .94 ± .09 and .95 ± .18 respectively). Similarly lead-210 may be completely supported by radium-226 in UTS-3 (activity ratio of .95 ± .06). Lead-210 may be slightly depleted in UTS-4 ($^{210}Pb/^{226}Ra$ = 0.84 ± 0.09).

There is no significant evidence for disequilibrium between thorium-232, radium-228, and thorium-228 in UTS-1 or UTS-2. It was decided to incorporate only the α-spectrometry measurements of thorium-232 in the radiochemistry consensus program, as neutron activation measurements of thorium are being incorporated in the chemical analysis certification program for these materials. Thorium-232 activities, calculated from chemical thorium values (Part A, Table 2) and the specific activity value 4.04 Bq mg^{-1} (B5), are 0.56 and 0.70 respectively for UTS-1 and UTS-2. These are about 25% lower than the recommended activities derived from radiochemical measurements; however the uncertainties allow for substantial agreement of both sets of results.

Comments on Precision and Accuracy

The confidence intervals generally indicate the current extent of agreement between participating laboratories as to the activities that are present in these Canadian tailings materials. On the average (over the five materials) the relative 95% confidence intervals are radium-226 (±6.5%), thorium-230, lead-210, and polonium-210 (all about ±13%), thorium-232 by alpha spectrometry (±19%), thorium-228 (±24%), and radium-228 (±34%)*.

The relative standard deviations of individual values from the consensus means provide a comparative indication of how closely a single determination can be expected to approach an interlaboratory consensus value at present (Tables B-18 and B-19). Averaged over the five materials, these are 17, 10, 10 and 12% respectively for thorium-230, radium-226, lead-210 and polonium-210. Averaged over the three materials the values are 19, 20 and 22% for thorium-232 (alpha-spectrometry), radium-228 and thorium-228.

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*The confidence intervals are of course weighted by the numbers of determinations and laboratories which are considerably different for the uranium-238 series and thorium-232 series isotopes in this program (11,B2).

Similarly, the values of average within-laboratory relative standard deviation (CV, %) indicate how closely an individual determination may be expected to approach the mean value of a number of measurements within an 'average' participating laboratory. Averaged over the materials, the values are 10, 6, 7 and 5% respectively and 12, 11 and 10% respectively for the uranium-238 series and thorium-232 series as above.

With the exception of lead-210 determinations, no consistent evidence for relative methodological bias between laboratories could be deduced from an examination of laboratory mean values for all materials. In the case of lead-210 the mean results from laboratory 2 were consistently the highest in all materials. If relative bias exists in other determinations, it is sufficiently small to be masked by the within-laboratory mean uncertainties. Some partial trends are observed; however, these are inconclusive in view of the small amount of data. That the laboratory mean values are otherwise apparently randomly distributed lobbies in favour of the hypothesis that the consensus mean, within its statistical uncertainty, is the best available estimator of the true value from the program data. The validity of this approach is supported by the results for UTS-5.

Note must be taken, however, of the results found to lie outside of the consensus, particularly in view of the strong between-laboratory components of variance and the statistically small number of laboratories involved in the program. Inclusion of sets declared as outliers would result in recommended values remaining within the current 95% confidence intervals, but would approximately double these uncertainties. The apparent disagreements do not appear to stem from consistently observed methodological or calibration differences.

The relatively high degree of between-laboratory accord in the determinations of radium-226 is noteworthy. This probably arises from a combination of the use of established separation and tracer methodologies and a generally higher degree of experience with the isotope than with others, the use of common-source high-precision calibration standards, and possibly a less idiosyncratic nature of radium-226 towards matrix and spectral interference problems.

The accuracy of the results herein, of course, can be no better than the accuracy of the instrumental calibrations by which they were obtained. Calibrations, for the most part, were obtained using commercially available isotope reference materials bearing certified total uncertainties (Table B-19). The uncertainties in the calibration standards are not explicitly incorporated in the statistical uncertainties reported herein. Any significant differences in calibration parameters, laboratory-to-laboratory, would of course be reflected in the between-laboratories variance and hence be incorporated in the recommended value uncertainty. Such consistent biases are apparently masked by larger within-laboratory variances with the one exception noted above.

The NBS radium standards used in the present work are certified as to radium mass; however, the uncertainty estimated for the specific activity and half-life of radium-226 of ±0.44% (3σ) increases the relative uncertainty in the calculated activity only slightly (B10).

Calibration solutions containing thorium-232, radium-228 and thorium-228 prepared from the 1906-refined thorium nitrate hydrated salt bear activity uncertainties propagated from uncertainties in the mass of thorium present* and the specific activity of thorium-232 (B11,B12). The activity uncertainties of all three isotopes are probably less than 5% in view of the age of the salt. This material (AM TYS-1) is employed as a calibration source for the alpha-spectrometry determinations of thorium isotopes and the beta-counting determination of radium-228 by several participants.

Laboratory 4 determined the activity of a thorium-228 solution relative to that of thorium-232 in a thorium nitrate solution from alpha activity ratios measured in a standard additions procedure. The thorium-230/thorium-228 activity ratios in spiked samples were then used to deduce thorium-230 activities. The ratios in unspiked samples were used to correct for the natural thorium-228 present, however, this interference was negligible except in the case of UTS-5. The activities of thorium-232 and thorium-228 in UTS-1, UTS-2 and UTS-5 were determined relative to thorium-230 from the alpha activity ratios from the unspiked samples.

Secular equilibrium assumptions are implicit in the use of chemical calibration standards by laboratory 1 (monazite sand for thorium-228 and radium-228), laboratory 4 (aged thorium oxide for radium-228), and laboratory 7 (BL-5 for thorium-230). The assumptions in the former two cases are probably reasonable from age considerations, and in the latter case by inference from indicated radium-226/uranium-238 secular equilibrium (3).

Laboratory 8 employed commercial americium-241 and lead-210 thin sources to determine the geometry/efficiency calibration for its alpha spectrometer and beta proportional counter respectively. Calibrations were confirmed with a standard radium-226 solution and other sources. This laboratory reported a problem with apparent changes in the activity concentration of radium-226 standard solution stored in a polyethylene bottle.

Calibrations for the determinations of lead-210 and polonium-210 were otherwise exclusively performed with commercial certified solutions

*One lot of this material was found to contain about 3.4% of sulphate and 40.75 ± .05% thorium vs. 39.45% for the formulation $Th(NO_3)_4\ 6H_2O$ (B11).

of lead-210/bismuth-210/polonium-210 (AM RBZ), polonium-210, or polonium-208. Polonium is notorious for plating out on containers and the RBZ reference solution certificate bears a caveat that there is no guarantee that the polonium remains in solution. If this occurred in calibrating solutions but not correspondingly in samples, a high bias in reported values would result. This is not indicated in the results for UTS-5.

No obvious general correlation was noted between the magnitude of activity (or nature of the material) and the magnitude of uncertainty of a given isotope. Laboratory 5 indicated that within-laboratory reproducibilities experienced for thorium-230 and radium-226 in UTS-4 were poorer than expected, and that gamma-ray measurements of radium-226 in UTS-3 and UTS-4 differed significantly from alpha spectrometry results, both for unknown reasons. Similarly, laboratory 6 reported 'poorer-than-expected' precisions or extended replicate determinations of radium-226 and lead-210 in UTS-2. Laboratory 8 found that natural barium in some materials degraded their alpha spectral resolution, sufficiently that tailing corrects were required, thereby raising the uncertainty.

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Table B-1 – Contributing laboratories for radiochemical determinations*

Lab-1**	Nuclear Activation Services Hamilton, Ontario (E.L. Hoffman)
Lab-2**	Chemex Laboratories Ltd. North Vancouver, British Columbia (M. Legeyt)
Lab-3	Monenco Analytical Laboratories Calgary, Alberta (J. Dean, N. Chiu) Contract 15SQ.23440-2-9145-4
Lab-4	Physics Department, University of Calgary Calgary, Alberta (C.J. Bland, P. Jarvis) Contract 15SQ.23440-2-9145-5
Lab-5	Saskatchewan Research Council Saskatoon, Saskatchewan (G. Smithson, V. Penner, M. Knelson, R. Ortlepp, L. MacDonald) Contract 15SQ.23440-2-9145-1
Lab-6	Eldorado Resources Ltd., Research and Development Division Ottawa, Ontario (M.C. Miedema, G.A. Dunlop, R. Jones, G.B. Moodie) Contract 15SQ.23440-2-9145-2
Lab-7	CANMET, Mineral Sciences Laboratories, Chemical Laboratory Ottawa, Ontario (J.L. Dalton, R. McCorkell, M. DeGagne)
Lab-8	Waterloo Research Institute, University of Waterloo Waterloo, Ontario (H. Sharma, B. Hauk) Contract 15SQ.23440-2-9145-6

*Supply and Services Canada contracts as indicated, contractor reports filed with CANMET Research Program Office and National Uranium Tailings Research Program Office.
**Sub-contracted by Materials Research Laboratories Ltd., Nepean, Ontario (S.K. Singh), contract 15SQ 23440-2-9145-3.

Table B-2 – Analytical results, laboratory means and standard deviations for ^{238}U-series isotopes in UTS-1

	Bq g^{-1}						Mean	S.D.
^{230}Th Lab-2 (α)	2.37	2.74	2.59	2.48			2.545	.158
Lab-3 (α)	3.52	4.81	3.52	5.37			4.305	.935
Lab-4 (α)	4.91	4.56	3.70	4.37			4.385	.509
Lab-5 (α)	3.83	3.46	4.04	3.64			3.743	.249
Lab-6 (α)	3.01	3.15	3.21	3.02			3.098	.098
Lab-7 (α)	2.59	3.77	4.11	3.29			3.440	.659
Lab-8 (α)	3.50	3.63	3.77	3.79			3.673	.135
^{226}Ra Lab-1 (α)	2.9	3.3	4.8	3.7			3.68	.82
Lab-3 (α)	4.03	3.81	3.42	3.59			3.713	.265
Lab-4 (α)	3.88	3.60	3.80	4.13			3.853	.219
Lab-5 (gα)	3.53	3.53	3.47	3.48	3.49		3.500	.028
Lab-5 (γ)	3.49	3.52					3.505	.021
Lab-6 (Rn)	3.38	3.65	3.44	3.33			3.450	.141
Lab-7 (α)	3.81	3.40	3.98	3.85			3.755	.245
Lab-8 (α)	3.79	4.04	3.59	4.15			3.893	.252
^{210}Pb Lab-2 (β)	3.52	3.44	3.48	3.52			3.490	.038
Lab-3 (β)	3.00	3.03	3.18	3.00			3.052	.086
Lab-4 (α, ^{210}Po)	3.33						3.33	
Lab-5 (β)	3.31	2.94	3.07	3.15	3.12	2.90	3.082	.149
Lab-6 (β)	3.27	3.24	3.23	3.15			3.223	.051
Lab-7 (β)*	2.52	2.40	2.52	1.81			2.313	.340
Lab-8 (β)	3.70	2.72	4.08	3.34			3.460	.579
^{210}Po Lab-2 (α)	3.44	3.22	3.26	3.03			3.238	.168
Lab-3 (α)	2.8	2.9	2.8	2.8			2.82	.05
Lab-4 (α)	3.60	3.77	3.46	3.34			3.542	.185
Lab-5 (α)	2.92	2.92	2.89	2.81	2.89	2.96		
	2.81						2.886	.057
Lab-6 (α)	3.00	2.81	3.03	2.85			2.922	.109
Lab-8 (α)	3.48	3.44	3.67	3.66			3.562	.120

*Outlier set

Table B-3 – Analytical results, laboratory means and standard deviations for ^{238}U-series isotopes in UTS-2

	Bq g^{-1}						Mean	S.D.
^{230}Th Lab-2 (α)	3.26	3.22	3.44	3.33			3.312	.096
Lab-3 (α)	5.84	3.57	5.98	4.70			5.108	1.120
Lab-4 (α)	4.96	4.08	4.80	4.62			4.610	.392
Lab-5 (α)	3.87	4.08	3.78	3.80	3.72		3.850	.139
Lab-6 (α)	3.06	3.33	3.18	3.14			3.179	.113
Lab-7 (α)	4.74	4.66	4.37	4.63			4.600	.160
Lab-8 (α)	6.22	6.66	6.77	6.70			6.588	.249
^{226}Ra Lab-1 (α)	5.9	5.5	5.9	7.3			6.15	.75
Lab-3 (α)	6.36	4.91	4.74	4.55			5.140	.827
Lab-4 (α)	6.25	5.84	6.06	6.07			6.055	.165
Lab-5 (gα)	5.80	5.80	5.89	5.65			5.785	.100
Lab-5 (γ)	5.64	5.00					5.320	.453
Lab-6 (Rn)	5.01	6.06	5.06	7.01	4.77	6.19	5.416	.910
	5.68	3.48	5.76	5.13	5.42			
Lab-7 (α)	6.10	5.65	6.07	6.03			5.965	.205
Lab-8 (α)	5.35	4.72	5.08	4.88			5.003	.270
^{210}Pb Lab-2 (β)	4.85	4.70	4.60	5.33			4.870	.323
Lab-3 (β)	4.40	4.44	4.22	4.29			4.338	.101
Lab-4 (α, ^{210}Po)	4.51						4.51	
Lab-5 (β)	4.62	4.67	4.49	4.65	4.93	4.71	4.721	.127
	4.69	4.82	4.85	4.76				
Lab-6 (β)	5.48	4.19	3.69	3.99	5.17	4.00	4.430	.778
	5.59	4.84	4.66	3.15	3.97			
Lab-7 (β)*	3.77	3.26	2.81	3.96			3.450	.519
Lab-8 (β)	3.82	3.75	4.75	5.24			4.390	.727
^{210}Po Lab-2 (α)	4.92	4.81	5.18	4.14			4.762	.443
Lab-3 (α)	4.2	4.3	3.66	4.5			4.165	.359
Lab-4 (α)	5.44	5.43	5.69	5.52			5.520	.120
Lab-5 (α)	3.88	4.25	3.88	3.88	4.37	4.07	4.127	.224
	4.44	4.37	4.00					
Lab-6 (α)	4.26	4.33	4.66	4.36			4.403	.177
Lab-8 (α)	4.11	3.65	3.48	3.50			3.735	.302

*Outlier set

Table B-4 – Analytical results, laboratory means and standard deviations for ^{238}U-series isotopes in UTS-3

	Bq g^{-1}						Mean	S.D.
^{230}Th Lab-2 (α)	10.2	10.0	10.5	11.0			10.42	.096
Lab-3 (α)	11.4	12.1	11.9	12.2			11.90	.356
Lab-4 (α)	11.6	13.0	13.7	13.2			12.88	.900
Lab-5 (α)	11.5	11.7	12.6	11.3	11.1		11.64	.581
Lab-6 (α)	10.9	10.6	10.4	10.4			10.58	.236
Lab-7 (α)	12.5	10.7	11.1	9.7			11.00	1.160
Lab-8 (α)	11.0	11.3	10.2	10.4			10.73	.512
^{226}Ra Lab-1 (α)	14.6	14.6	14.6	7.3			12.8	3.7
Lab-3 (α)	13.0	14.0	13.4	14.3			13.68	.585
Lab-4 (α)	14.4	14.9	14.5	13.7			14.38	.499
Lab-5 (gα)	12.9	12.5	12.8	12.9			12.78	.189
Lab-5 (γ)	14.6	14.4					14.5	.14
Lab-6 (Rn)	13.1	12.0	12.8	12.9			12.70	.483
Lab-7 (α)	13.9	13.2	13.8	13.6			13.63	.310
Lab-8 (α)	12.2	12.0	12.9	13.4			12.60	.668
^{210}Pb Lab-2 (β)	13.1	13.9	14.1	13.3			13.60	.476
Lab-3 (β)	11.5	12.1	12.3	13.2			12.28	.704
Lab-4 (α,^{210}Po)	11.4						11.4	
Lab-5 (β)	12.8	12.4	11.5	12.3	13.3	12.2	12.84	.814
	13.4	13.7	14.0					
Lab-6 (β)	12.0	12.7	11.9	12.7			12.32	.435
Lab-7 (β)	10.8	12.8	14.8	9.29			11.92	2.397
Lab-8 (β)	13.9	13.2	13.1	11.9			13.02	.830
^{210}Po Lab-2 (α)	13.2	13.6	13.7	12.7			13.30	.455
Lab-3 (α)	11.7	10.1	11.8	10.9			11.12	.793
Lab-4 (α)	11.8	11.0	11.2	11.2			11.30	.346
Lab-5 (α)	11.84	11.84	11.84	11.65	12.02	11.84	11.88	.241
	12.21	12.21	11.47					
Lab-6 (α)	11.11	9.99	11.03	10.99			10.78	.529
Lab-8 (α)	12.8	13.1	12.3	12.3			12.62	.395

Table B-5 – Analytical results, laboratory means and standard deviations for ^{238}U-series isotopes in UTS-4

	Bq g^{-1}						Mean	S.D.
^{230}Th Lab-2 (α)	22.5	23.4	22.8	23.2			22.98	.404
Lab-3 (α)*	40.8	42.7	36.6	41.5			40.40	2.652
Lab-4 (α)	24.2	24.2	24.5	23.1			24.00	.616
Lab-5 (α)	16.6	16.5	18.6	20.6			18.08	1.941
Lab-6 (α)	24.4	24.6	22.6	23.0			23.65	.998
Lab-7 (α)	27.2	17.9	27.1	24.6			24.20	4.369
Lab-8 (α)	23.6	26.2	24.1	24.7			24.65	1.127
^{226}Ra Lab-1 (α)	38.6	47.6	47.6	40.2			43.0	5.51
Lab-3 (α)	37.1	40.0	38.7	38.9			38.68	1.196
Lab-4 (α)	39.4	39.9	40.4	39.1			39.70	.572
Lab-5 (gα)	39.2	31.2	38.0	31.4	37.1	37.0		
	32.1						35.14	3.433
Lab-5 (γ)	35.9	35.6					35.75	.212
Lab-6 (Rn)	39.4	36.5	35.0	39.5			37.60	2.223
Lab-7 (α)	38.0	39.7	39.3	39.6			39.15	.785
Lab-8 (α)	40.5	39.4	42.7	40.7			40.82	1.374
^{210}Pb Lab-2 (β)	36.7	35.3	36.6	37.0			36.40	.753
Lab-3 (β)	33.0	30.7	32.0	33.7			32.35	1.303
Lab-4 (α ^{210}Po)	30.6						30.6	
Lab-5 (β)	30.4	29.2	28.8	34.6	33.4	28.9		
	31.5						30.97	2.303
Lab-6 (β)	35.9	35.2	35.9	35.6			35.65	.332
Lab-7 (β)	30.0	31.4	35.8	32.6			32.45	2.473
Lab-8 (β)	28.8	26.2	26.9	29.7			28.40	1.176
^{210}Po Lab-2 (α)	34.8	34.6	34.5	38.0			35.48	1.685
Lab-3 (α)	23.5	25.4	23.0	24.5			24.10	1.068
Lab-4 (α)	35.3	37.8	32.1	37.4			35.65	2.608
Lab-5 (α)	29.6	30.3	28.9	28.1	31.1	27.8		
	29.6	29.6					29.38	1.090
Lab-6 (α)	33.6	34.0	33.9	36.4			34.48	1.295
Lab-8 (α)	27.9	27.4	26.9	27.6			27.45	.420

*Outlier set

Table B-6 – Analytical results, laboratory means and standard deviations for ^{238}U-series isotopes in UTS-5

	Bq g^{-1}						Mean	S.D.
^{230}Th Lab-2 (α)	15.9	16.5	16.4	16.4			16.30	.271
Lab-3 (α)	14.6	14.8	14.6	14.1			14.52	.613
Lab-4 (α)	15.6	15.3	16.0	16.3			15.80	.440
Lab-5 (α)	15.7	15.6	15.9	15.8	16.3	15.8	15.60	.294
	15.1							
Lab-6 (α)	15.3	15.7	14.3	15.8			15.28	.685
Lab-7 (α)	16.1	18.5	16.6	18.2			17.35	1.179
Lab-8 (α)	13.0	12.4	13.4	12.2			12.75	.551
^{226}Ra Lab-1 (α)	21.6	22.0	18.3	18.3			20.05	2.027
Lab-3 (α)	13.9	14.2	14.7	15.3			14.52	.613
Lab-4 (α)	16.7	16.9	16.5	16.6			16.68	.171
Lab-5 (gα)	15.3	16.3	15.7	15.6	15.1	15.0	15.83	.764
Lab-5 (γ)	17.8	17.5					17.65	.212
Lab-6 (Rn)	16.2	14.8	16.1	15.0			15.52	.727
Lab-7 (α)	15.6	16.3	15.8	16.5			16.05	.420
Lab-8 (α)	16.1	15.3	15.1	15.7			15.56	.444
^{210}Pb Lab-2 (β)	16.4	17.0	17.3	16.9			16.90	.374
Lab-3 (β)	13.4	13.9	13.8	14.8			13.98	.591
Lab-4 (α, ^{210}Po)	13.8						13.8	
Lab-5 (β)	14.0	14.1	14.4	14.5	14.6	14.5	14.27	.327
	13.7							
Lab-6 (β)	14.3	14.8	14.7	14.7			14.62	.222
Lab-7 (β)	17.7	13.7	13.0	16.8			15.30	2.299
Lab-8 (β)	11.4	10.5	9.2	11.9			10.75	1.185
^{210}Po Lab-2 (α)	15.7	17.7	16.6	17.7			16.92	.967
Lab-3 (α)	11.3	10.4	11.5	14.2			11.85	1.638
Lab-4 (α)	14.2	14.8	15.2	14.5			14.68	.427
Lab-5 (α)	11.5	12.6	13.0	12.6	11.8	11.8	12.33	.618
	13.0							
Lab-6 (α)	14.5	15.1	16.2	15.5			15.32	.714
Lab-8 (α)	11.9	12.4	12.6	10.1			11.75	1.139

Table B-7 – Analytical results, laboratory means and standard deviations for ^{232}Th-series isotopes in UTS-1

	Bq g^{-1}					Mean	S.D.
^{232}Th Lab-1 (α)	.514	.579	.551	.555		.550	.027
Lab-2 (α)	.510	.586				.548	.054
Lab-2 (γ, daught)	.570	.570				.570	
Lab-3 (α)	.58	.82	.61	.93		.735	.168
Lab-4 (α)	.810	.774	.614	.717		.729	.086
Lab-5 (α)	.66	.55	.65	.65		.626	.052
Lab-5 (NAA)	.61					.61	
Lab-8 (α)	.76	.70	.73	.68		.715	.031
^{228}Ra Lab-1 (β)	<1.	<1.	<1.	<1.			
Lab-3 (β)	.74	.92	.74	.63		.758	.120
Lab-4 (γ)	.736					.736	
Lab-5 (β)	.54	.53	.55	.52	.57	.542	.019
Lab-8 (β)	.88	.71	.69	.75		.758	.085
^{228}Th Lab-2 (α)	.6	.6	.6	.6		.6	
Lab-3 (α)	.64	.67	.67	.92		.725	.131
Lab-4 (α)	.85	.79	.62	.75		.752	.097
Lab-5 (α)	.65	.58	.65	.65		.632	.035
Lab-8 (α)	1.22	.78	.77	.66		.858	.248

Table B-8 – Analytical results, laboratory means and standard deviations for ^{232}Th-series isotopes in UTS-2

	Bq g^{-1}						Mean	S.D.
^{232}Th Lab-1 (NAA)	.693	.656	.680	.680			.677	.015
Lab-2 (α)	.76	.69					.725	.050
Lab-2 (γ, daught)	.688	.692					.690	.003
Lab-3 (α)	.57	.65	1.20	.83			.812	.260
Lab-4 (α)	1.10	.85	.98	.98			.976	.102
Lab-5 (α)	.56	.65	.85	.84	.77		.740	.121
Lab-5 (NAA)	.74						.74	
Lab-8 (α)	.91	.91	1.27	1.26			1.088	.205
^{228}Ra Lab-1 (β)	<1.	<1.	<1.	<1.			<1.	
Lab-3 (β)	1.07	1.11	1.26	1.15			1.148	.082
Lab-4 (γ)	1.26						1.26	
Lab-5 (β)	.91	.80	.69	.80	.80	.85	.842	.050
Lab-8 (β)	1.18	1.16	1.23	1.09			1.165	.058
^{228}Th Lab-1 (α)	.6	.6	.6	.6			.6	
Lab-3 (α)	.65	.81	1.27	1.18			1.028	.232
Lab-4 (α)	1.07	.86	1.03	1.00			.990	.091
Lab-5 (α)	.56	.68	.87	.79	.74		.728	.117
Lab-8 (α)	1.23	1.29	1.21	1.44			1.293	.104

Table B-9 – Analytical results, laboratory means and standard deviations for ^{232}Th-series isotopes in UTS-5

	Bq g^{-1}						Mean	S.D.
^{232}Th Lab-1 (NAA)	1.89	1.89	1.88	1.86			1.880	.014
Lab-2 (α)	1.97	1.90					1.935	.050
Lab-2 (γ, daught)	1.94	1.88					1.910	.042
Lab-3 (α)	2.07	2.40	1.92	1.85			2.060	.244
Lab-4 (α)	2.42	2.31	2.34	2.43			2.375	.059
Lab-5 (α)	1.69	1.72	1.93	1.71			1.762	.112
Lab-5 (NAA)	1.76						1.76	
Lab-8 (α)	1.82	1.46	2.01	1.35			1.660	.308
^{228}Ra Lab-1 (β)	1.0	2.0	1.0	1.0			1.25	.500
Lab-3 (β)	2.2	2.2	2.1	2.6			2.28	.222
Lab-4 (γ)	2.95						2.95	
Lab-5 (β)	1.70	1.59	1.66	1.59	1.66	1.88		
	1.59						1.667	.104
Lab-8 (β)	1.95	1.73	1.80	1.85			1.833	.092
^{228}Th Lab-1 (α)	1.8	1.8	1.8	1.8			1.80	
Lab-3 (α)	1.97	2.40	2.04	1.85			2.065	.237
Lab-4 (α)	2.55	2.40	2.47	2.55			2.492	.072
Lab-5 (α)	1.69	1.72	2.02	1.98			1.852	.171
Lab-8 (α)	1.61	1.53	1.84	1.87			1.712	.168

Table B-10 – Analytical results and laboratory mean values for ^{232}Th and ^{228}Th in UTS-3 and UTS-4 (Lab-3 only, alpha spectroscopy)

	Bq g^{-1}				Mean	S.D.
UTS-3 ^{232}Th	.15	.16	.22	.10	.16	.05
^{228}Th	.13	.16	.23	.13	.16	.04
UTS-4 ^{232}Th	.40	.56	.73	.48	.48	.07
^{228}Th	.22	.27	.19	.26	.23	.04

Table B-11 – Analytical results and laboratory mean values for ^{231}Pa (Lab-3 only, alpha spectroscopy)

	Bq g^{-1}				Mean	S.D.
UTS-1	.23	.23	.20	.18	.21	.03
UTS-2	.30	.37	.51	.31	.37	.11
UTS-3	.54	.78	.61	.85	.70	.15
UTS-4	2.2	2.3	2.1	2.8	2.4	.2

Table B-12 – Summary of radiochemical procedures (a, b)

Lab-1 Decompositions of repeated multiple acid digestion; HC1, HNO_3, HF, H_2SO_4. Residues fused with $LiBO_2$. Dissolved in 10% HNO_3/10% HC1.

Separations: (volumetric aliquots)

^{226}Ra	– (c), α-spectroscopy.
^{228}Ra	– (c), (no ^{133}Ba), β-counting.
^{232}Th, ^{228}Th	– two-stage IX, SX (TTA), extract concentrated and evaporated on a steel planchet α-spectroscopy, ^{232}Th also by NAA.

Lab-2 Decomposition by repeated multiple acid digestion; HNO_3, HF, $HClO_4$ (0.25 to 0.5 g).

Separations:

^{210}Pb	– (d) (1).
^{210}Po	– Ag deposition with ascorbic acid reductant (1), α-spectrometry.
^{230}Th	– Successive coprecipitations with lanthanum as hydroxide and fluoride, with Ba 'hold-back' carrier for Ra, final ppt. collected on 0.45 μm filter for α-spectrometry. ^{234}Th tracer by β-counting. ^{230}Th additions implied negligible self-absorption.

Lab-3 Decompositions: for Ra, Th: 2 g by KF and pyrosulphate fusion (2).
for Po, Pb: 1 g by repeated multiple digestions: HNO_3, HF, $HClO_4$.

Separations:

^{226}Ra	– (c), α-spectrometry.
^{228}Ra	– (e) (1, 3, 4, 5), ^{228}Ac β-counting.
^{230}Th, ^{232}Th, ^{228}Th	– supernate from (c) volumetrically diluted, and split into two equal portions, one spiked with ^{228}Th. Following SC (HDEHP) (5), thorium coprecipitated with cerous hydroxide, collected on 0.1 μm filter for α-spectrometry (6).
^{210}Pb	– (d) (1).
^{210}Po	– coprecipitation with PbS, dissolved in HC1, Ni deposition for α-spectrometry (7). ^{208}Po tracer used.

Lab-4 Decompositions by HF treatment followed by KF and pyrosulphate fusions of 1 g samples (8, 9).

Separations:

^{226}Ra	– (c), α-spectrometry.
^{232}Th, ^{228}Th, ^{230}Th	– coprecipitated with cerous hydroxide in presence of EDTA from supernate from (C) filtered onto 0.1 μm filter for α-spectrometry.
^{210}Po	– coprecipitated with tellurium in presence SO_2, $TiCl_3$, collected on 0.1 μm filter. (^{208}Po tracer used, α-spectrometry.)
^{228}Ra	– no separations, γ-spectrometry of ^{228}Ac on 40-75 g samples.
^{210}Pb	– indirectly, as supported ^{210}Po.

Table B-12 (Cont'd)

Lab-5 Decompositions of 1 g samples by repeated digestion with HF, H_2HSO_4 followed by pyrosulphate fusions. Radium samples dissolved in HNO_3, others in HC1.

Separations:

^{226}Ra	– (c) (1) (gross-alpha counting, with decay/ingrowth corrections), (also, gamma-spectrometry, 10 g).
^{228}Ra	– (e) (1, 5) (β-counting of ^{228}Ac).
^{210}Pb	– (d) (1) (β-counting of ^{210}Bi).
^{210}Po	– Ag deposition in presence of ascorbic acid and thioacetamide. (^{208}Po tracer used.)
^{232}Th, ^{228}Th, ^{230}Th	– a series of iron hydroxide and barium sulphate coprecipitation steps followed by SX (TTA), back extraction (2 M HNO_3), coprecipitation with cerous hydroxide with collection on a 0.1 μm filter of α-spectrometry. (^{234}Th tracer). (^{232}Th also by NAA).

Lab-6 Decompositions: for Ra by fusion with Rushing flux, $Na_2Co_3/K_2CO_2/Na_2B_4O_7$ (10). For Th, Pb by KF and pyrosulphate fusions (11). For Po by multiple acid digestion; HF, HNO_3, H_2SO_4, HC1 (1-2 g samples.)

Separations:

^{226}Ra	– ^{222}Rn emanated after 6 d. Storage of de-emanated solution in a sealed bubbler, ^{222}Rn and daughters measured by α-scintillation.
^{210}Pb	– (d).
^{210}Po	– Ni deposition from HC1 medium (^{208}Po tracer, α-spectrometry).
^{230}Th	– thorium in supernate from (c) coprecipitated with titanium hydroxide at pH 14 and filtered for alpha spectrometry.

Lab-7 Decompositions: for Ra, Th – repeated mixed acid digestion with HNO_2, HF, H_2SO_4, followed by pyrosulphate fusion and dissolution in HC1. For Pb – acid digestion with HNO_3, $HC1O_4$; residue treated with HF, H_2SO_4; dissolved in HC1. (All 1 g samples).

Separations:

^{226}Ra	– (c) (1), α-spectrometry.
^{210}Pb	– (d) (1), β-counting of ^{210}Bi, no recovery tracer.
^{230}Th	– coprecipitation with lanthanum as hydroxide and then fluoride followed by SX (TTA), back extraction (HNO_3) (1), coprecipitation with titanium hydroxide and filtration for alpha spectrometry.

Table B-12 (Cont'd)

Lab-6	Decompositions of 2 g samples by repeated acid digestion (HF, H_2SO_4) followed by pyrosulphate fusion and dissolution in 10% HC1.	
	Separations:	
	^{226}Ra	– (c).
	^{230}Th, ^{232}Th, ^{228}Th	– IX (cation, loaded pH 2-3, cations eluted with 12 M HC1, Th then eluted with 0.5 M oxalic acid) (13). Oxalate destroyed with HNO_3, SX (TTA), back-extracted into 2 M HNO_3 and Th isotopes electro-deposited (14) for α-spectrometry. (^{234}Th tracer used.)
	^{210}Pb	– IX eluent (above) passed through anion IX (to remove U) and Bi extracted by SX (DDTC), evaporated on planchet for β-counting 20 min and 1 d after extraction.
	^{228}Ra	– (e).
	^{210}Po	– Ag deposition, pH 0.3, 95°C, α-spectrometry, no tracer.

(a) Reports of contractors entitled 'Radiochemical determinations for tailings reference materials and references cited should be consulted for details (Table 1, Table 13).

(b) Abbreviations: IX, ion-exchange chromatography; SX, solvent extraction; TTA, theonyltrifluoroacetone; NAA, neutron activation analysis; HDEHP, bis(2-ethly-hexyl) phosphoric acid; DDTC, diethyldithiocarbanate; EDTA, ethlyenediamine-tetraacetic acid; DTPA, diethylenetriaminepentaacetic acid; AAS, atomic absorption spectrophotometry.

(c) Radium (and other radioisotope) are coprecipitated with lead sulphate; the precipitate is dissolved alkaline EDTA (or DTPA) and radium is selectively coprecipitated with barium sulphate which is collected on a membrane filter for α-spectrometry or gross alpha counting. Barium-133, added at the decomposition stage, is usually as a recovery tracer, employing gamma-ray spectrometry (e.g. (1)).

(d) Lead and bismuth carriers are added prior to decomposition; the dissolved sample is in 2M HC1. Bismuth SX (0.1% DDTC/$CHC1_3$), extracts evaporated, decomposed with HNO_3, and bismuth hydroxides precipitated (pH 8). The precipitate is dissolved in HC1, and BiOC1 (precipitated by dilution) is filtered for β-counting of ^{210}Bi after decay of other β-emitters. Correction is applied for decay of ^{210}Bi, and recovery is traced by AAS measurements of Bi carrier (1b).

(e) The Ba(Ra) SO_4 from (a) is redissolved in alkaline DTPA following a 3 d ingrowth of ^{228}Ac. Ba(Ra)SO_4 is removed by precipitation and ^{228}Ac is separated by SX (HDEHP/alkanes), back-extraction into 1M HNO_3 and coprecipitation with lanthanum oxalate which is filtered for β-counting (1, 5).

Table B-13 - References cited for radiochemical procedures

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1(b) Smithson, G., Fahri, M. and Petrow, M. "Radiochemical determination of lead-210 in environmental samples and samples resulting from uranium mining-milling operations"; ibid, Appendix B.

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Table B-14 – Calibration reference materials and recovery tracers used for radiochemical measurements

	SEM (uncertainty, %)* [Tracer]						
	^{226}Ra	^{210}Pb	^{210}Po	^{230}Th	^{232}Th	^{228}Th	^{228}Ra
Lab 1	NBS 4964-B (0.5) [^{133}Ba]	–	–	–	[thorium]	st. monazite (1.2)	[cf. ^{226}Ra]
Lab 2	–	AM RBZ (2.3) [Bi]	AM RBZ (2.3) [st. addn ^{210}Po]	IPL (6.) [^{234}Th]	–	–	–
Lab 3	NBS 4957 (1.8) [^{133}Ba]	AM RBZ (2.5) [Bi]	AM PMS (5) [^{208}Po int. std.]	AM RLZ (3.5) [^{228}Th int. std.]	AM TYS-1 (<5) [cf. ^{230}Th]	AM RLS (3.5) [cf. ^{230}Th]	AM TYS-1 (5) [cf. ^{226}Ra]
Lab 4	NBS 4957 (1.8) [^{133}Ba]	–	AM PMS (5) [^{208}Po int. std.]	[^{228}Th int. std.]	thorium nitrate [cf. ^{230}Th]	[cf. ^{230}Th]	'aged' ThO_2 [no sepn.]
Lab 5	NBS 4953-C (1.8) [^{133}Ba]	AM RBZ (2.3) [Bi]	AM RBZ (2.3) [^{208}Po]		AM TYS-1 (<5) [^{234}Th]		[cf. ^{226}Ra]
Lab 6	AM RAY-31 (5) [n.i.]	AM RBZ (1.9) [Bi]	AM PDZ (3.4) [^{208}Po]	USEPA 1838 3 (5.7) [^{234}Th]	–	–	–
Lab 7	NBS 4959 (1.3) [^{133}Ba]	AM RBZ (2.3) [n.i.]	–	CCRMP BL-5 (?) [n.i.]	–	–	–
Lab 8	AM RAY -31 (5) [^{133}Ba]	NEN NES-200F (5) [n.i.]	[n.i.]	NEN NES-316 (4.5)	[^{234}Th]		NEN NES-200F (5) [c.f. ^{226}Ra]

**Abbreviations*: AM, Amersham Corp.; IPL, Isotope Product Laboratories; NBS, National Bureau of Standards (U.S.); NEN, New England Nuclear Ltd.; USEPA, U.S. Environmental Protection Agency; n.i., not indicated.

Table B-15 – Results of analysis of variance, ^{238}U-series isotopes*

		^{230}Th	^{226}Ra	^{210}Pb	^{210}Po
UTS-1	Ratio	7.02	0.89	8.31 [0.5]	34.9
	$F_{.95}$	2.57	2.44	2.60 2.81	2.68
	f_b, f_w	6,21	7,23	6,20 5,17	5,21
UTS-2	Ratio	26.3	1.77	3.18 [0.5]	20.7
	$F_{.95}$	2.55	2.35	2.56	2.64
	f_b, f_w	6,22	7,29	5,28	5,23
UTS-3	Ratio	7.08	1.00	1.29	16.55
	$F_{.95}$	2.55	2.46	2.53	2.64
	f_b, f_w	6,22	7,22	6,23	5,23
UTS-4	Ratio	41.6 [5.55]	3.90	10.7	45.3
	$F_{.95}$	2.57 2.77	2.40	2.56	2.66
	f_b, f_w	6,21 5,18	7,25	6,21	5,22
UTS-5	Ratio	24.7	14.0	12.9	21.5
	$F_{.95}$	2.51	2.42	2.57	2.68
	f_b, f_w	6,24	7,24	6,21	5,21

**Ratio of 'between-set' to 'within-set' mean-square variance with degrees of freedom f_b and f_w, resp. (4).*
[], value after removal of probable outlier set.

Table B-16 – Results of analysis of variance, ^{232}Th-series isotopes*

		^{232}Th	^{228}Ra	^{228}Th
UTS-1	Ratio	2.92 [1.94]	7.32	2.34
	$F_{.95}$	2.61 3.18	3.71	3.05
	f_b, f_w	7,17 4,13	3,10	4,15
UTS-2	Ratio	3.14 [2.88]	33.9	17.8
	$F_{.95}$	2.58 3.11	3.59	3.01
	f_b, f_w	7,18 4,14	3,11	4,16
UTS-5	Ratio	6.12 [7.87]	13.52	16.4
	$F_{.95}$	2.61 3.18	3.06	3.05
	f_b, f_w	7,17 4,13	4,15	4,15

**Alpha spectrometry methodology only.*