

STATE OF NEVADA

Department of Conservation & Natural Resources

Jim Gibbons, Governor

Allen Biaggi, Director

DIVISION OF ENVIRONMENTAL PROTECTION

Leo M. Drozdoff, P.E., Administrator

February 6, 2009

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Re. **BMI Plant Sites and Common Areas Projects, Henderson, Nevada**
Guidance for Evaluating Radionuclide Data for the BMI Plant Sites and Common Areas Projects

Dear Sirs and Madam:

All of the parties listed above shall be referred to as “the Companies” for the purposes of this letter. Guidance for evaluating radionuclide data is provided in Attachment A. This guidance is a supplement to the secular equilibrium tool issued via electronic mail on January 22, 2009 and the secular equilibrium guidance document issued on February 6, 2009.

Please contact me with any questions (tel: 702-486-2850 x247; e-mail: brakvica@ndep.nv.gov).

Sincerely,

Brian A Rakvica, P.E.
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ATTACHMENT A

Introduction

Issues were raised in the latter part of 2007 when datasets of radioactivity in soil samples from several of the BMI Companies (hereinafter “the Companies”) continued to both pass and fail soil-based background comparisons for radionuclides in the same chain. This brought into question the appropriateness of some of the radionuclide data, since radionuclides in the same chain should obtain similar background comparison results under the assumption of secular equilibrium. The Nevada Division of Environmental Protection (NDEP) issued a letter to the Companies dated December 7, 2007 (BMI Plant Sites and Common Areas Projects, Henderson, Nevada: *Advisement Regarding Radionuclide Analysis for Uranium*) asking specific questions about radiochemical analysis methods for potentially affected projects and datasets. The Companies have provided responses, and all relevant issues within each correspondence are addressed as part of this report.

The goals of this guidance are to describe some of the chronology of how the issue arose and interactions and information shared with the Companies, evaluate analytical methods and data, and provide recommendations for recovering from historical issues that have caused apparent bias in the radionuclide data. There are three Companies involved that have submitted data to the NDEP thus far: Basic Remediation Company (BRC), Titanium Metals Corporation (TIMET) and TRONOX LLC (TRONOX) (collectively, also referred to as “the Companies” for the purposes of this letter). Several radionuclides from two radionuclide chains are of primary concern: The uranium (U)-238 chain (uranium chain) focusing on the long-lived radionuclides U-238, U-234, Thorium (Th)-230 and Ra-226; and, the thorium-232 chain (thorium chain) focusing on the long-lived radionuclides Th-232, Th-228 and Ra-228. These radionuclides are of interest because the projects require their data collection to support human health risk assessment. Other radionuclides, with the exception of U-235 are not included directly in these risk assessment. No evaluation of the U-235 decay chain data was performed since most radionuclides appear to be barely discernable from the minimum detectable concentration. Nevertheless, issues raised by the Companies pertaining to Polonium (Po)-210 and Lead (Pb)-210 are also discussed in this report.

Secular equilibrium (SE) exists when the quantity of a radioactive isotope remains constant because its production rate (due to the decay of a parent isotope) is equal to its decay rate. In theory, if secular equilibrium exists, the parent isotope activity should be equivalent to the activity of all daughter radionuclides. Pure secular equilibrium is not expected in environmental samples because of the effect of natural chemical and physical processes. For example, characteristics such as partitioning and solubility differ by element, and, for the entire uranium and thorium chains, radon is a gas that can escape the environmental system. In addition, differences in analytical methods could also cause minor effects or relative bias in the radionuclide data. However, approximate secular equilibrium is expected under background conditions. Natural abundance ratios of the uranium isotopes also offer a metric by which background radionuclide conditions can be evaluated. It should be noted that failure of secular equilibrium or natural abundance ratios implies contamination, whereas lack of failure does not

imply lack of contamination; rather, it implies lack of contamination or contamination that maintains the relevant proportions. Although natural abundance ratios could be used to evaluate the presence of radionuclides, it is easier to perform the evaluation using secular equilibrium because the activities of isotopes within a chain should be approximately equivalent.

This memorandum is divided into three main sections¹. The first section addresses some of the underlying historical radionuclide data assembled by BRC, TIMET and TRONOX. Of specific interest are the radiochemical analytical methods used in the different investigations. Background data sets are available from three investigations: the original 2005 BRC/TIMET background study; the 2008 supplemental BRC background study; and, the 2008 BRC deep background study. Site data sets are available from seven investigations: TRECO; TRONOX Parcels A/B and Parcels C/D/F/G; the BRC Utility Corridor; the BRC upgradient groundwater wells soil sampling; BRC's Parcel 4B; and the BRC northeast area wells soils investigation. The focus is the soil sampling and analysis that was performed for these 10 investigations. Exploratory data analyses are presented and secular equilibrium is evaluated using an equivalence testing procedure, which is described in NDEP's guidance *Statistical Methods for Secular Equilibrium: For Radionuclide Data from Soil Samples Collected at the BMI Complex and Common Areas in Henderson, Nevada* (Statistical Methods for Secular Equilibrium Guidance), document dated January, 2009. The second section addresses a TIMET technical memorandum concerning a methods comparison for estimating radium (Ra)-228. The third section addresses the concerns regarding polonium-210 and lead-210. The report concludes with recommendations on how questionable radionuclide activity data from these studies can be used to support background comparisons and risk assessment, and describes the radiochemical analytical methods that should be used for future investigations.

Evaluation of the Uranium and Thorium Radionuclide Chains

The December 2007 NDEP memorandum highlighted issues relating to radiochemical analytical methods used for isotopic uranium analysis. The primary issue at hand was whether laboratory preparation methods were performed using hydrofluoric acid (HF). The NDEP requested that the Companies identify datasets that were prepared using a non-HF procedure. The NDEP also requested the Companies propose a plan to rectify all affected datasets under the assumption that non-HF methods would yield low-bias radioactivities. In response to the NDEP request, BRC listed all affected datasets and proposed a plan to salvage those data that were compromised. These datasets included datasets associated with BRC investigations and TRONOX investigations. TIMET stated from a response to NDEP comments as recent as January 29, 2008 that isotopic uranium and thorium preparation used the same method employed for the 2005 BRC/TIMET Shallow Soils Background data. TIMET did however identify issues with the preparation and analytical methods for Ra-228 and Pb-210, which are discussed later in this report. The results of exploratory and statistical analyses are presented below that shed light on the identified datasets and evaluate the proposed correction measure proposed by BRC and TRONOX. Datasets for TIMET were not specifically evaluated as it was believed that this would not add value to the development of this guidance document.

¹ All references to the Henderson site datasets included in this analysis are provided at the end of this report in a section titled "References for the Henderson Site datasets".

Exploratory data analysis (EDA) performed on the BRC and TRONOX data includes box plots, correlation matrices, and summary statistics tables for the uranium and thorium radionuclide chains. These analyses were performed to qualitatively assess if the radionuclide data exhibit secular equilibrium. The EDA is followed by statistical analysis that involves equivalence testing for secular equilibrium, as described in NDEP's Statistical Methods for Secular Equilibrium Guidance (January 2009), and recommendations are made regarding recovery of historical data and radiochemical analysis for future studies.

Exploratory Data Analysis

Several of the soil datasets identified by BRC that were affected by the preparatory methods exhibited noticeable differences in the box plots and summary statistics between radionuclides within each chain (see Appendices A and B below). Some of the most noticeable differences between radionuclides (both thorium and uranium chains) were identified for datasets flagged by BRC as "requiring correction". These datasets include: BRC Parcel 4B, TRONOX Parcels C/D/F/G, BRC northeast area wells, and BRC upgradient groundwater wells. Comparison between radionuclides and comparison with the background data sets are helpful when interpreting the EDA.

Although there are some small differences in the box plots and summary statistics for the three background datasets, they appear to exhibit approximate secular equilibrium. They also show radioactivities that are a little greater than 1 pCi/g on average for radionuclides in the uranium chain, with high values around 3 pCi/g. Radioactivities in the thorium chain are a little greater than 1.5 pCi/g on average, with high values around 3 pCi/g again. Of further interest is that the correlations appear to be high within the uranium chain, but correlations with Ra-228 appear very low in the thorium chain (see Appendix C below). These are useful references for evaluation of the seven site datasets.

The BRC Parcel 4B data show clear differences in the uranium chain, with Ra-226 showing much higher activities than Th-230, which in turn are much higher than those for the uranium isotopes. Differences between Ra-228 and the thorium isotopes are also clear in the thorium chain (see Appendices A and B below). For both chains, the Ra results appear to be roughly in line with background. Hence, the uranium and thorium data appear to be too low.

The TRONOX Parcels A/B data exhibit noticeable differences in both radionuclide chains, however these data were identified in the BRC memorandum as not requiring further corrections because they were corrected for the No Further Action Determination (NFAD) for these parcels (see Appendices A and B below). The uranium chain box plot shows that the Ra-226 data are similar to background, and the Th-230 data are slightly higher than the Ra-226 data. However, the radioactivities for the uranium isotopes appear to be too low. Results for the thorium chain appear to be reasonable. Of interest again is that the correlations are low with Ra-228. The lack of correlation with Ra-228 is a recurring theme.

The BRC upgradient groundwater wells and the BRC northeast area wells data exhibit the same general pattern as the TRONOX Parcels A/B data. However, the correlations with Ra-228 are high for these two datasets, and are the exceptions in this regard across the 10 datasets evaluated.

The TRONOX Parcels C/D/F/G and the Utility Corridor data show similar patterns with respect to the uranium and thorium chains, although there is some greater variability in the TRONOX Parcels C/D/F/G data. The correlations with Ra-228 are again quite low.

The TRECO study was performed a few years earlier than the other site studies reported here. The uranium chain data appear to be in line with background with the exception of the Ra-226 data, which appear to be greater than the data for the other isotopes. The Ra-226 data also appear to be greater than background. The data imply either an analytical issue, or low levels of Ra-226 contamination at TRECO. For the thorium chain, the data appear to be similar to background and they are in approximate secular equilibrium. However, the mean for Ra-228 is lower than for the thorium isotopes. The correlations with Ra-228 again appear to be low.

The EDA and correlations suggest some potential issues with the radionuclide data. When the radioactivities are too low, the implication is an analytical issue, which has been traced back to the preparation method for uranium, and possibly for thorium, for some of the investigations. If the radionuclides are in secular equilibrium, then their correlations should be expected to be high. Consequently, the lack of correlation with Ra-228 is also of concern. Correlations in the uranium chain are generally high, but there are exceptions. For example, the correlations with Ra-226 at BRC Parcel 4B are negative, which further brings into question the analytical methods for that investigation. The correlations with Ra-226 at TRECO are also low.

Equivalence Test for Secular Equilibrium

The EDA involves comparison of data in the box plots and summary statistics that does not address the inherent correlation if secular equilibrium holds. That is, distributions might appear to be similar, but lack of correlation is also a concern. Conversely, a strong correlation does not imply similar results for the radionuclides. For example, the correlations in the uranium chain for the BRC upgradient groundwater wells soil data are strong, but there are clear differences between the uranium isotopic activities and those of radium-226 and thorium-230. In other cases where differences occur, the correlations are also low. The comparison issues are, apparently, complex. To further the evaluation, equivalence tests are presented to evaluate secular equilibrium. Equivalence testing, unlike standard classical significance testing, evaluates whether means are approximately equal, as opposed to exactly equal. The equivalence testing approach compares mean radioactivities while accounting for the correlation in the data. The approach is described in NDEP's Statistical Methods for Secular Equilibrium Guidance (January 2009).

Statistical equivalence testing essentially involves reversing the standard null and alternative hypotheses used in analysis of variance (ANOVA), and, in the process, allowing for non-point valued null hypothesis statements. Equivalence testing allows some flexibility in how approximate secular equilibrium is defined. The hypotheses allow a family of possible options, instead of the point null hypothesis that is common in classical statistics, by specifying that the

mean radioactivities can be close to the same as opposed to exactly equal. The result of equivalence testing for secular equilibrium will either indicate that the radionuclides are in approximate secular equilibrium (the alternative hypothesis), or that they are not (the null hypothesis). If the radionuclide data do not exhibit secular equilibrium, then there is some indication of radionuclide specific contamination. If the radionuclide data exhibit secular equilibrium, then either the data are similar to background, or there is more general contamination for all radionuclides in the decay chain.

The equivalence testing approach involves establishing an allowable difference between the mean activities for the radionuclides in the same decay chain. Specification of this difference is not necessarily straightforward. In this case, however, it seems reasonable to assume approximate secular equilibrium for the background data. Equivalence tests were performed on the background data for several possible allowable differences. The equivalence tests start to fail when the allowable difference is much less than 10%, in which case a difference of 10% was used to test the site data.

The results of the equivalence testing are presented in Table 1 (uranium chain) and Table 2 (thorium chain). Several sites did not meet the conditions of secular equilibrium (SE) for the uranium chain. These are TRECO, TRONOX Parcels A/B, the BRC upgradient groundwater wells, and the BRC northeast area wells. In BRC's response to a NDEP memorandum dated January 10, 2008, many of these datasets were flagged as requiring correction (with the exception of TRECO). The only site for which the conditions of secular equilibrium were not met was BRC Parcel 4B. Although the correlations with Ra-228 are often very low, the means are sufficiently close that the hypothesis of secular equilibrium is supported using the equivalence testing approach.

Table 1. Equivalence testing results for the uranium chain.

Site	Delta	p-value	Secular Equilibrium	Mean Proportion			
				Ra-226	Th-230	U-233/234	U-238
2005 BRC/TIMET Background	0.1	0.00	Yes	0.2401	0.2720	0.2448	0.2431
2008 Supplemental Background	0.1	0.03	Yes	0.2114	0.2934	0.2716	0.2236
2008 Deep Background	0.1	0.00	Yes	0.2430	0.2562	0.2569	0.2438
TRECO	0.1	0.50	No	0.3168	0.1925	0.1956	0.2951
Tronox Parcels A/B	0.1	0.50	No	0.3367	0.3799	0.1705	0.1128
Tronox Parcels C/D/F/G	0.1	0.00	Yes	0.2530	0.2159	0.2360	0.2951
Utility Corridor	0.1	0.00	Yes	0.2494	0.2585	0.2709	0.2211
Upgradient Groundwater Wells	0.1	0.50	No	0.2906	0.4122	0.1634	0.1338
BRC Parcel 4B	0.1	0.50	No	0.5249	0.2586	0.1145	0.1021
Northeast Area Wells	0.1	0.50	No	0.3447	0.3058	0.1863	0.1632

Results highlighted in yellow indicate that the uranium chain is not in secular equilibrium. Note that p-values reported as 0.50 are greater than or equal to 0.50.

Table 2. Equivalence testing results for the thorium chain

Site	Delta	p-value	Secular Equilibrium	Mean Proportion		
				Ra-228	Th-228	Th-232
2005 BRC/TIMET Background	0.1	0.00	Yes	0.3599	0.3270	0.3130
2008 Supplemental Background	0.1	0.00	Yes	0.3143	0.3647	0.3210
2008 Deep Background	0.1	0.00	Yes	0.3117	0.3586	0.3297
TRECO	0.1	0.00	Yes	0.3571	0.3406	0.3023
Tronox Parcels A/B	0.1	0.00	Yes	0.3786	0.3191	0.3022
Tronox Parcels C/D/F/G	0.1	0.00	Yes	0.3564	0.3324	0.3113
Utility Corridor	0.1	0.00	Yes	0.3507	0.3615	0.2878
Upgradient Groundwater Wells	0.1	0.00	Yes	0.3440	0.3375	0.3185
BRC Parcel 4B	0.1	0.50	No	0.4616	0.2671	0.2713
Northeast Area Wells	0.1	0.00	Yes	0.3291	0.3615	0.3095

Results highlighted in yellow indicate that the Th-232 chain is not in secular equilibrium. Note that p-values reported as 0.50 are greater than or equal to 0.50.

Preparation and Analysis Methods

The results of the secular equilibrium tests confirm some of the findings in the EDA and correlation analyses. Differences occur in the data for each radionuclide in the uranium chain for some sites, but the issue appears to be low radioactivities, implying an issue with the radiochemical analysis. However, secular equilibrium is observed in the thorium chain (with the exception of BRC Parcel 4B), despite the lack of correlation with Ra-228 in many of the datasets. After some investigation, the main issue appears to be associated with the preparation method used for the uranium and thorium analyses.

The methods and analyses used for isotopic uranium and thorium analysis for the sites that are addressed as part of this memorandum are presented in Table 3. There is some clear relationship between methods used and the statistical analysis results presented above. For example, the comparatively low uranium radioactivities correspond to investigations that did not use HF acid in the sample preparation (prep) step for dissolution of the sample. Results of the thorium analysis for BRC Parcel 4B might be a consequence of a similar issue. The data are compelling, but there is no other evidence to support the apparently low thorium activities at this site.

There are two reasons why it is recommended that all future isotopic uranium and thorium analysis for soils/sediments/solid samples should be digested using HF for total dissolution with subsequent analysis by alpha spectroscopy (spec). The first is that this is how the background data have been analyzed, and comparison of site and background data require comparability between datasets. The second is that based on the statistical analysis presented, it appears that this approach will provide the most reliable data for these radionuclides. This recommendation is consistent with how GEL and STL-Saint Louis have performed analysis for the thorium and uranium isotopes for the sampling events listed in Table 3, and is also consistent with how STL-Richland performed these analyses for the 2008 BRC deep soils background analysis.

Table 3. Radionuclide Methods.

Event	Pass U SE?	Pass Th SE?	Laboratory and Date	U preparation and analysis methods	Th preparation and analysis methods	Ra-226 preparation and analysis methods	Ra-228 preparation and analysis methods
2005 BRC/TIMET Background*	Y	Y	STL-SL, 2005	HF, alpha spec.	HF, alpha spec.	Prep acids unknown, Alpha spec. GFPC 9315.	Prep acids unknown, Beta spec, 9320.
2008 Supplemental Background	Y	Y	GEL, April 2008	HF, alpha spec.	HF, alpha spec.	Prep acids unknown, 903.1 Lucas cell alpha.	Prep acids unknown, 904.0 beta.
2008 Deep Background	Y	Y	STL-RICH, 2008	HF, alpha spec.	HF, alpha spec.	non-HF acids, 903.1, alpha scintillation counting.	non-HF acids, 904.0, GPC beta
TRECO	N	Y	STL-SL, 2005	Likely HF, alpha spec.	Likely HF, alpha spec.	Prep acids unknown, Alpha spec. GFPC 9315.	Prep acids unknown, Beta spec, 9320.
Tronox Parcels A/B (also #47)	N	Y	STL-RICH, 2007	Non HF, alpha spec.	HF**, alpha spec	gamma (soils)	gamma
Tronox Parcels C/D/F/G	Y	Y	STL-RICH, 2007	Non HF, alpha spec.	HF**, alpha spec	gamma	gamma
Utility Corridor (DVSR #50)	Y	Y	GEL, April 2008	HF, alpha spec.	HF, alpha spec.	Prep acids unknown, 903.1 Lucas cell alpha.	Prep acids unknown, 904.0 beta.
Upgradient Groundwater Wells (#47)	N	Y	STL-RICH, 2007	Non HF, alpha spec.	HF**, alpha spec	gamma	gamma
BRC Parcel 4B (#43)	N	N	STL-RICH, 2007	Non HF, alp ha spec.	HF**, alpha spec	gamma	gamma
Northeast Area Wells (#46)	N	N	STL-RICH, 2007	Non HF, alpha spec.	HF**, alpha spec	gamma	gamma

* Ra-226 and Ra-228 were re-analyzed at STL-Richland due to anomalies using isotopic barium carrier using the digestions prepared at STL-SL.

** Per email from Erika Jordan (Richland) all thorium used HF, uranium non-HF prior to 2008 Deep Background investigation. STL-ST: Severn Trent Laboratories, St. Louis. STL-RICH: Severn Trent Laboratories, Richland.

The issues regarding radium are less clear. Radium results often seem reasonable. However, a lack of correlation in some cases is of concern. For radium-226, correlations are highest at the BRC upgradient groundwater wells and the BRC northeast area wells sites, but neither of these sites demonstrates approximate secular equilibrium because of issues with the uranium analysis. Correlations are also quite high in the three background datasets and the BRC utility corridor data, all of which involves alpha spectroscopy (spec) analysis following HF acid preparation. Although there is not much evidence of analytical issues with the gamma spectroscopy method for radium-226, the main reason for using alpha spectroscopy is that this is the method used for the background data, and comparability of data is important for background comparisons.

The same applies to the radium-228 analysis; that is, beta spectroscopy should be used for site investigations because this is the method that was used for the background data. However, there is some evidence in the radium-228 data, based on the correlation analysis, for the BRC upgradient groundwater wells and the BRC northeast area wells sites that the gamma spec method outperforms the beta emissions methods. The lack of correlation could also be related to lack of sensitivity of the methods at the radioactivity levels being reported.

For the BRC 2008 deep background data the preparation method for both radium isotopes involved non-HF acids, in which case underestimation of the radium data might be expected. The results seem reasonable, however. A possible explanation is that radium is more soluble than thorium and radium, and HF acid is not necessary to obtain reliable data. Further discussion of radium-228 analysis is presented in the next section in response to TIMET's side-by-side study of gamma and beta spectroscopy analysis for this isotope.

Based on the observations made, and the analytical methods that were used for the background data, it is recommended that soils/sediments/solid being analyzed for Ra-226 should use alpha spectroscopy consistent with EPA methods 903.0/903.1 and 9315. It is recommended that isotopically labeled barium be used as the tracer. For Ra-228, soils/sediments/solid samples should be analyzed using beta spectroscopy consistent with EPA methods 904.0 and 9320. It is also recommended that isotopically labeled barium be used as the tracer.

Evaluating BRC's proposed correction approach and recommended decision logic

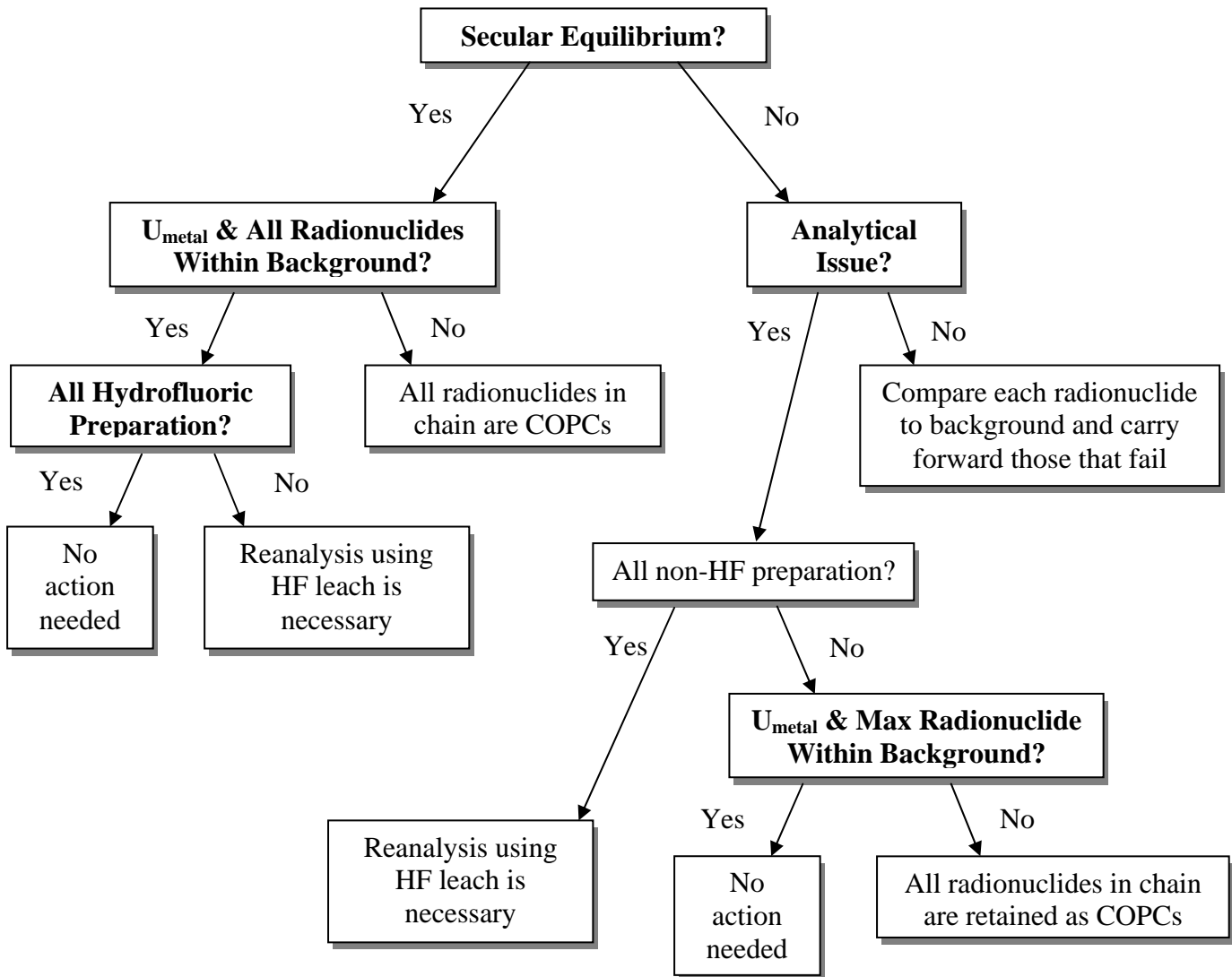
In BRC's response to the NDEP memorandum dated January 10, 2008, BRC proposed a correction factor approach in an attempt to salvage existing data sets that were affected by differences in preparatory methods. BRC constructed a dataset of 14 randomly chosen samples from the BRC 2008 deep soil background dataset and five randomly chosen samples from the TRONOX Parcels A/B dataset that were digested using the HF procedure and then reanalyzed. A ratio was then calculated for each sample by taking the HF acid reanalysis result and dividing it by the initial non-HF result for U-238, U-235/236, and U-233/234. An average correction ratio was then calculated for each nuclide. The correction procedure is then accomplished by multiplying the existing U-238 and U-233/234 activities analyzed using non-HF acid dissolution methods by the nuclide-specific average ratio.

Based on the statistical analyses presented above, the correction factor approach is likely to provide unreliable and unsupported results. The correction factor approach can only be applied if the data to which it is applied exhibits the same problem as the data on which the correction factor is based. The

difference in data for the site datasets implies this is unlikely to be the case. For example, the mean uranium activity at the BRC Parcel 4B site is about 0.2 pCi/g, whereas at the BRC upgradient groundwater wells site, the mean is about 0.6 pCi/g. Although there are problems with the data, a single correction factor approach seems unreasonable. NDEP's recommended approach is presented in Figure 1. This flowchart describes a decision framework that is applicable to all metallic uranium and radionuclide datasets that have been collected to date.

If secular equilibrium is exhibited in the isotope chains, then background comparisons should be performed to confirm if all the radionuclides in a decay chain are similar to background. If they are greater than background, then all the radionuclides would be carried forward in a risk assessment. If they are not greater than background and HF acid dissolution was used, then no further action is needed. If HF acid dissolution was not used, however, then reanalysis is necessary because all the radionuclide activities are probably underestimated.

If secular equilibrium is not exhibited, but there are no analytical issues (e.g., use of non-NDEP-approved analytical methods, or non-HF acid dissolution for uranium and thorium), then background comparisons can be performed for each radionuclide separately and for uranium as a metal. If there are analytical issues for all the radionuclides then reanalysis is necessary. If the analytical issues apply only to some of the radionuclides (such as uranium in the case of several of the datasets studied in this report, and thorium in BRC Parcel 4B), then the approach that NDEP will support for the historical data is to perform background comparisons with metallic uranium concentrations (if such data were collected at the site), and with the radionuclide for which the analytical methods are reasonable (usually radium-226 and radium-228).



COPCs indicates “chemicals of potential concern”.
 U_{metal} denotes metallic uranium.

Figure 1. Flowchart describing the decision framework for radionuclide historical dataset usability for Sites within the BMI Complex and Common Areas, Henderson, NV.

Method comparison of radium-228 in soils (TIMET)

TIMET responded to an NDEP comment dated January 11, 2008 to identify all datasets that are not comparable. Specific to this section of the memorandum, TIMET identified differences in preparation and analytical methods for soil samples for Ra-228. To address this issue, a TIMET memorandum dated May 9, 2008 outlined method comparisons of gamma spectroscopy (Gamma Spec) to gas flow proportional counting (GFPC) for estimating Ra-228. The purpose of the TIMET memorandum was to provide a basis for using gamma spec Ra-228 data to support background comparisons, although it was clearly indicated that this approach had not previously been approved by the NDEP.

There are several issues brought to light by this TIMET memorandum. TIMET states that back quantitation of Ra-228 from parent radionuclide (Th-232) should not be performed because of issues of comparability between the TIMET Hydrogeologic Investigation and the TIMET Vertical Delineation Investigation data, and the BRC/TIMET shallow soil background data. Data was not presented to support these statements.

Instead, in order to use Ra-228 data from non-NDEP-approved gamma spectroscopy techniques, TIMET proposed using samples from four boring locations that were analyzed by both gamma spec and GFPC (the NDEP approved method) to predict Ra-228 activity based on the gamma spec results. This would potentially allow those data analyzed by gamma spec to be used in future background comparisons at the site.

Several concerns regarding this approach are as follows:

Regression equation

The regression equation (see Figure 2) is surprising perhaps in that the intercept is significant, implying that a value of zero from gamma spec would not predict a value near zero for GFPC. This is not necessarily a problem, provided the regression model is used only within the range of the experimental data. However, the positive intercept and the slope of about 1/2 demonstrate that the model under-predicts GFPC results at high gamma spec values, and over-predicts at low gamma spec values. There is some cause for concern because this implies that the predicted distribution will be tighter than the input gamma spec distribution (see below).

Range of the data

Regression analyses should only be used within the range of the available data. Extrapolation is rarely supported. The range of the gamma spec data is from a minimum of 0.4 pCi/g to a maximum of 1.9 pCi/g. The range of the GFPC data is from a minimum of 1.0 pCi/g to a maximum of 2.2 pCi/g. In both cases, this is a much tighter range than has been observed in the background data and in data from other BMI sites. The range of data for this study needs to be increased for potential use of the regression equation to predict GFPC results.

The removal of ‘outlying’ data

TIMET used three statistical criteria to evaluate whether or not “outliers” or “influential points” existed in the data in order to improve the fit of their ordinary least squares model. These criteria are studentized residuals, hat matrix diagonals, and Cook’s D influence. From these three criteria, TIMET identified one residual as an outlier and two data points as influential. The outlier was the only point removed before TIMET revised the model. It is not clear that it is appropriate to remove an outlier without further justification simply to support an improved statistical model that is based on statistical assumptions that might not hold. Also, the difference between the two models is not sufficient to justify preference of the model without the outlier, and the regression lines are not very different. The small difference is probably because the outlier is not far outside the criteria used for its identification. Also, with 33 data points, identification of one outlier is not surprising. The unadjusted model should be used.

It is also not clear why a discussion of methods for identifying influential values is presented, when the TIMET memorandum does not include any regression analysis without these values.

Artificial tightening of post-hoc GFPC values, how will standard deviation / variance in prediction be addressed?

The issue here involves the fact that the original GFPC values in this data set had a standard deviation of 0.32 pCi/g where the gamma corrected GPFC predicted values have a standard deviation of about 0.16 pCi/g, or half that of the original data. This means that the confidence intervals constructed around these data will be much tighter and could have an effect on distributional background comparison tests, given the dependence of the distributional tests on the variance of the underlying data sets.

Heteroscedacity in variance around prediction line

This is likely a minor issue relative to the aforementioned, but there does appear to be heteroscedacity in the variance (i.e., different variances) around the prediction line as shown in Figure 2. Normally, this issue can be addressed by utilizing some form of a generalized linear model that accounts for the lack homogeneity in the residuals.

Variability between boring sites

There is some concern about the boring site variability. Figure 2 plots Ra-228 values from GFPC against those from gamma spec and clearly shows that grouping is occurring with respect to the boring site variable. Borings TMSB-131 and TMSB-135 are nearly always under-predicted while borings TMSB-132 and TMSB-133 are nearly always over-predicted. If all four borings can be assumed to be representative of the site then this is not a concern. The model accurately captures the “mean” behavior of the borings, however it cannot be applied to any particular boring and thus inferences should not be made about particular locations with this prediction model.

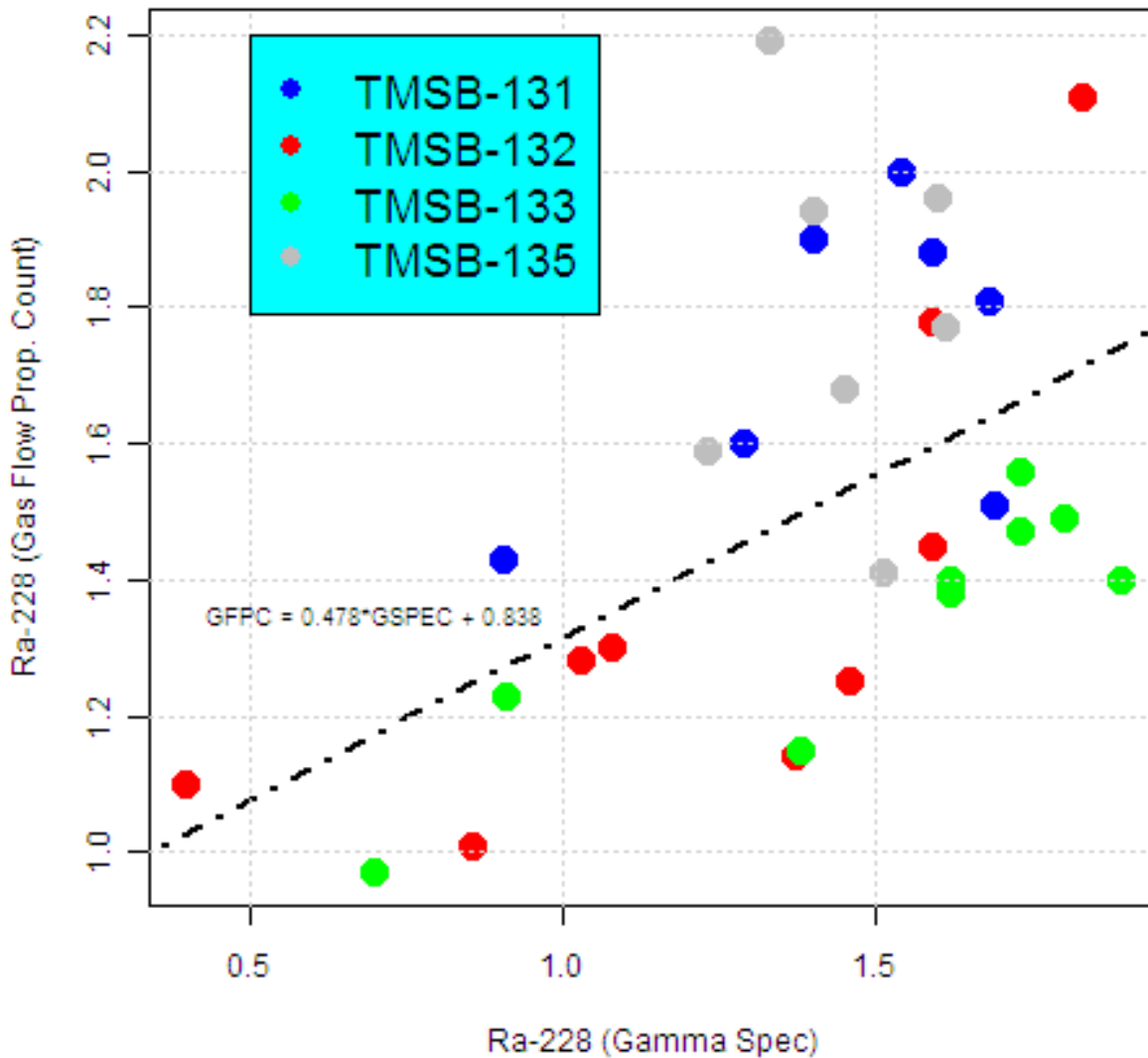
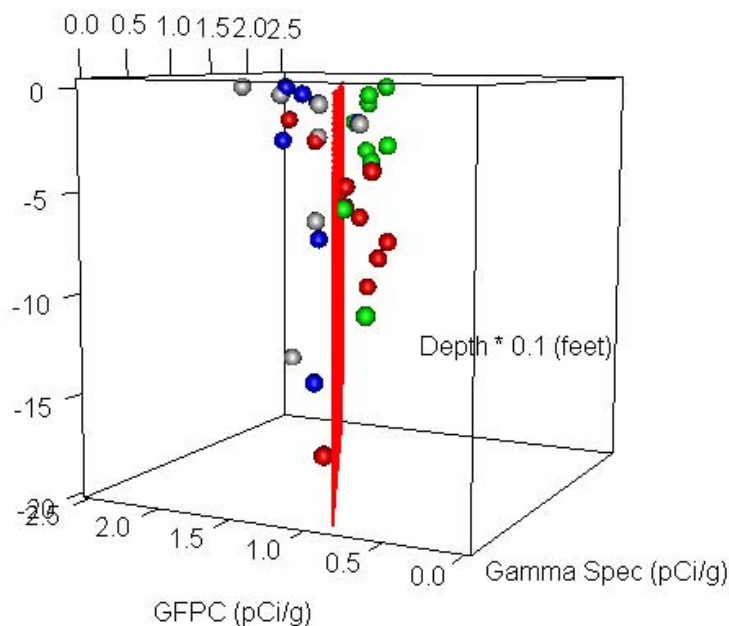


Figure 2: Radium-228 data from GFPC and Gamma Spec analysis (including prediction line)

Seemingly no relationship to depth

Diagnostically, from Figure 3, it appears that there is little relationship of prediction ability with depth of sample. A “side” shot, viewing down the prediction line (projected onto depth) shows that there is little deviation away from the prediction line as a function of depth. Therefore, these data do not support the inclusion of depth as part of the prediction model.



Color points share the same legend as presented in Figure 2.

Figure 3. Radium-228 data from GFPC and Gamma Spec analysis (with the regression line projected onto depth).

TIMET Lead-210 & Polonium-210 issues

TIMET proposed to conduct statistical correlations of results within the uranium decay chain to evaluate secular equilibrium for the analytical methods for Pb-210 and Po-210 (see TIMET's response to NDEP comments dated January 29, 2008). No further information has been provided. The most recent correspondence between TIMET and the NDEP dated April 11, 2008, indicates that TIMET has not yet completely resolved the Pb-210 and Po-210 analytical methods comparability issue, therefore we cannot comment further. If TIMET has conducted this analysis or have collected relevant data, then NDEP can perform a review. Otherwise, in light of the focus of human health risk assessment for the BMI sites on uranium, thorium and radium isotopes only, there is no need to pursue this issue further.

Summary

The path forward for radionuclide analysis seems clear based on the analysis presented in this report. Uranium and thorium isotopic analysis should be performed using alpha spectroscopy following HF acid dissolution. This approach is clearly more reliable than alternative approaches for these two elements, and is consistent with how the background data were obtained.

To resolve analytical issues with past data, BRC proposed a “correction factor” approach. Datasets flagged as potentially impacted by the analytical methods used for uranium and thorium were both qualitatively and quantitatively assessed to more comprehensively evaluate this proposed solution. The finding is that the proposed corrective factor approach should not be used. The side-by-side study that is used as the basis for the correction factor approach involved analysis of 19 samples for uranium isotopes. Although a simple correction factor approach was devised, the effect of method differences appears to be more complicated. Reported radioactivity for the uranium isotopes varies considerably when a non-HF acid dissolution was used. Possible explanations are the type of acid used and the amount of acid used for dissolution. Regardless, the correction factor estimated from the 19-sample study cannot be applied reliably to all affected datasets. In addition, correction factors were not developed for the thorium chain for BRC Parcel 4B and the BRC northeast area wells datasets, both of which failed the statistical test for secular equilibrium. An approach to resolving historical datasets is presented in Figure 1. NDEP requires that this approach be followed for historical data sets that are affected by analytical method issues. The approach basically allows the datasets to be evaluated (compared to background) based on uranium as a metal, and, usually, the radium isotopes. This is because the analytical problems are usually associated with the uranium and thorium analytical methods, whereas, the radium data, despite some analytical issues, appear to be comparatively reliable. NDEP also requires that appropriate methods as described in Table 4 are used for future investigations.

For the radium isotopes the situation is not as clear. It appears that Ra-226 analysis by alpha spectroscopy is marginally more reliable than analysis by gamma spectroscopy. The inter-isotope correlations within the uranium decay chain when alpha spectroscopy is used are often stronger than those when gamma spectroscopy is used. A more compelling argument to use alpha spectroscopy for Ra-226 is comparability with the background data. It should be noted, however, that HF acid dissolution was not used for the Ra-226 analyses in the background investigations. The Ra-226 results in background nevertheless seem reasonable (for example, they match results for other isotopes in the uranium chain). A possible explanation is that radium is more soluble than thorium and uranium, or that it is not so tightly bound in the soil matrix, so that a weaker acid dissolution is sufficient. It is not possible to draw firm conclusions in this regard without further information. For example, this could be achieved through a side-by-side study in which dissolution method is the variable of interest, including complete understanding of the acids used in the radiochemical analysis for radium. For radium-228 the situation is more difficult. The gamma spectroscopy results for the five sites included in this report seem reasonable, and, in two instances (BRC upgradient groundwater wells and BRC northeast area wells soils investigations) provide some of the highest correlations with the thorium isotopes from the thorium chain. However, the correlations are low in the other eight investigations presented in this report. In addition, the side-by-side study performed by TIMET does not provide a compelling argument for using gamma spectroscopy analysis for radium-228. The regression between the gamma spectroscopy results and the GFPC method does not provide a very good fit to the data, and the range of the data is smaller than the range of the background data, further reducing the effectiveness of the regression model for prediction

from gamma spectroscopy data. The overriding issue again is that the background data were collected using beta spectroscopy, in which case this analytical method should also be applied to the site investigations.

TIMET's side-by-side study for radium-228 analysis leads to a regression equation that relates gamma spectroscopy data to beta spectroscopy data. The regression model is not a very good fit to the data. The purpose of the investigation was to determine if beta spectroscopy data could be predicted from gamma spectroscopy data for radium-228. An implicit assumption was that the beta spectroscopy data are reliable. However, this assumption is not borne out by the analysis of the data from the three background and seven site investigations. The regression analysis and lack of correlation with radum-228 in many of the datasets might be suggestive of a sensitivity issue with the beta spectroscopy method. However, insufficient information is available to test this hypothesis. Also, the regression equation proposed is limited by the underlying data. The range of the radium-228 data in the side-by-side study is small compared to the range of the background and site investigations data. Extrapolation of regression equations is often difficult to defend. The regression proposed is not adequate for correcting existing gamma spectroscopy data without first addressing issues associated with the range of the data.

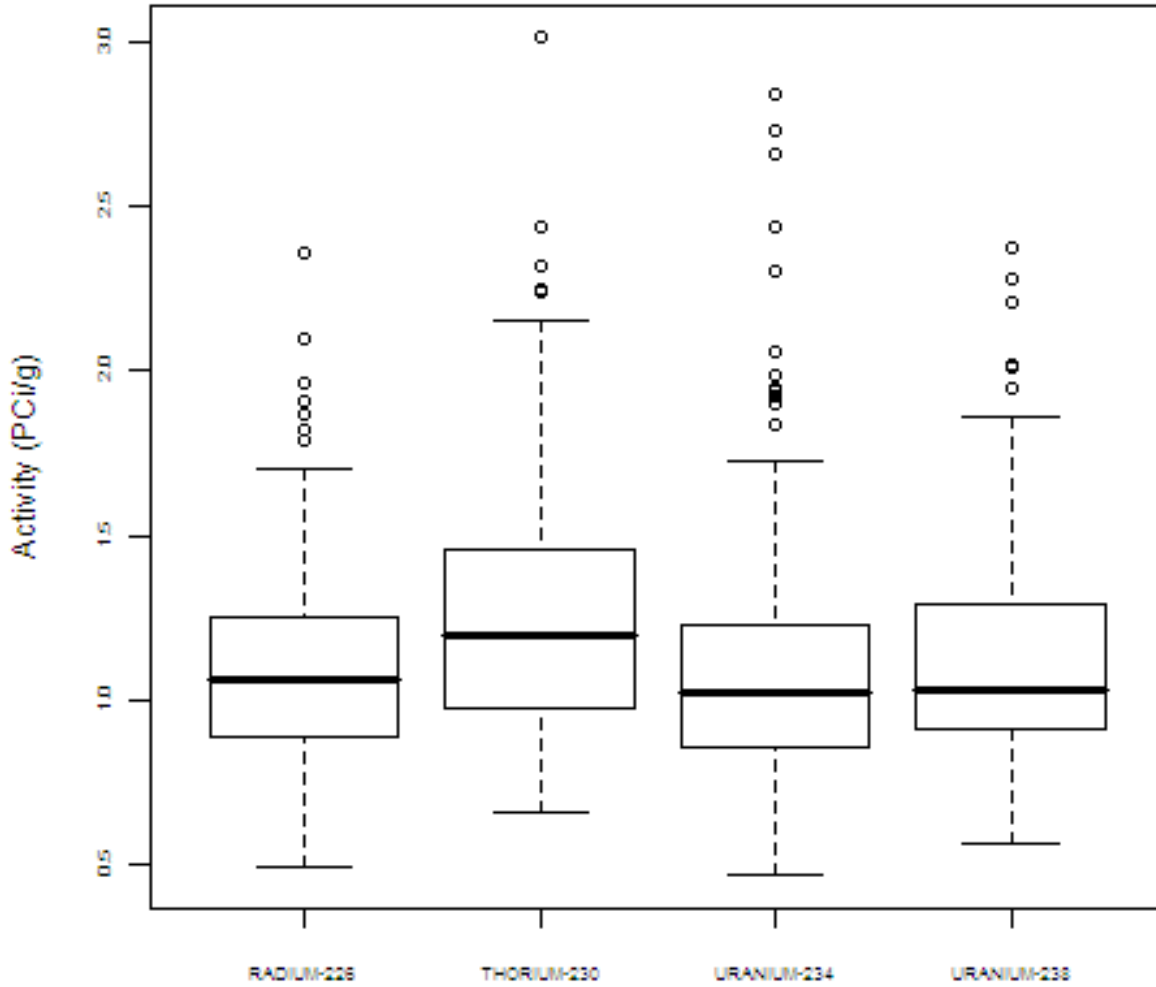
A full understanding of the analytical issues is not possible without recourse to some further information. Side-by-side studies across a greater range of radioactivities are needed to better form regression models and correlations between results. In addition, a study involving standards or performance evaluation samples would resolve many issues regarding the reliability of the analytical methods. Such a study should be performed blind to the laboratories involved. It also appears as though there are some sensitivity issues, at least for the radium-228 analytical methods. One issue with sensitivity that is always difficult is the role that ambient background subtractions play in the reported values. Ambient background data that are used in reporting data should also be reported and captured in the Companies databases. The following analytical methods are recommended for future site investigations:

Table 4: Recommended Radiochemical Analytical Methods

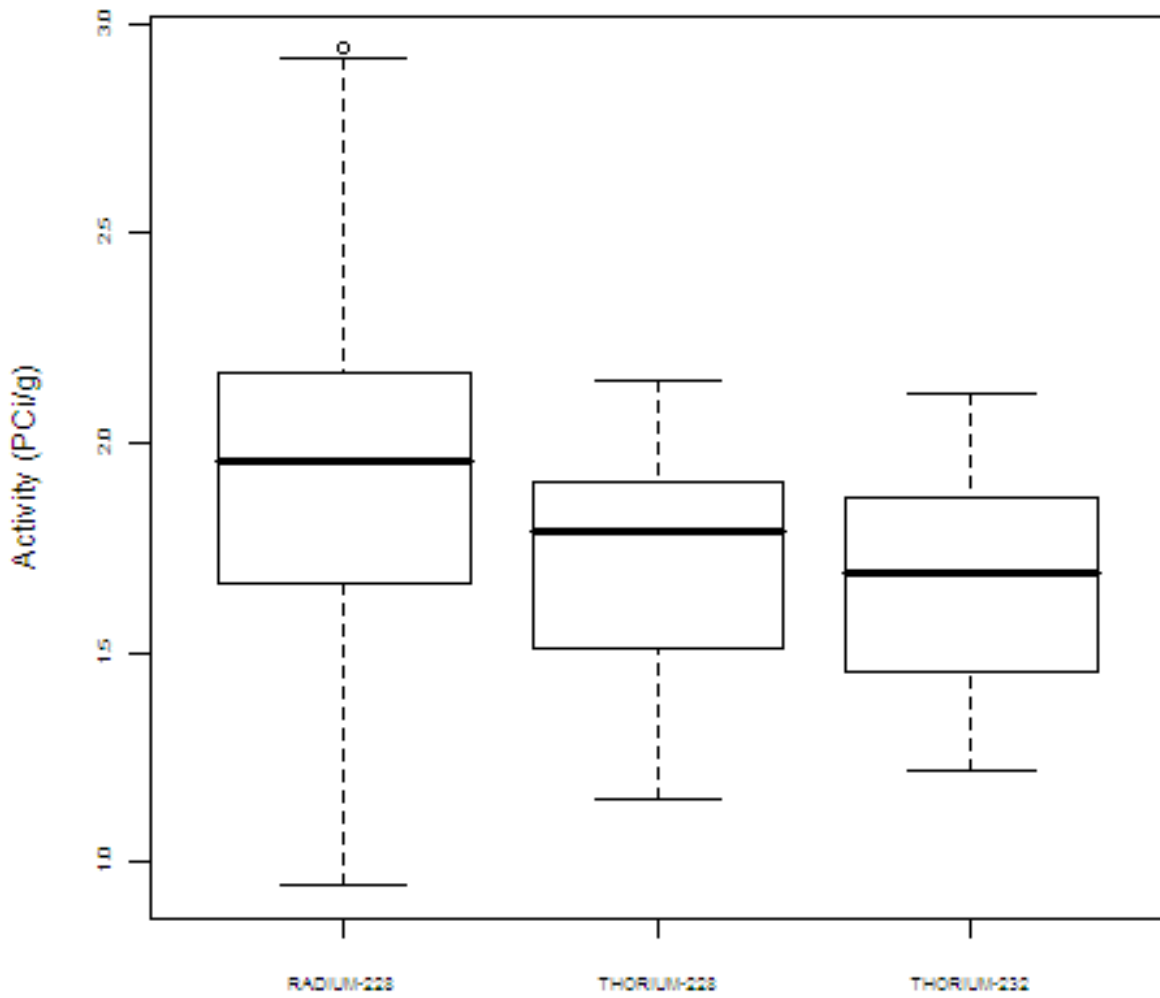
Radionuclide	Preparation Method	Analytical Method
Uranium isotopes	HF dissolution	Alpha spectroscopy consistent with DOE EML HASL-300 for isotopic uranium.
Thorium isotopes	HF dissolution	Alpha spectroscopy consistent with DOE EML HASL-300for isotopic thorium.
Radium-226	Requires further investigation	Alpha spectroscopy consistent with EPA methods 903.0/903.1 and 9315 with isotopically labeled barium as the tracer
Radium-228	Requires further investigation	Beta spectroscopy consistent with EPA methods 904.0 and 9320 with isotopically labeled barium as the tracer

Appendix A

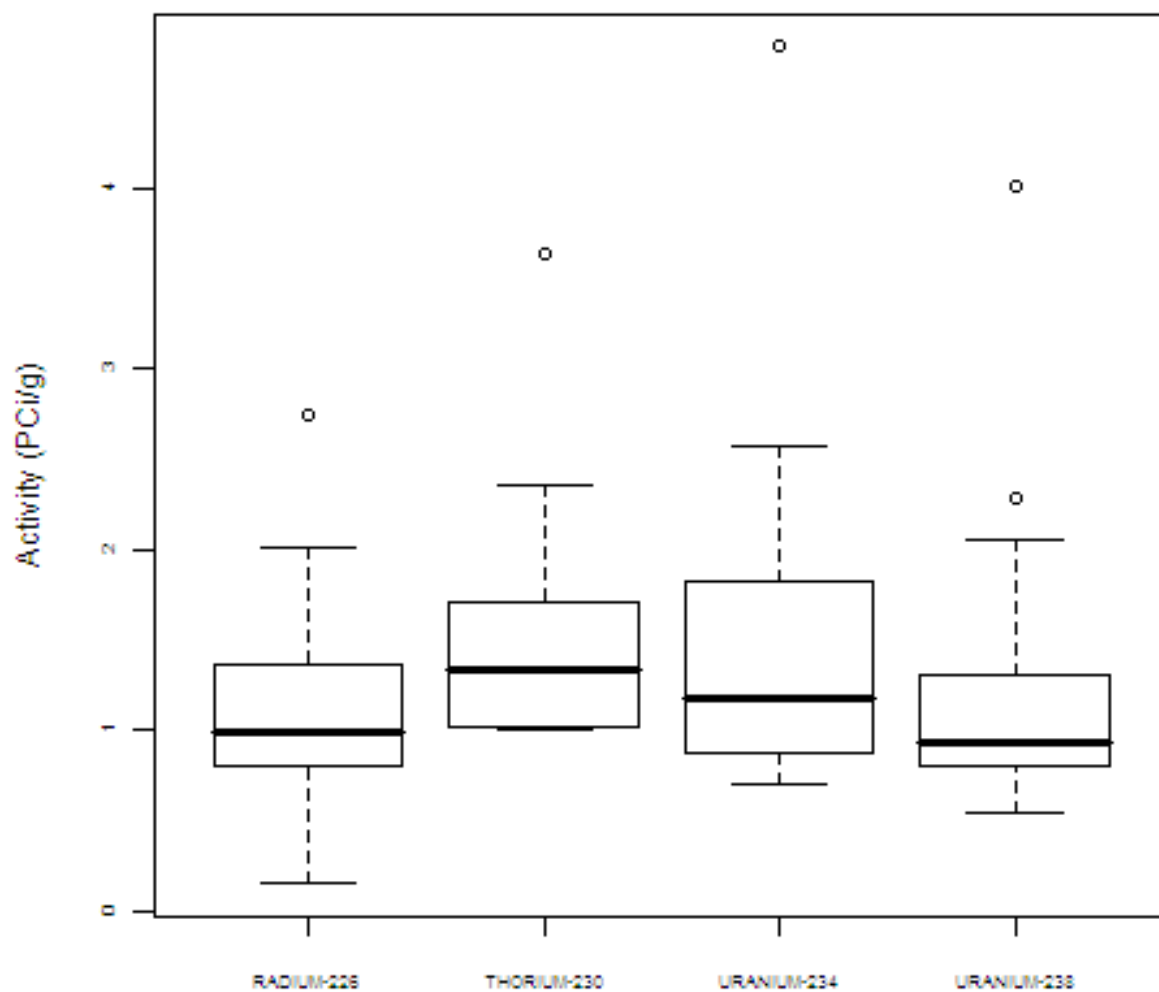
2005 BRC/TIMET Background



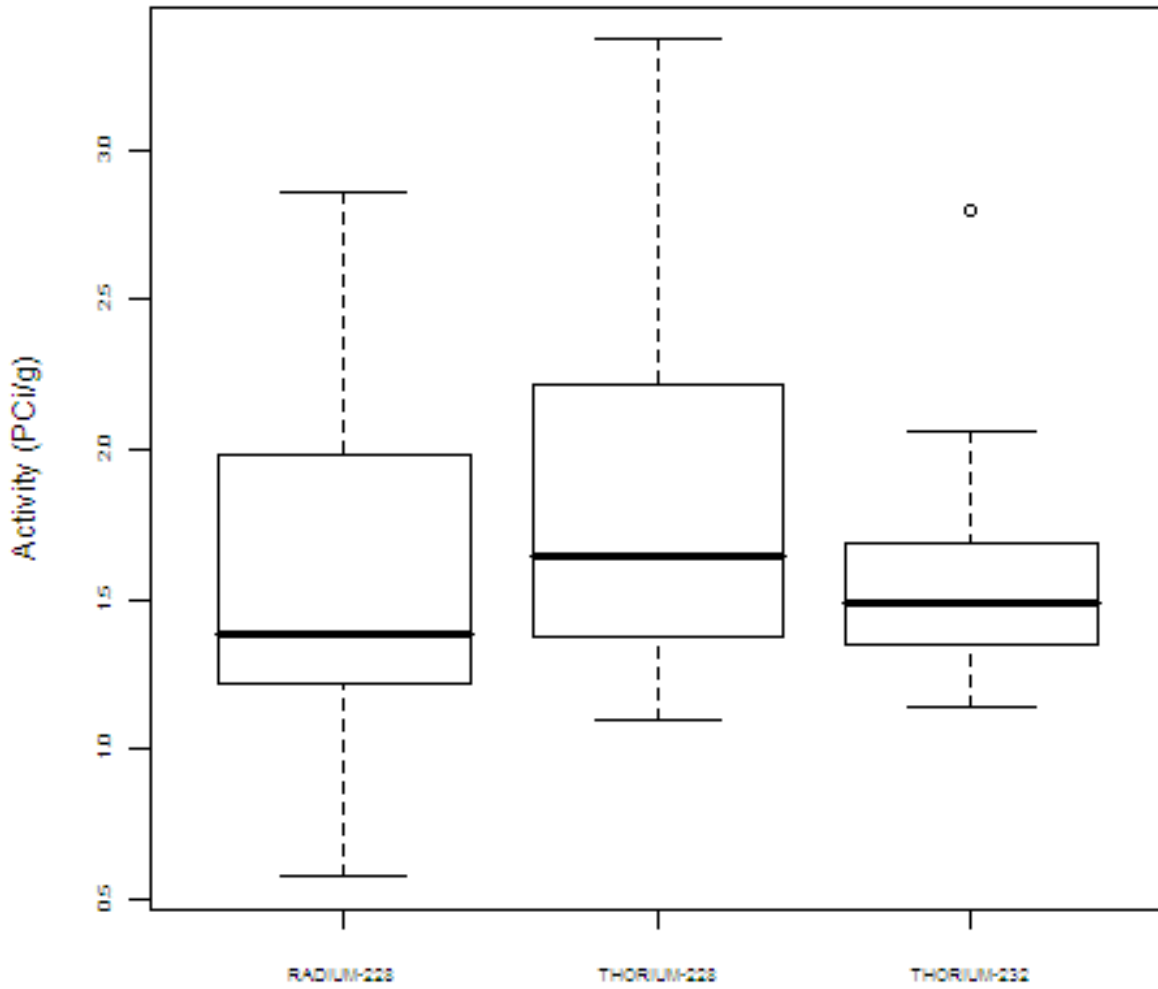
2005 BRC/TIMET Background



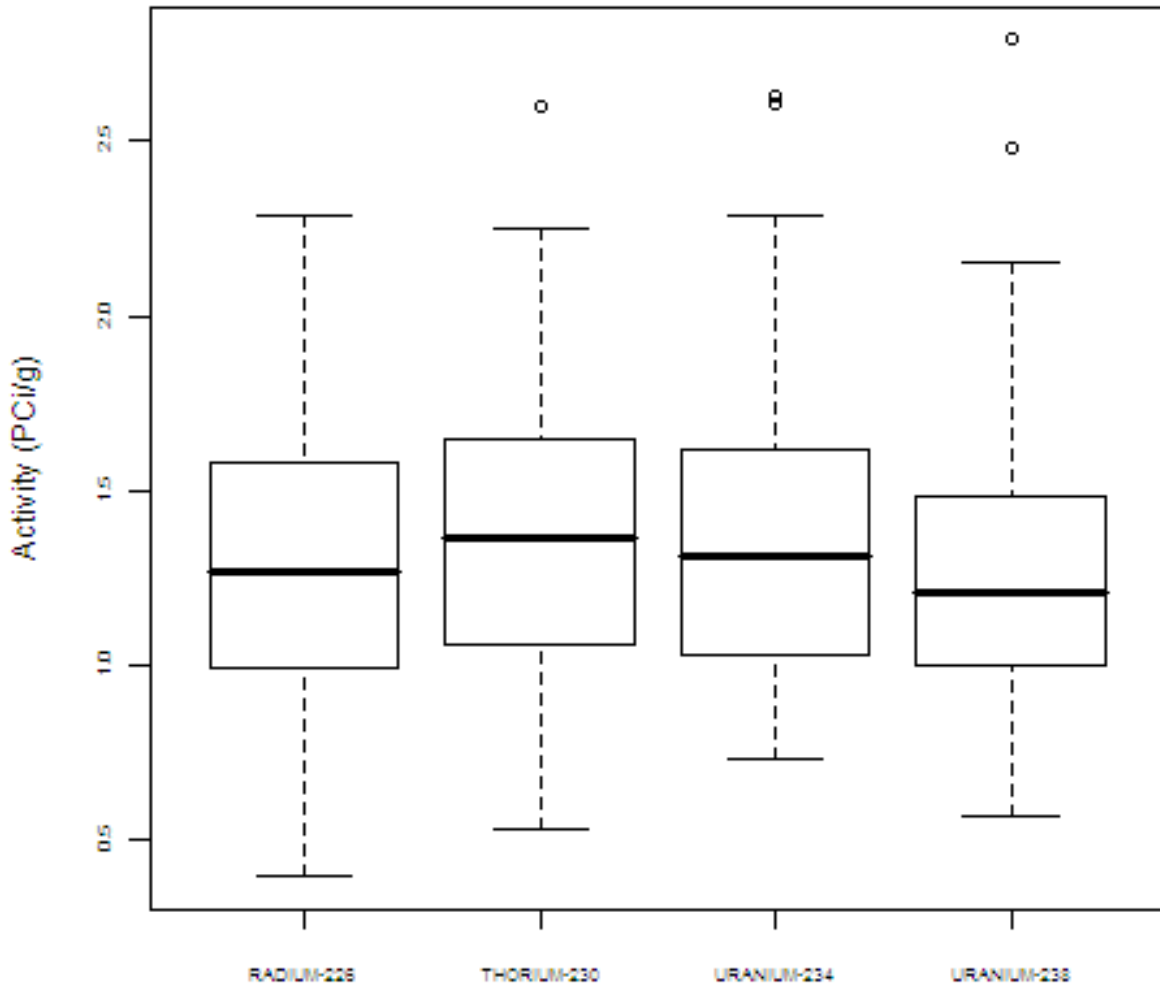
2008 Supplemental Background



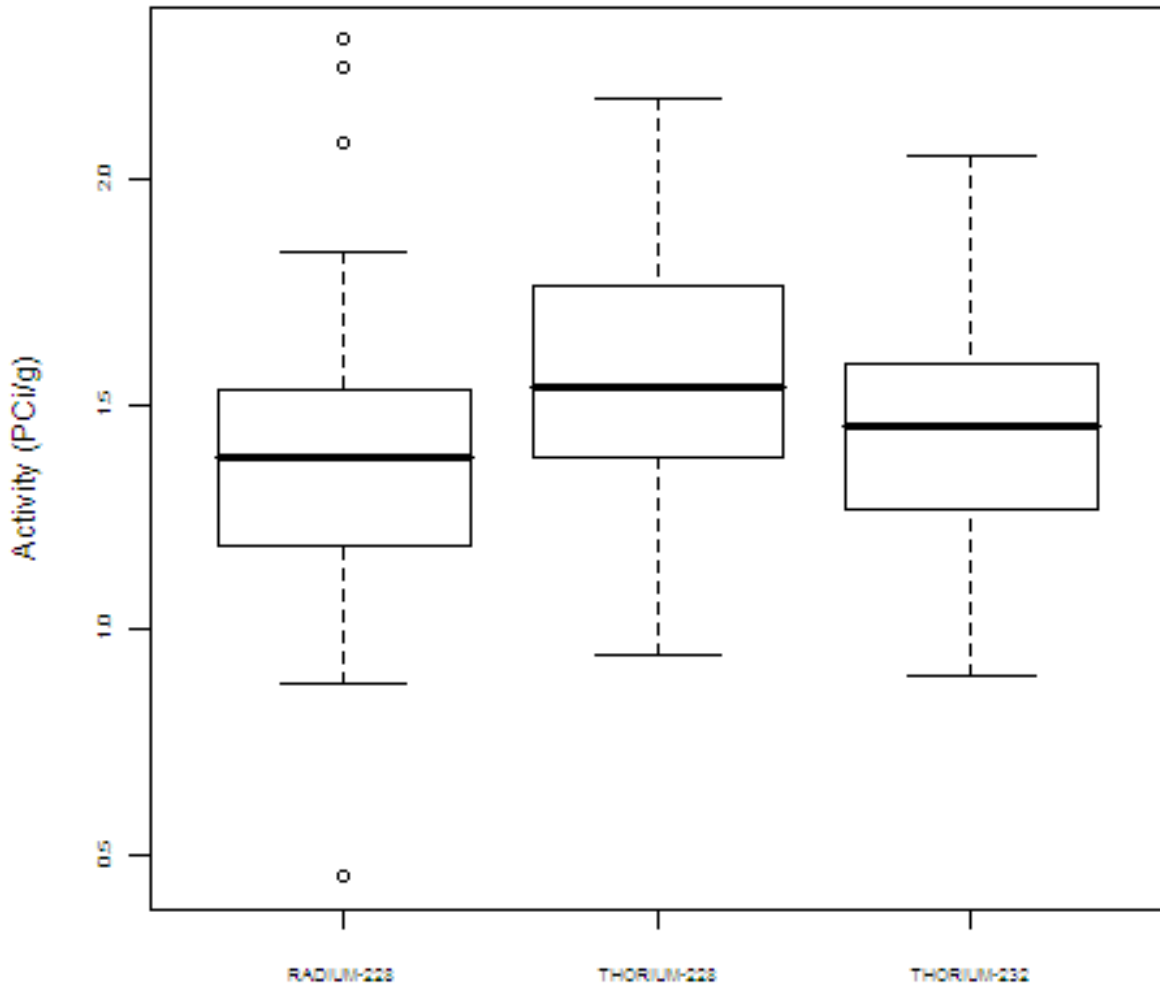
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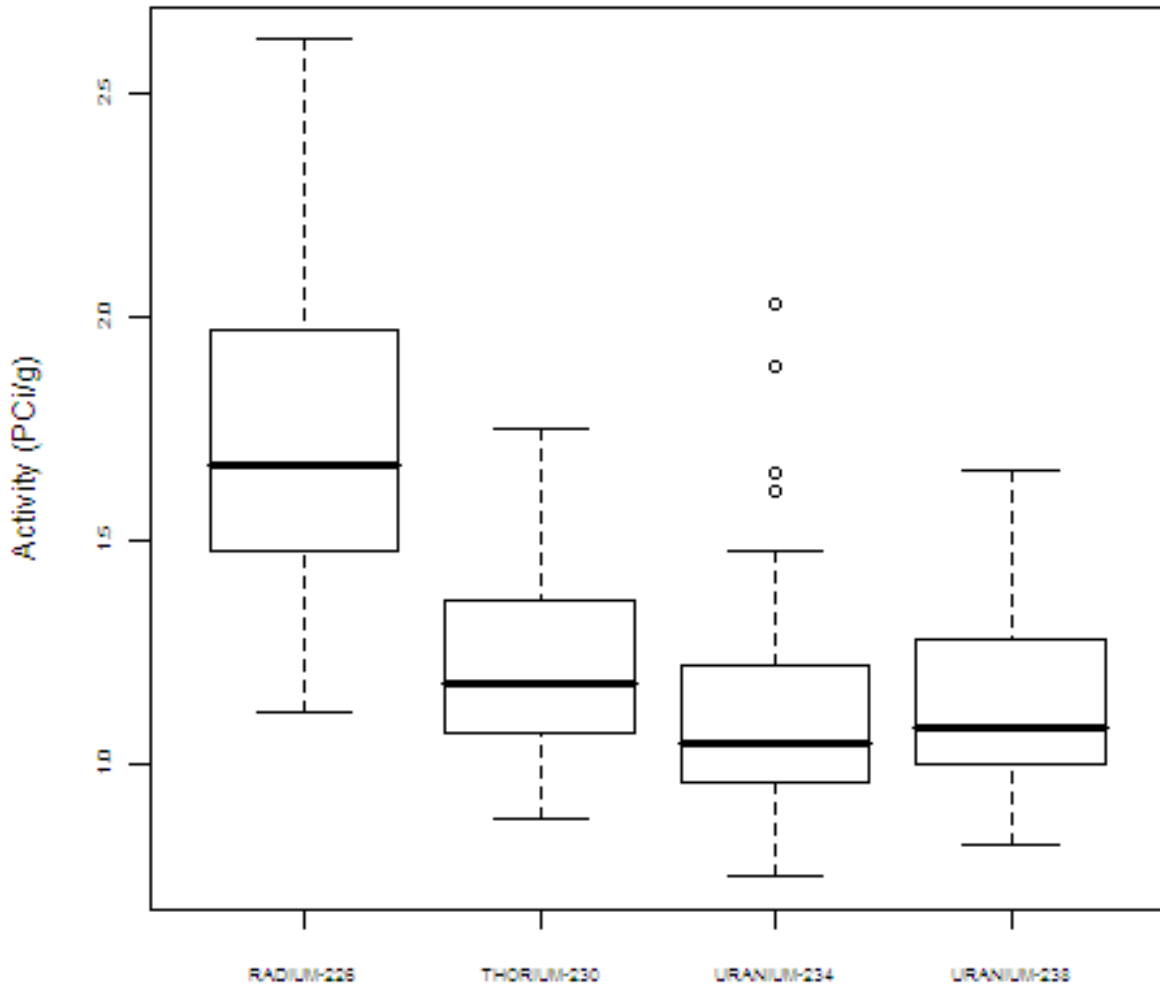
2008 Deep Background



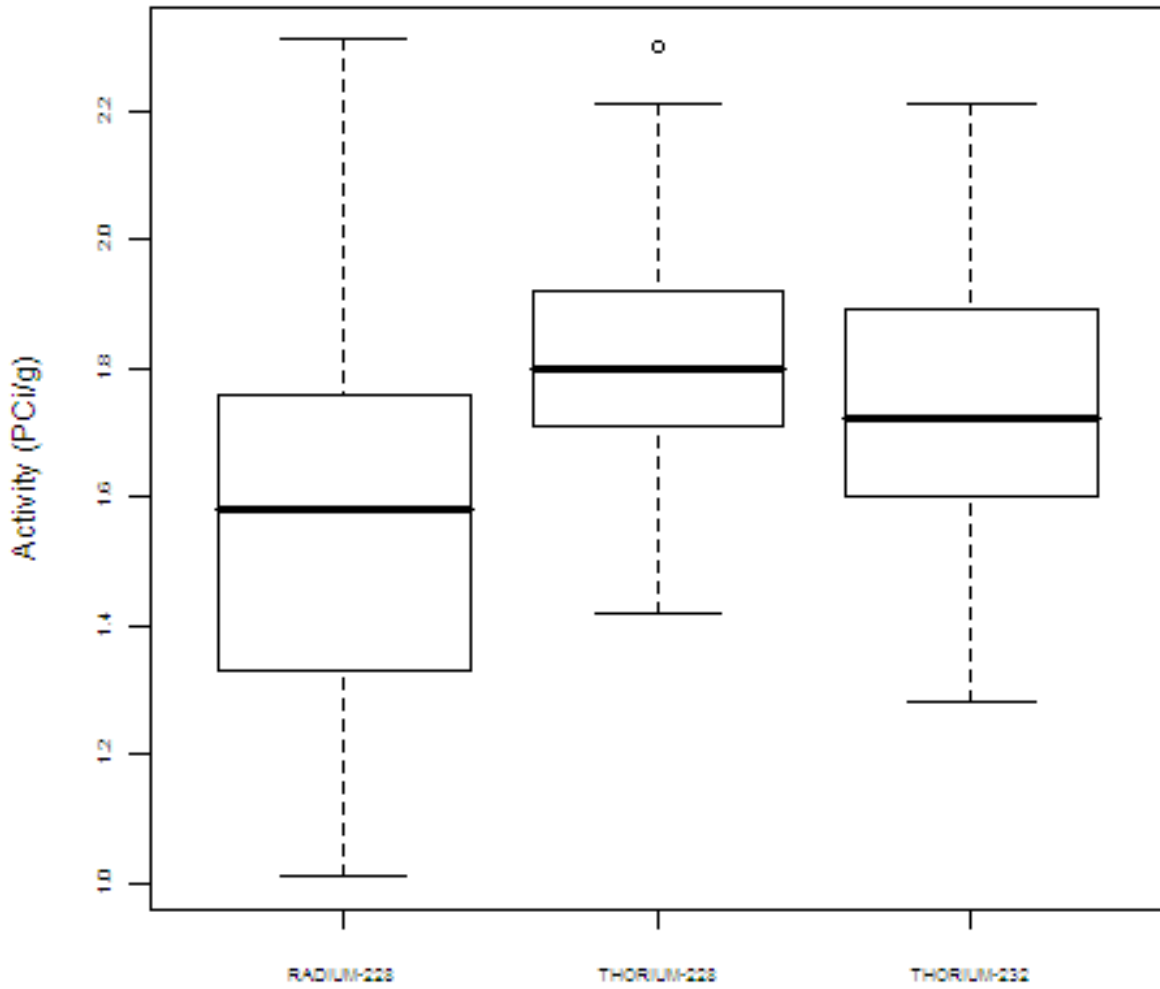
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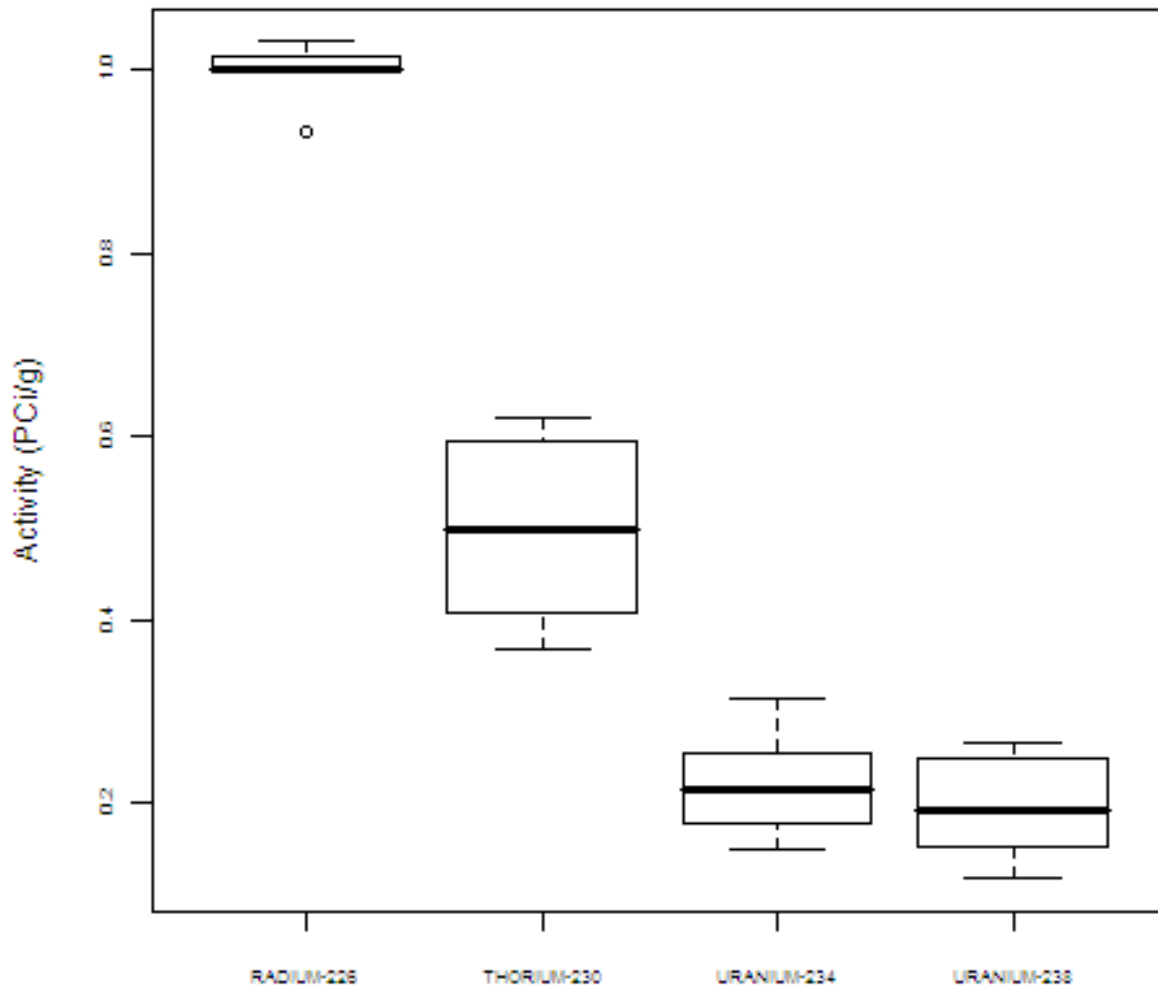
TRECO



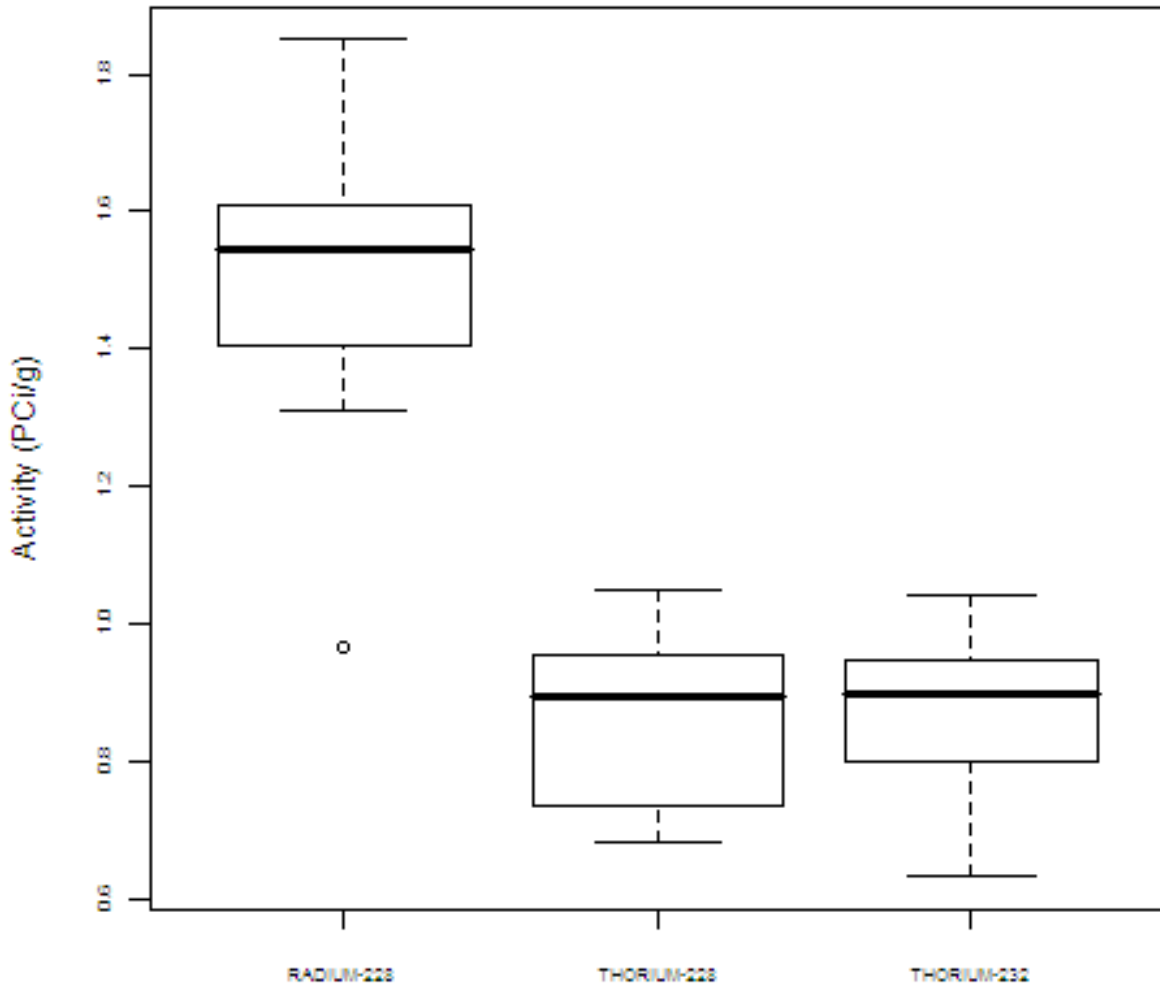
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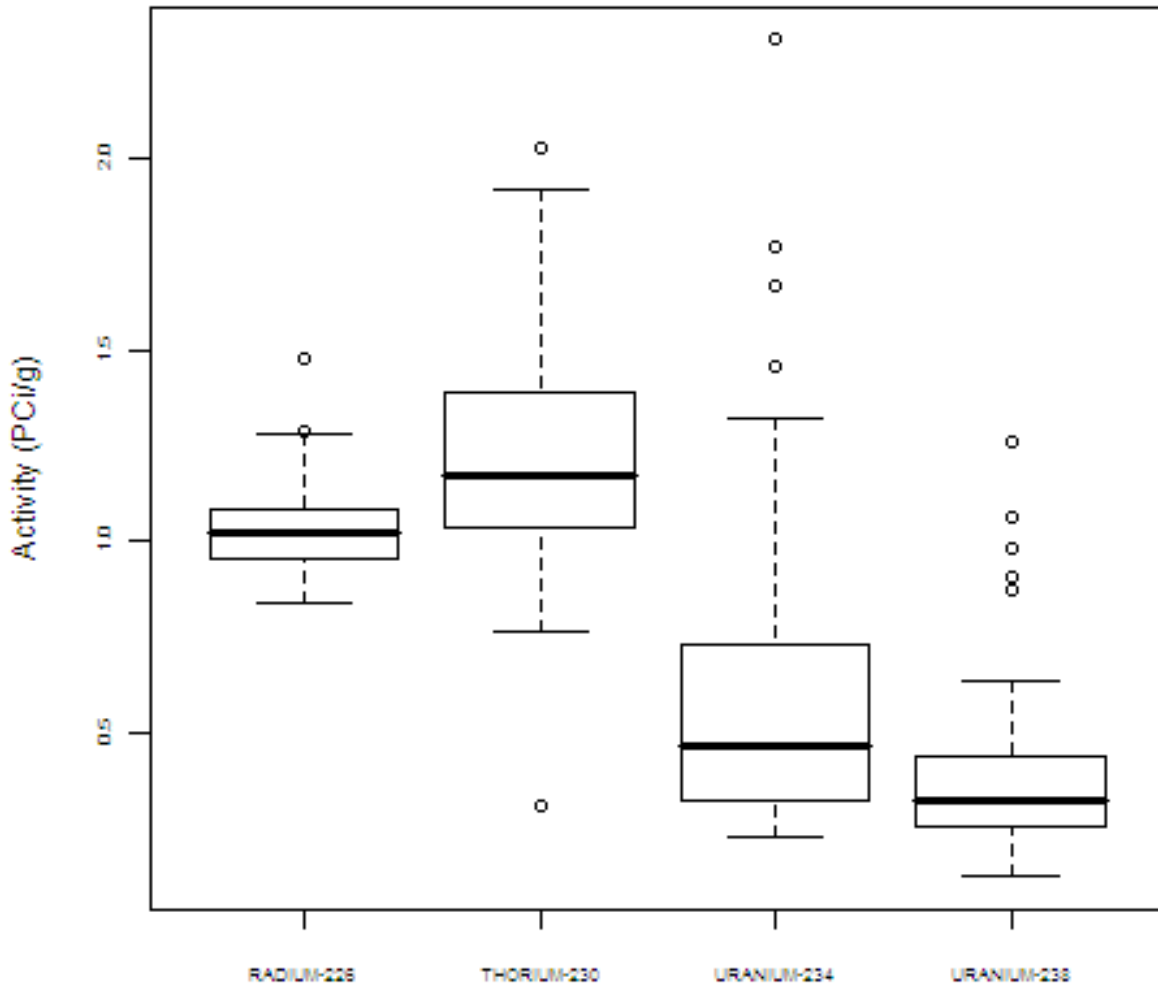
BRC Parcel 4B



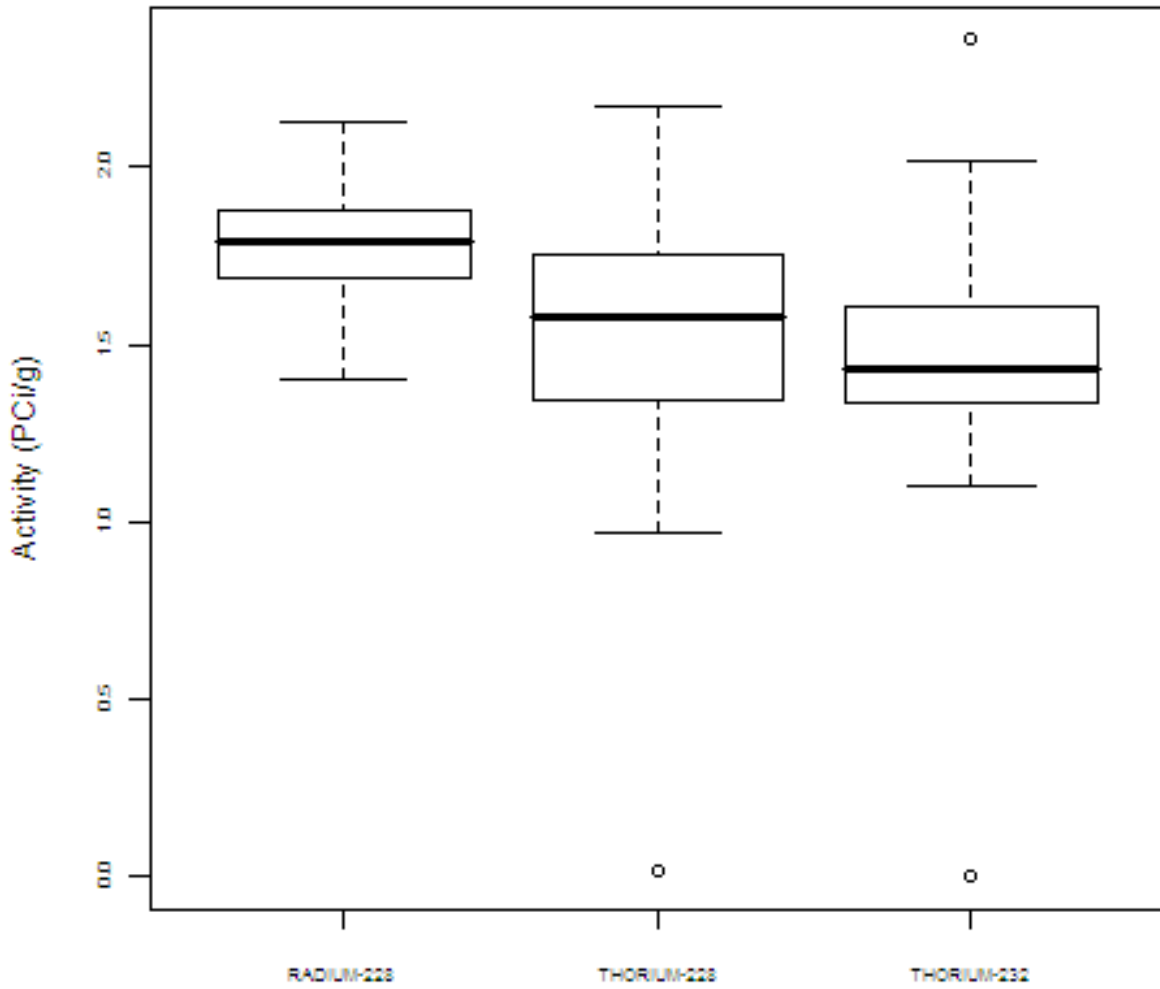
BRC Parcel 4B



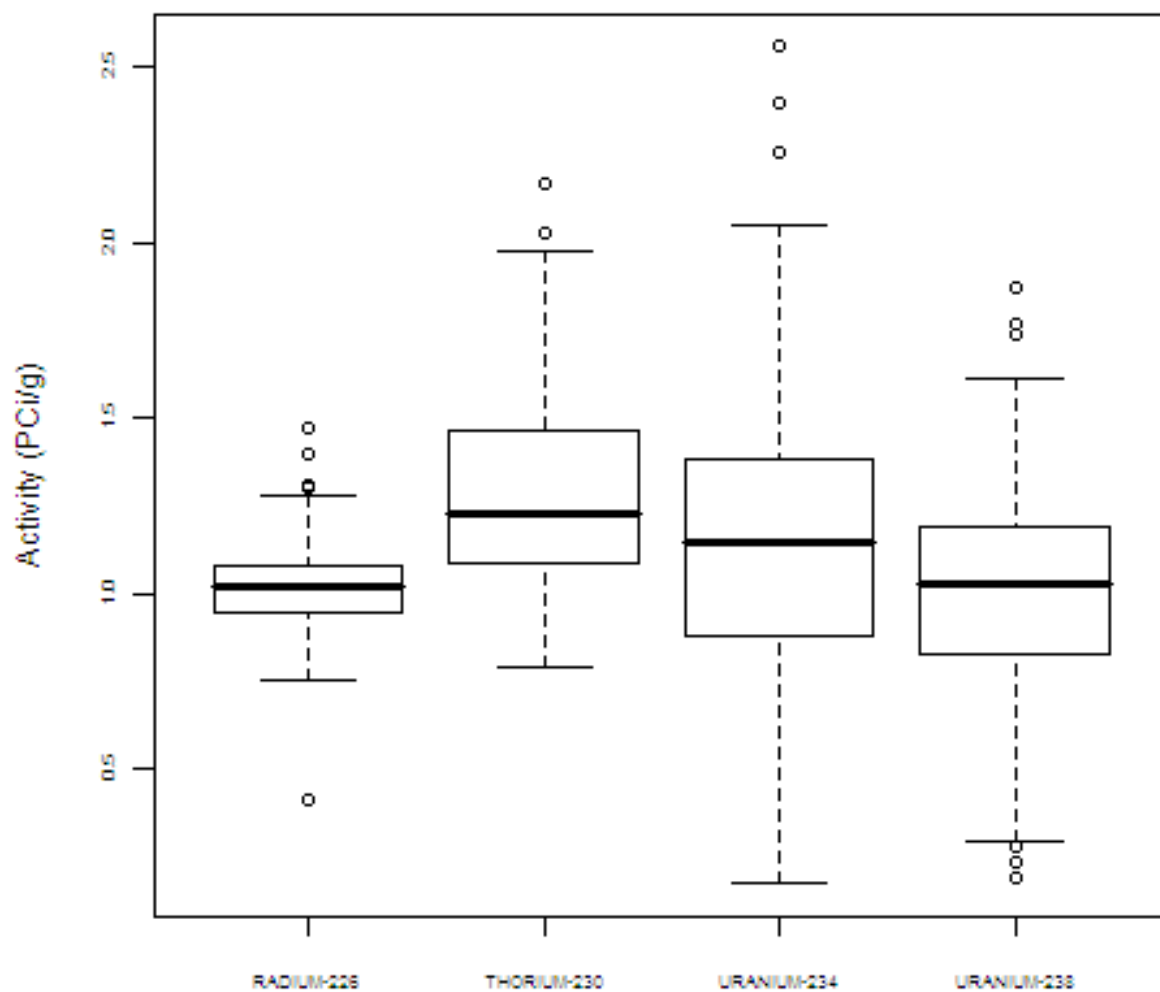
Tronox Parcels A/B



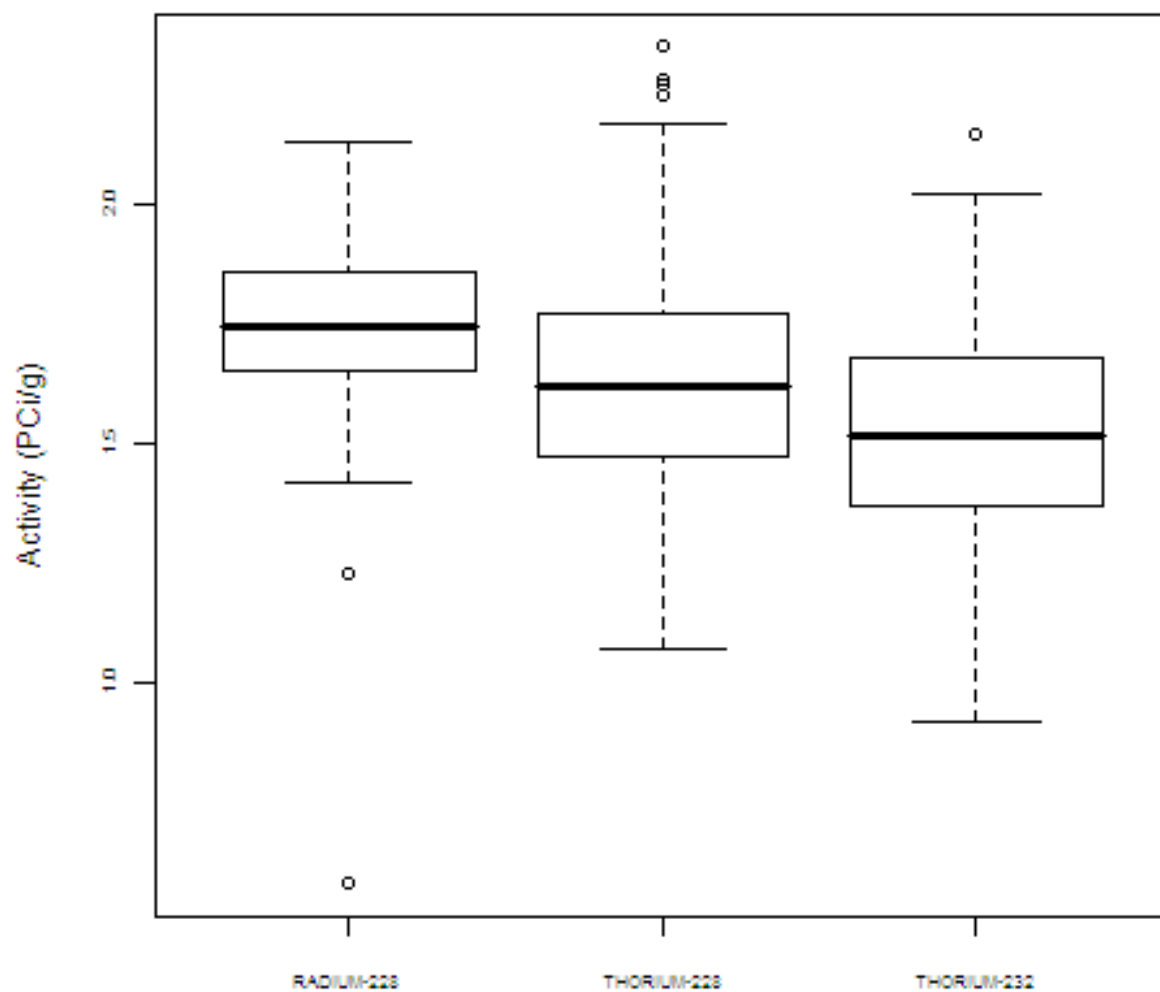
Tronox Parcels A/B



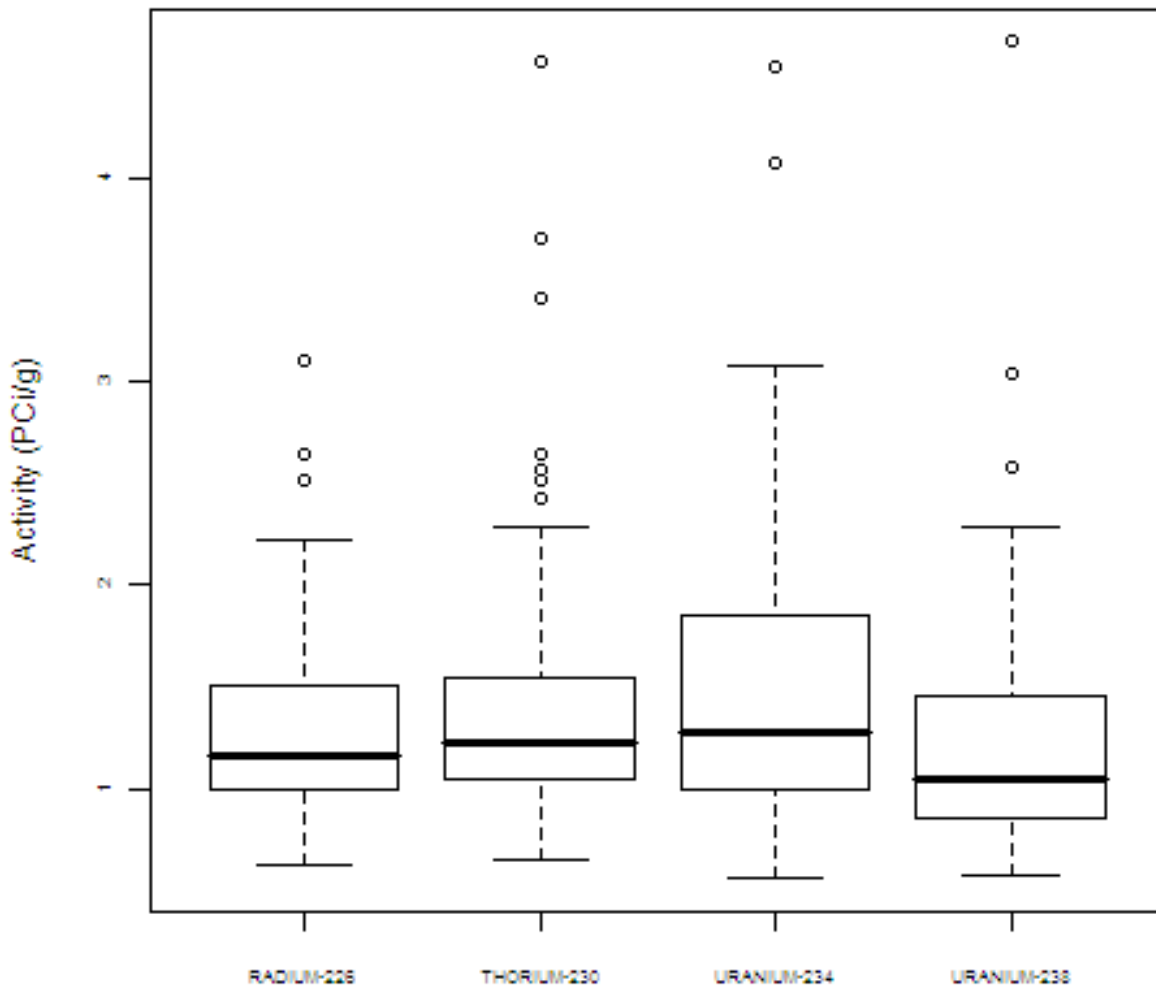
Tronox Parcels C/D/F/G



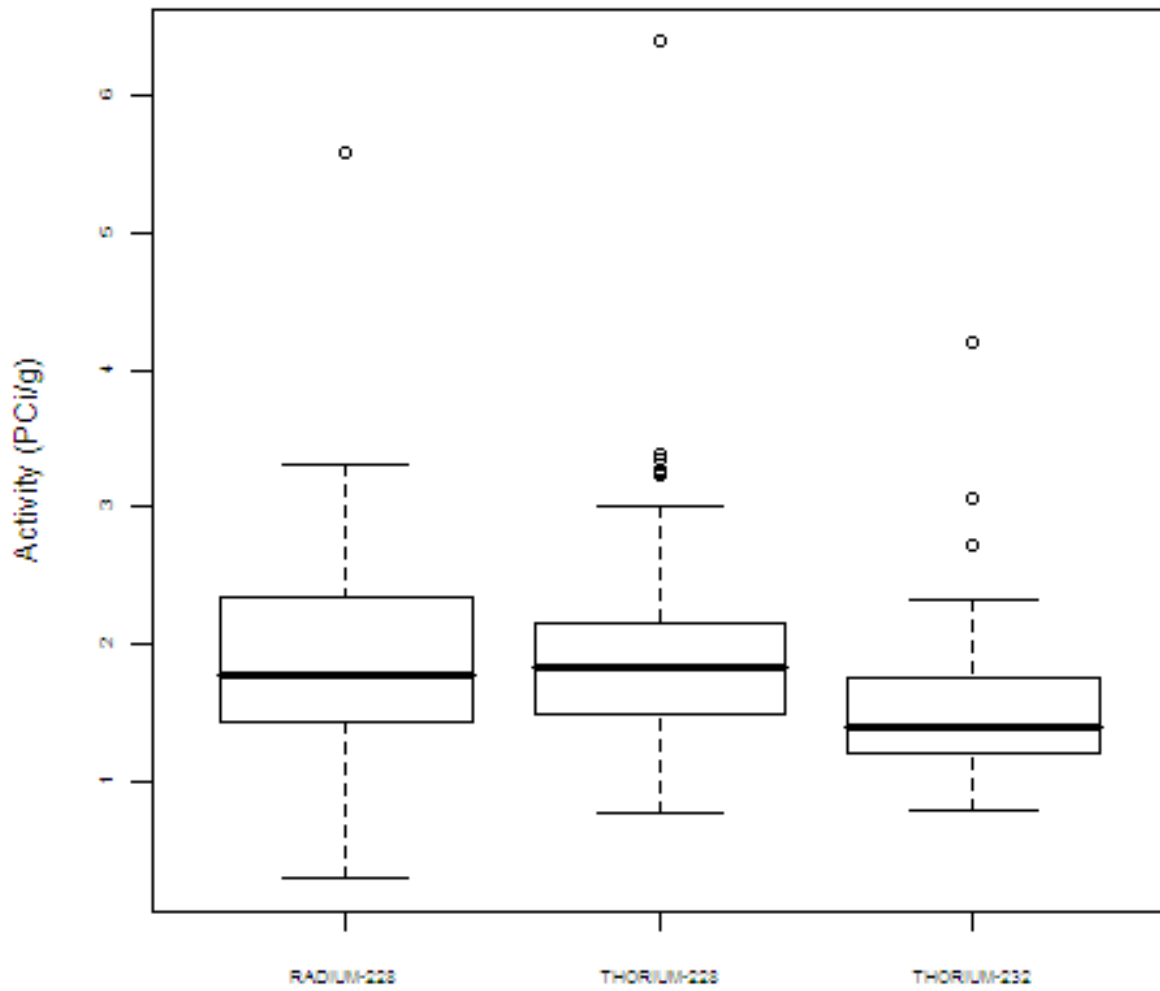
Tronox Parcels C/D/F/G



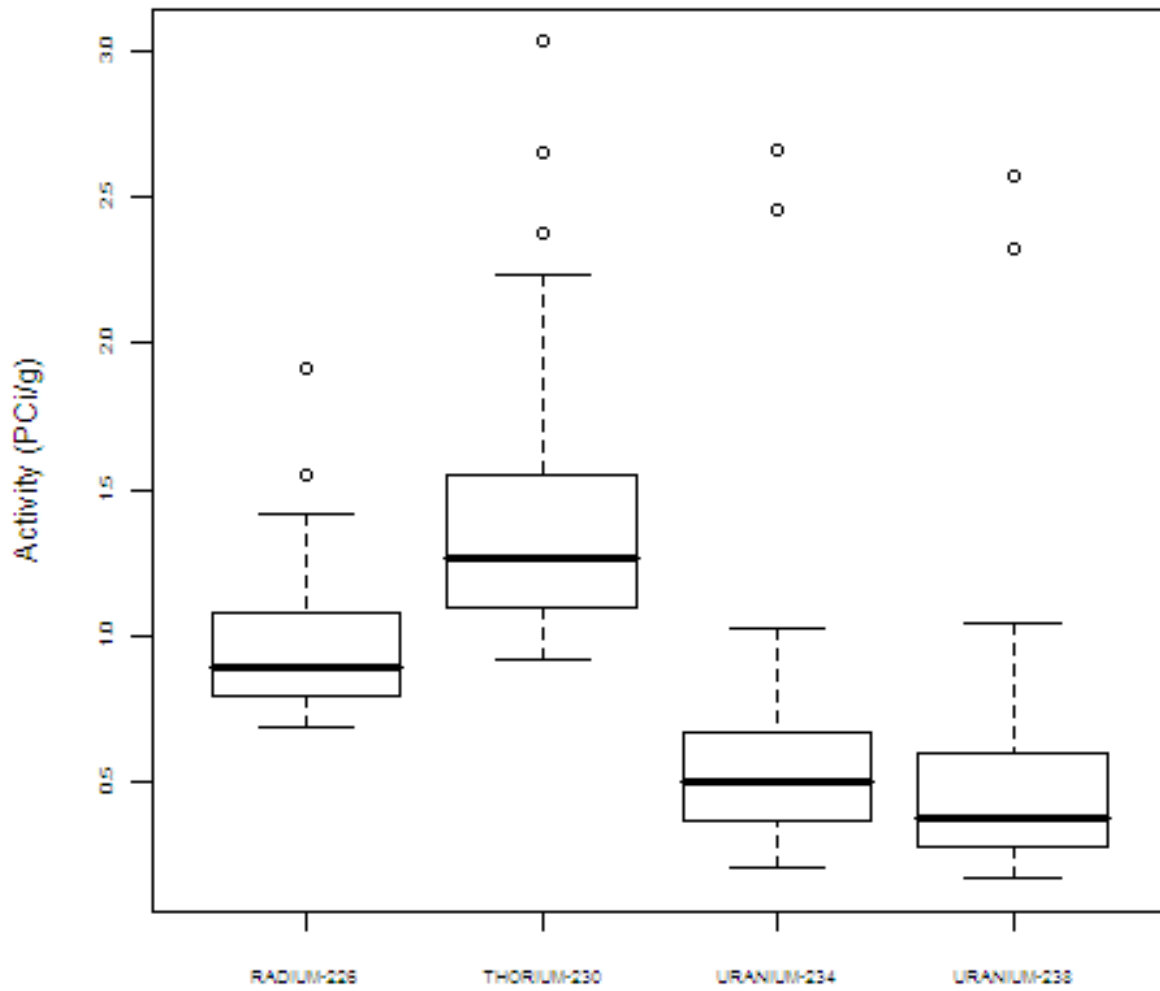
Utility Corridor



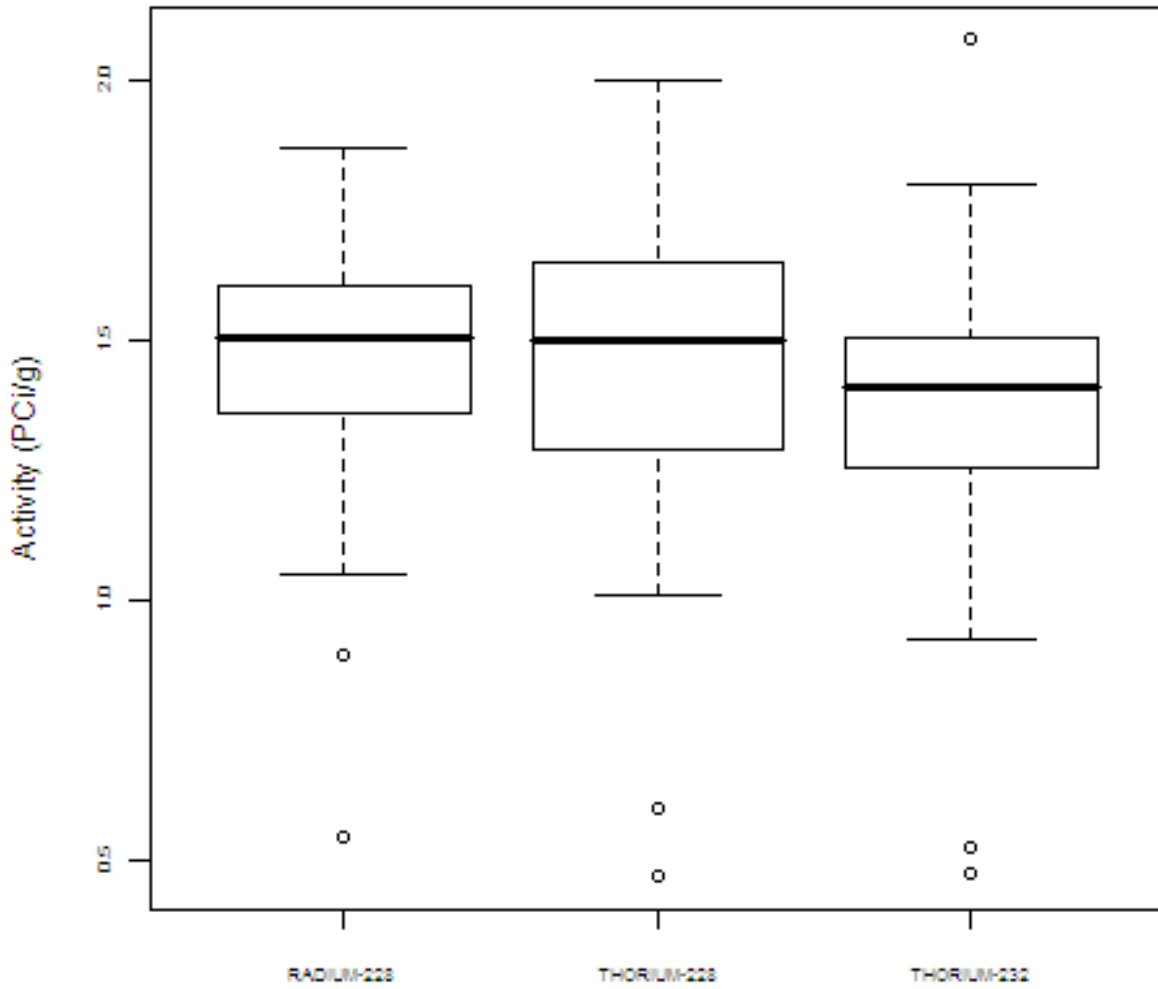
Utility Corridor



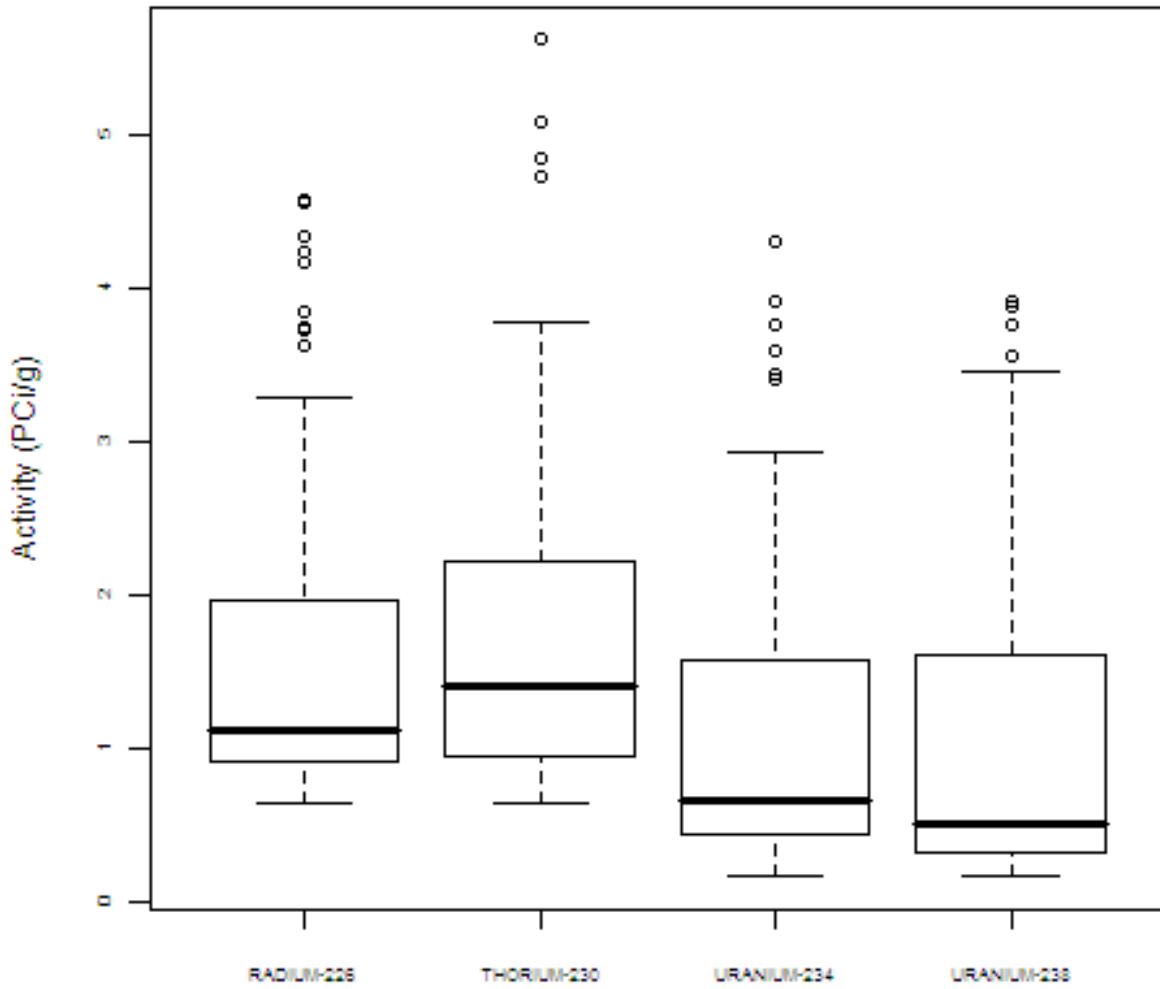
Upgradient Wells



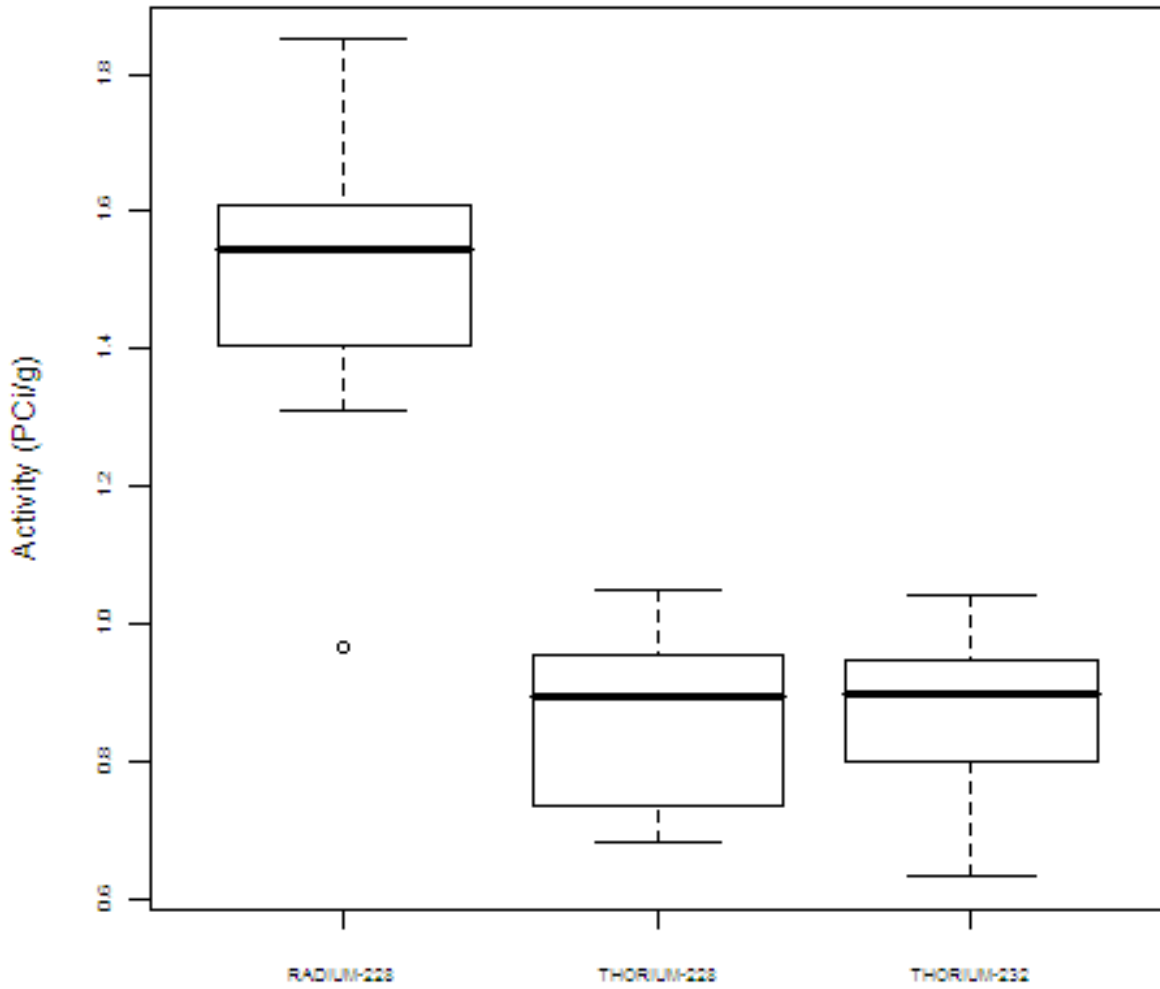
Upgradient Wells



Northeast Area Wells



Northeast Area Wells



Appendix B – Summary Statistics for the Uranium and Thorium Chains

	N	Mean	Std.Dev.	Min	Median	Max
2005 BRC/TIMET Shallow Background						
Radium-226	104	1.1122	0.3472	0.4940	1.0650	2.3600
Thorium-230	104	1.2651	0.4034	0.6600	1.2000	3.0100
Uranium-233/234	104	1.1607	0.4659	0.4700	1.0250	2.8400
Uranium-238	104	1.1352	0.3706	0.5700	1.0350	2.3700
Radium-228	84	1.9157	0.4046	0.9460	1.9600	2.9400
Thorium-228	84	1.7290	0.2552	1.1500	1.7900	2.1500
Thorium-232	84	1.6563	0.2553	1.2200	1.6900	2.1200
2008 Supplemental Shallow Background						
Radium-226	33	1.1008	0.5054	0.1530	0.9920	2.7500
Thorium-230	33	1.4948	0.5693	1.0000	1.3400	3.6400
Uranium-233/234	33	1.4618	0.8145	0.7000	1.1700	4.7800
Uranium-238	33	1.1976	0.6718	0.5450	0.9380	4.0100
Radium-228	33	1.5450	0.5490	0.5730	1.3800	2.8600
Thorium-228	33	1.7855	0.5074	1.1000	1.6400	3.3700
Thorium-232	33	1.5448	0.3228	1.1400	1.4900	2.8000
2008 Deep Soil Background						
Radium-226	92	1.2974	0.4232	0.3940	1.2650	2.2900
Thorium-230	92	1.3670	0.4254	0.5300	1.3650	2.6000
Uranium-233/234	92	1.3620	0.3938	0.7290	1.3150	2.6300
Uranium-238	92	1.2890	0.3745	0.5700	1.2050	2.7900
Radium-228	99	1.3744	0.2903	0.4520	1.3800	2.3100
Thorium-228	99	1.5820	0.2772	0.9440	1.5400	2.1800
Thorium-232	99	1.4546	0.2561	0.8980	1.4500	2.0500
TRECO						
Radium-226	57	1.7333	0.3927	1.1200	1.6700	2.6200
Thorium-230	57	1.2142	0.2061	0.8800	1.1800	1.7500
Uranium-233/234	57	1.1279	0.2549	0.7500	1.0500	2.0300
Uranium-238	57	1.1400	0.1854	0.8200	1.0800	1.6600
Radium-228	57	1.5602	0.2751	1.0100	1.5800	2.3100
Thorium-228	57	1.8333	0.1839	1.4200	1.8000	2.3000
Thorium-232	57	1.7519	0.2104	1.2800	1.7200	2.2100
Tronox Parcels A/B						
Radium-226	64	1.0376	0.1295	0.8370	1.0200	1.4800
Thorium-230	64	1.2070	0.3035	0.3080	1.1700	2.0300
Uranium-233/234	64	0.5908	0.4021	0.2250	0.4670	2.3100
Uranium-238	64	0.3832	0.2227	0.1250	0.3260	1.2600
Radium-228	64	1.7777	0.1560	1.4000	1.7900	2.1300
Thorium-228	64	1.5508	0.3327	0.0167	1.5800	2.1700
Thorium-232	64	1.4630	0.2983	0.0000	1.4300	2.3600

Appendix B (continued) – Summary Statistics for the U-238 and Th-232 Chains

	N	Mean	Std.Dev.	Min	Median	Max
Tronox Parcels C/D/F/G						
Radium-226	104	1.0179	0.1382	0.4120	1.0200	1.4700
Thorium-230	104	1.2972	0.2966	0.7920	1.2250	2.1700
Uranium-233/234	104	1.1701	0.4463	0.1730	1.1450	2.5600
Uranium-238	104	0.9907	0.34768	0.186	1.03	1.87
Radium-228	104	1.7425	0.1912	0.5800	1.7450	2.1300
Thorium-228	104	1.6340	0.2552	1.0700	1.6200	2.3300
Thorium-232	104	1.5296	0.2318	0.9200	1.5150	2.1500
Utility Corridor						
Radium-226	70	1.3517	0.5398	0.6240	1.1650	3.1000
Thorium-230	70	1.4361	0.7061	0.6440	1.2300	4.5700
Uranium-233/234	70	1.5353	0.7762	0.5570	1.2750	4.5500
Uranium-238	70	1.2404	0.6534	0.5700	1.0500	4.6700
Radium-228	70	1.8969	0.7880	0.2860	1.7700	5.5900
Thorium-228	70	1.9655	0.8309	0.7640	1.8200	6.4000
Thorium-232	70	1.5237	0.5442	0.7910	1.3950	4.2100
Upgradient Groundwater Wells						
Radium-226	44	0.9836	0.2834	0.6850	0.8950	1.9100
Thorium-230	44	1.4171	0.4756	0.9150	1.2650	3.0300
Uranium-233/234	44	0.6211	0.4782	0.2100	0.5035	2.6600
Uranium-238	44	0.5268	0.4749	0.1710	0.3745	2.5700
Radium-228	44	1.4574	0.2369	0.5440	1.5050	1.8700
Thorium-228	44	1.4442	0.2931	0.4680	1.5000	2.0000
Thorium-232	44	1.3643	0.2874	0.4720	1.4100	2.0800
BRC Parcel 4B						
Radium-226	8	0.9989	0.0306	0.9310	1.0000	1.0300
Thorium-230	8	0.4983	0.0983	0.3670	0.4970	0.6210
Uranium-233/234	8	0.2201	0.0550	0.1510	0.2155	0.3150
Uranium-238	8	0.1968	0.0558	0.1180	0.1930	0.2670
Radium-228	8	1.4918	0.2607	0.9640	1.5450	1.8500
Thorium-228	8	0.8616	0.1310	0.6810	0.8930	1.0500
Thorium-232	8	0.8700	0.1294	0.6320	0.8985	1.0400
Northeast Area Wells						
Radium-226	141	1.5190	0.8963	0.6400	1.1200	4.5700
Thorium-230	141	1.7226	0.9858	0.6300	1.4100	5.6200
Uranium-233/234	141	1.1061	0.9381	0.1700	0.6500	4.3100
Uranium-238	141	1.0252	0.9499	0.1600	0.5000	3.9200
Radium-228	59	1.1702	0.3201	0.3300	1.2700	1.7200
Thorium-228	59	1.1068	0.3723	0.1500	1.2100	1.8900

Appendix C – Correlation Matrices for the U-238 and Th-232 Chains

2005 BRC/TIMET Shallow Background

	<i>Ra-226</i>	<i>Th-230</i>	<i>U-233/234</i>	<i>U-238</i>
Ra-226	1.0000	0.6632	0.6911	0.7068
Th-230	0.6632	1.0000	0.7838	0.7796
U-233/234	0.6911	0.7838	1.0000	0.8763
U-238	0.7068	0.7796	0.8763	1.0000

	<i>Ra-228</i>	<i>Th-228</i>	<i>Th-232</i>
Ra-228	1.0000	0.2967	0.3049
Th-228	0.2967	1.0000	0.7323
Th-232	0.3049	0.7323	1.0000

2008 Supplemental Soil Background

	<i>Ra-226</i>	<i>Th-230</i>	<i>U-233/234</i>	<i>U-238</i>
Ra-226	1.0000	0.7019	0.7857	0.8115
Th-230	0.7019	1.0000	0.8305	0.8393
U-233/234	0.7857	0.8305	1.0000	0.9314
U-238	0.8115	0.8393	0.9314	1.0000

	<i>Ra-228</i>	<i>Th-228</i>	<i>Th-232</i>
Ra-228	1.0000	0.0101	-0.1041
Th-228	0.0101	1.0000	0.5484
Th-232	-0.1041	0.5484	1.0000

2008 Deep Soil Background

	<i>Ra-226</i>	<i>Th-230</i>	<i>U-233/234</i>	<i>U-238</i>
Ra-226	1.0000	0.7550	0.7646	0.7508
Th-230	0.7550	1.0000	0.8300	0.8024
U-233/234	0.7646	0.8300	1.0000	0.9335
U-238	0.7508	0.8024	0.9335	1.0000

	<i>Ra-228</i>	<i>Th-228</i>	<i>Th-232</i>
Ra-228	1.0000	0.2016	0.2570
Th-228	0.2016	1.0000	0.6722
Th-232	0.2570	0.6722	1.0000

TRECO

	<i>Ra-226</i>	<i>Th-230</i>	<i>U-234</i>	<i>U-238</i>
Ra-226	1.0000	0.3294	0.1671	0.1148
Th-230	0.3294	1.0000	0.5555	0.5760
U-234	0.1671	0.5555	1.0000	0.6645
U-238	0.1148	0.5760	0.6645	1.0000

	<i>Ra-228</i>	<i>Th-228</i>	<i>Th-232</i>
Ra-228	1.0000	0.2316	0.2295
Th-228	0.2316	1.0000	0.5647
Th-232	0.2295	0.5647	1.0000

Tronox Parcels A/B

	<i>Ra-226</i>	<i>Th-230</i>	<i>U-233/234</i>	<i>U-238</i>
Ra-226	1.0000	0.6548	0.4585	0.4636
Th-230	0.6548	1.0000	0.5058	0.5069
U-233/234	0.4585	0.5058	1.0000	0.9819
U-238	0.4636	0.5069	0.9819	1.0000

	<i>Ra-228</i>	<i>Th-228</i>	<i>Th-232</i>
Ra-228	1.0000	0.2626	0.0036
Th-228	0.2626	1.0000	0.6560
Th-232	0.0036	0.6560	1.0000

Tronox Parcels C/D/F/G

	<i>Ra-226</i>	<i>Th-230</i>	<i>U-233/234</i>	<i>U-238</i>
Ra-226	1.0000	0.4141	0.3186	0.2439
Th-230	0.4141	1.0000	0.4961	0.3746
U-233/234	0.3186	0.4961	1.0000	0.9028
U-238	0.2439	0.3746	0.9028	1.0000

Utility Corridor

	<i>Ra-226</i>	<i>Th-230</i>	<i>U-233/234</i>	<i>U-238</i>
Ra-226	1.0000	0.6224	0.5992	0.5520
Th-230	0.6224	1.0000	0.7368	0.7290
U-233/234	0.5992	0.7368	1.0000	0.8330
U-238	0.5520	0.7290	0.8330	1.0000

Upgradient Wells

	<i>Ra-226</i>	<i>Th-230</i>	<i>U-233/234</i>	<i>U-238</i>
Ra-226	1.0000	0.8075	0.8322	0.8423
Th-230	0.8075	1.0000	0.7793	0.7995
U-233/234	0.8322	0.7793	1.0000	0.9850
U-238	0.8423	0.7995	0.9850	1.0000

BRC Parcel 4B

	<i>Ra-226</i>	<i>Th-230</i>	<i>U-234</i>	<i>U-238</i>
Ra-226	1.0000	-0.2998	-0.4563	-0.0389
Th-230	-0.2998	1.0000	0.3565	0.3748
U-234	-0.4563	0.3565	1.0000	0.0298
U-238	-0.0389	0.3748	0.0298	1.0000

Northeast Area Wells

	<i>Ra-226</i>	<i>Th-230</i>	<i>U-233/234</i>	<i>U-238</i>
Ra-226	1.0000	0.9349	0.9208	0.9206
Th-230	0.9349	1.0000	0.9038	0.9072
U-233/234	0.9208	0.9038	1.0000	0.9859
U-238	0.9206	0.9072	0.9859	1.0000

	<i>Ra-228</i>	<i>Th-228</i>	<i>Th-232</i>
Ra-228	1.0000	0.2062	0.2237
Th-228	0.2062	1.0000	0.5664
Th-232	0.2237	0.5664	1.0000

	<i>Ra-228</i>	<i>Th-228</i>	<i>Th-232</i>
Ra-228	1.0000	0.3163	0.1109
Th-228	0.3163	1.0000	0.6544
Th-232	0.1109	0.6544	1.0000

	<i>Ra-228</i>	<i>Th-228</i>	<i>Th-232</i>
Ra-228	1.0000	0.7280	0.6814
Th-228	0.7280	1.0000	0.7009
Th-232	0.6814	0.7009	1.0000

	<i>Ra-228</i>	<i>Th-228</i>	<i>Th-232</i>
Ra-228	1.0000	0.6190	0.1974
Th-228	0.6190	1.0000	0.8198
Th-232	0.1974	0.8198	1.0000

	<i>Ra-228</i>	<i>Th-228</i>	<i>Th-232</i>
Ra-228	1.0000	0.8674	0.8154
Th-228	0.8674	1.0000	0.9047
Th-232	0.8154	0.9047	1.0000

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TRECO

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Northeast Area Wells

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