521

~~5-303.1~~

5-502.1

TRANSMITTAL OF PRELIMINARY AREA 1, PHASE I REMEDIAL ACTION DATA

11/21/96

DOE-0212-97
DOE-FEMP EPAS
25
REPORT

Department of Energy

Ohio Field Office
Fernald Area Office
P. O. Box 538705
Cincinnati, Ohio 45253-8705
(513) 648-3155

NOV 21 1996

DOE-0212-97

Mr. James A. Saric, Remedial Project Director
U.S. Environmental Protection Agency
Region V - 5HSF-5J
77 W. Jackson Boulevard
Chicago, Illinois 60604-3590

Mr. Tom Schneider, Project Manager
Ohio Environmental Protection Agency
401 East 5th Street
Dayton, Ohio 45402-2911

Dear Mr. Saric and Mr. Schneider:

TRANSMITTAL OF PRELIMINARY AREA 1, PHASE I REMEDIAL ACTION DATA

The purpose of this letter is to transmit a preliminary, partial set of the real time and laboratory analytical results from the ongoing efforts in the Area 1, Phase I Remedial Action Project to support our meeting on November 25, 1996. These partial analytical and insitu radioactivity measurement results reflect the data which is currently available. In order to have sufficient analytical data to compare to the insitu radiation measurements data at our upcoming meeting these laboratory analytical results were obtained from the analysis of both Waste Acceptance Criteria (WAC) attainment samples and duplicate samples taken from the comparability study. The laboratory analytical method used to measure total uranium was the BromoPADAP method. Isotopic uranium was measured using the Inductively Coupled Plasma - Mass Spectrometry (ICP-MS) method (total uranium obtained by summing individual uranium isotopes). As outlined in the Remedial Action Work Plan, the certification samples and the comparability study samples are being analyzed using alpha/gamma spectrometry. These results are not yet available.

In addition, as per the request from the Ohio Environmental Protection Agency (OEPA), the Fernald Environmental Management Project (FEMP) has provided to the OEPA the complete RTRAK radiation measurements data file in the requested File Transfer Protocol (FTP) format. However, the FEMP must emphasize that this data is very preliminary and has not been reviewed.

If you or your staff should have any questions, please contact Robert Janke at (513) 648-3124.

Sincerely,

Susan M Peter

for Johnny Reising
Fernald Remedial Action
Project Manager

FEMP:R.J. Janke

Enclosure: As Stated

cc w/enc:

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C. Little, FDF/2
EDC, FDF/52-7

ENCLOSED DATA PACKAGE

November 21, 1996

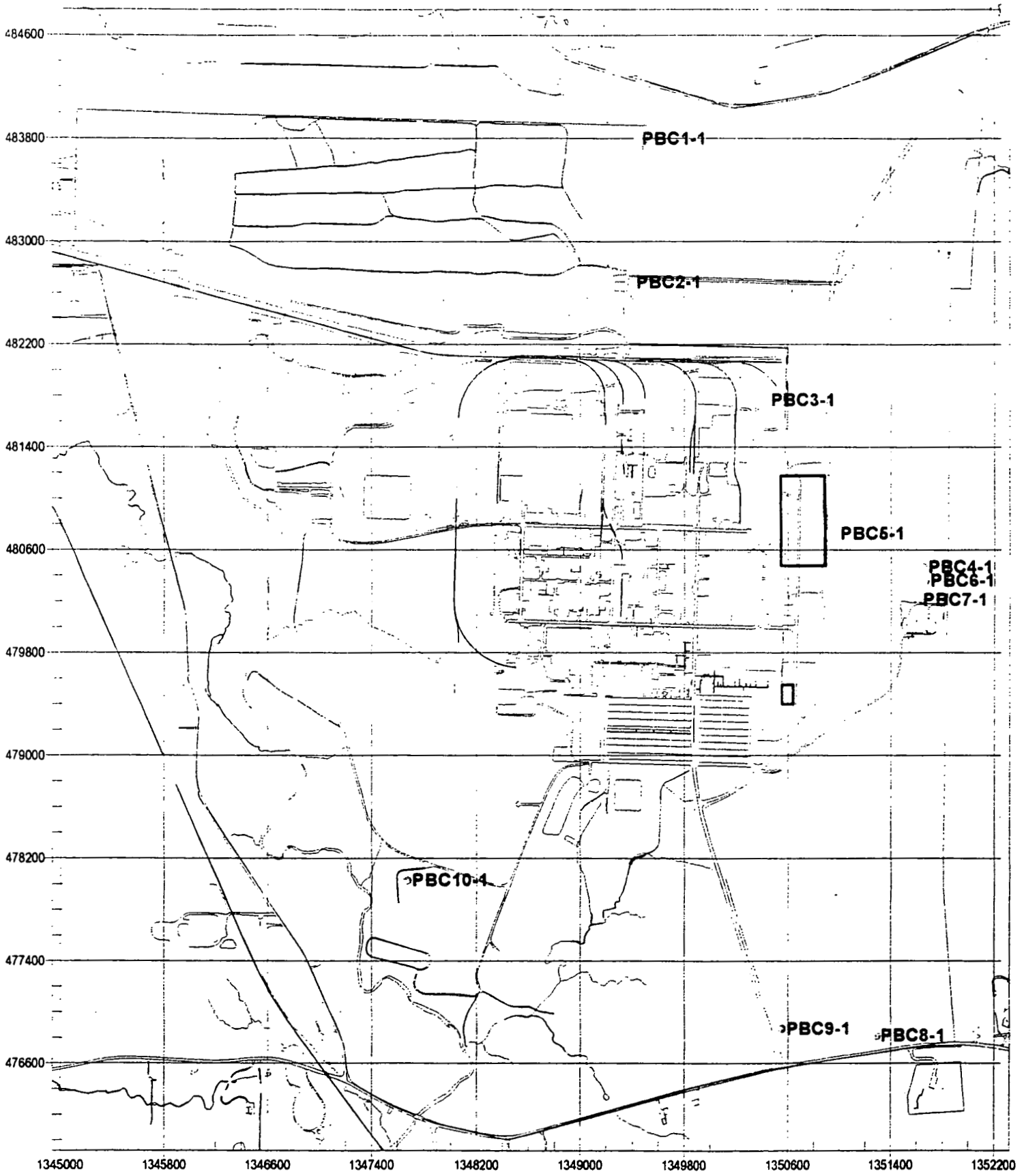
Enclosed is an assimilation of selected data that has been obtained in the work that has been done thus far on the FEMP Area 1 Phase I soil remediation project. The purpose of this data package is to present some preliminary results that compare levels of radionuclides in the FEMP soil obtained by different methods including the RTRAK, HPGe detector systems, and discrete sampling and analytical testing. Most of the data presented was generated as a result of the initial stages of Part B of the Comparability Study Report scheduled to be submitted in March of 1997. This Preliminary data package is not to be viewed as representing this Comparability Study but is only an review of preliminary data points obtained by various methods.

The laboratory results from physical soil samples contained in this data package were analyzed by BromoPADAP and ICP-MS. These techniques are described by the enclosed method descriptions. Both of these methods have proven to be practical and accurate analytical methods for determining total uranium in a soil matrix. The soil samples collected in the Comparability Study are also being analyzed for uranium and other radionuclides by the more conventional isotopic alpha and gamma spec. methods. At the time this package was prepared this data was not yet available for these samples.

The HPGe systems used in the tests were positioned at both 1 ft (25 cm) and 1 m for purpose of comparing read heights. Multiple readings were then taken to demonstrate repeatability. These preliminary results indicate that the output of HPGe field instrument is consistent with the average of physical samples obtained from the instrumentation read area. When submitted the Comparability Study will examine in more detail gamma discernable isotopes in FEMP soil other than uranium. Additional information enclosed includes comparability between physical samples, HPGe, and RTRAK obtained during the WAC attainment effort in the East Field.

Keith Nelson

PART B COMPARABILITY STUDY
Sample Locations



COMPARABILITY STUDY

SAMPLE ID		% H2O	BrPADAP ppm	% H20 corrected	ICP-MS ppm	% H20 corrected	Alpha Spec ppm	% H20 corrected	HPGe Field Results	
200264124	PBC 1-1			0.0		0.0		0.0	.31 meters	9.0
200264125	PBC 1-2	23.6	16.3	12.5		0.0		0.0	1.0 meters	8.0
200264128	PBC 1-5	24.4	14.0	10.6	14.8	11.2		0.0		
200264130	PBC 1-7	22.1	11.6	9.0	12.7	9.9		0.0		
200264198	PBC 2-2	24.2	47.5	36.0	60.0	45.5		0.0	.31 meters	31.3
200264201	PBC 2-5	24.3	42.4	32.1		0.0		0.0	1.0 meters	26.0
200264202	PBC 2-6	22.2	23.0	17.9		0.0		0.0		
200264206	PBC 2-10	30.1	38.6	27.0		0.0		0.0		
200264209	PBC 3-2	25.9	351.1	260.2	294.1	217.9		0.0	.31 meters	290.8
200264210	PBC 3-3	26.1	339.8	251.1	274.2	202.6		0.0	1.0 meters	286.6
200264142	PBC 3-4	25.0	419.0	314.3	337.8	253.4		0.0		
200264149	PBC 4-7	25.3	60.2	45.0		0.0		0.0	.31 meters	56.6
200264152	PBC 4-10	24.6	77.1	58.1		0.0		0.0	1.0 meters	58.6
200264131	PBC 5-1	21.8	47.4	37.1	43.2	33.8		0.0		
200264132	PBC 5-2	24.0	53.1	40.4	49.1	37.3		0.0		
200264133	PBC 5-3	24.9	54.0	40.6	50.4	37.9		0.0	.31 meters	42.2
200264134	PBC 5-4	23.9	53.3	40.6	51.8	39.4		0.0	1.0 meters	47.9
200264136	PBC 5-6	27.8	91.3	65.9	94.8	68.4		0.0		
200264137	PBC 5-7	25.5	85.9	64.0	86.1	64.1		0.0		
200264138	PBC 5-8	22.3	43.8	34.0		0.0		0.0		
200264139	PBC 5-9	21.4	64.3	50.5	66.2	52.0		0.0		
200264140	PBC 5-10	26.0	59.3	43.9	59.7	44.2		0.0		
200264141	PBC 5-11	26.0	71.8	53.1	75.5	55.9		0.0		
200264162	PBC 6-9	24.1	92.9	70.5	105.4	80.0		0.0	.31 meters	76.8
									1.0 meters	75.9
200264165	PBC 7-1	28.4	193.8	138.8	208.4	149.2		0.0		
200264166	PBC 7-2	29.5	235.4	166.0	245.4	173.0		0.0		
200264167	PBC 7-3	28.4	215.6	154.4	227.1	162.6		0.0		
200264168	PBC 7-4	26.6	195.1	143.2	157.7	115.8		0.0		
200264169	PBC 7-5	27.5	247.6	179.5		0.0		0.0	.31 meters	141.2
200264170	PBC 7-6	27.0	284.3	207.5		0.0		0.0	1.0 meters	140.4
200264171	PBC 7-7	27.2	251.9	183.4		0.0		0.0		
200264173	PBC 7-9	28.7	218.2	155.6	231.7	165.2		0.0		
200264175	PBC 7-11	27.7	189.1	136.7	198.8	143.7		0.0		
200264178	PBC 7-14	23.7	210.3	160.5	234.3	178.8		0.0		
200264179	PBC 7-15	28.9	264.2	187.8		0.0		0.0		
200264117	PBC 8-1	29.9	7.6	5.3		0.0		0.0		
200264118	PBC 8-2	35.1	7.9	5.1		0.0		0.0		
200264119	PBC 8-3	26.3	7.4	5.5		0.0		0.0	.31 meters	5.9
200264121	PBC 8-5	28.2	6.2	4.5	4.7	3.4		0.0	1.0 meters	5.3
200264122	PBC 8-6	34.0	10.2	6.7		0.0		0.0		
200264123	PBC 8-7	32.6	8.6	5.8		0.0		0.0		
200264110	PBC 9-1	29.3	10.0	7.1	8.6	6.1		0.0		
200264111	PBC 9-2	27.3	8.9	6.5		0.0		0.0		
200264112	PBC 9-3	26.3	8.0	5.9		0.0		0.0		
200264113	PBC 9-4	26.3	8.9	6.6	8.1	6.0		0.0	.31 meters	6.6
200264114	PBC 9-5	26.4	8.7	6.4		0.0		0.0	1.0 meters	6.9
200264115	PBC 9-6	24.8	10.0	7.5	8.1	6.1		0.0		
200264116	PBC 9-7	25.6	10.2	7.6		0.0		0.0		

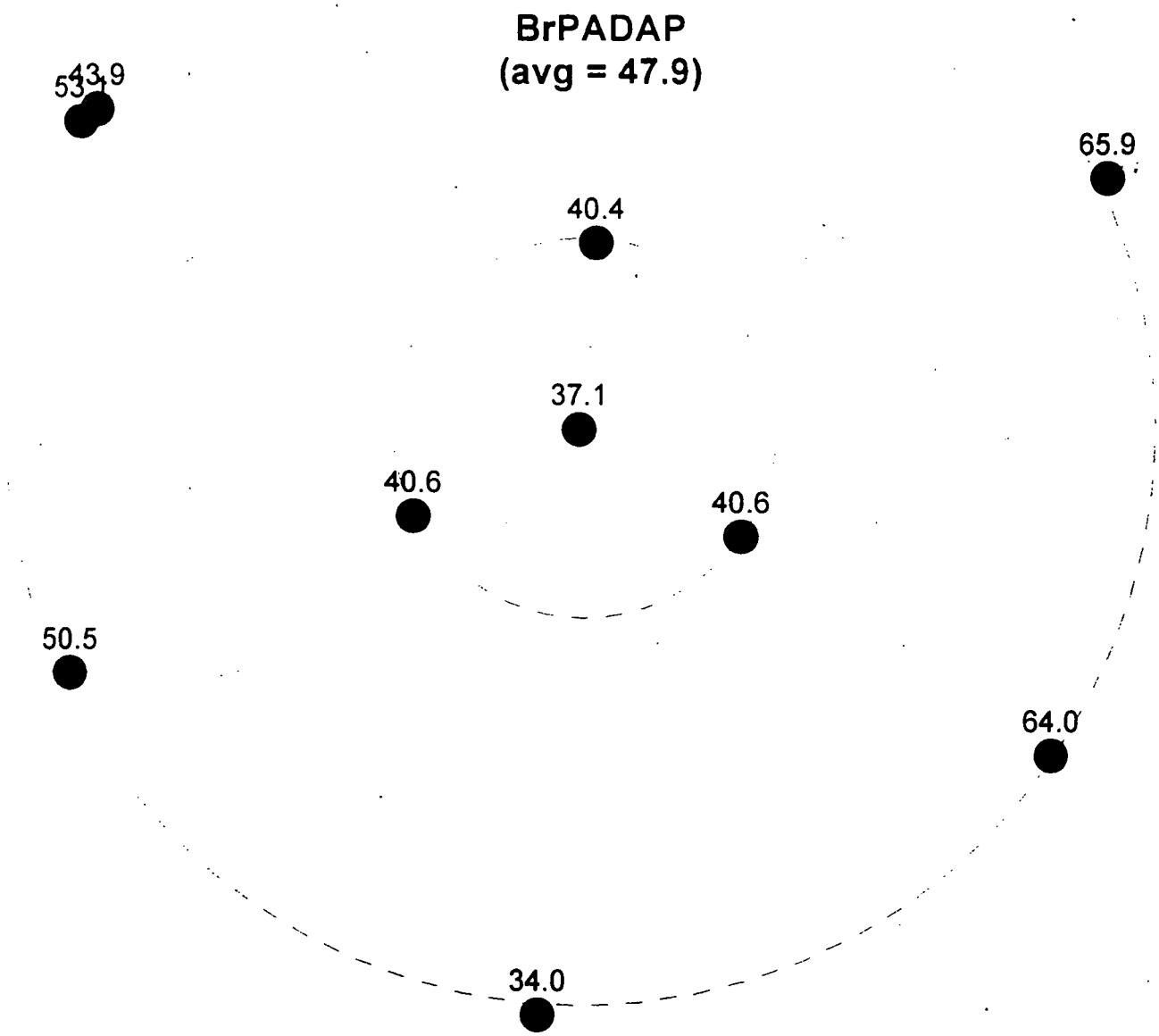
Dry wt ÷ (1 - %H2O) = Wet Wt

Comparability Study: HPGe Data

STATION	EASTING	NORTHING	Total U (ppm)	Det. Ht.
PBC1-1	1349440.62	483799.87	8.5	0.31
PBC1-1	1349440.62	483799.87	9.4	0.31
PBC1-1	1349440.62	483799.87	0.1	1
PBC1-1	1349440.62	483799.87	8.0	1
PBC2-1	1349396.32	482683.72	29.9	0.31
PBC2-1	1349396.32	482683.72	32.6	0.31
PBC2-1	1349396.32	482683.72	24.0	1
PBC2-1	1349396.32	482683.72	27.9	1
PBC3-1	1350430.20	481767.08	283.4	0.31
PBC3-1	1350430.20	481767.08	298.1	0.31
PBC3-1	1350430.20	481767.08	281.0	1
PBC3-1	1350430.20	481767.08	292.2	1
PBC4-1	1351652.76	480466.86	55.5	0.31
PBC4-1	1351652.76	480466.86	57.6	0.31
PBC4-1	1351652.76	480466.86	57.3	1
PBC4-1	1351652.76	480466.86	59.9	1
PBC5-1	1350968.78	480728.44	41.6	0.31
PBC5-1	1350968.78	480728.44	42.8	0.31
PBC5-1	1350968.78	480728.44	46.7	1
PBC5-1	1350968.78	480728.44	49.1	1
PBC6-1	1351667.82	480361.04	75.0	0.31
PBC6-1	1351667.82	480361.04	75.5	0.31
PBC6-1	1351667.82	480361.04	76.4	0.31
PBC6-1	1351667.82	480361.04	77.7	0.31
PBC6-1	1351667.82	480361.04	77.9	0.31
PBC6-1	1351667.82	480361.04	78.0	0.31
PBC6-1	1351667.82	480361.04	72.9	1
PBC6-1	1351667.82	480361.04	74.4	1
PBC6-1	1351667.82	480361.04	76.1	1
PBC6-1	1351667.82	480361.04	76.4	1
PBC6-1	1351667.82	480361.04	81.2	1
PBC7-1	1351611.96	480211.30	140.3	0.31
PBC7-1	1351611.96	480211.30	142.1	0.31
PBC7-1	1351611.96	480211.30	138.9	1
PBC7-1	1351611.96	480211.30	141.8	1
PBC8-1	1351286.27	476816.82	5.7	0.31
PBC8-1	1351286.27	476816.82	6.0	0.31
PBC8-1	1351286.27	476816.82	5.1	1
PBC8-1	1351286.27	476816.82	5.5	1
PBC9-1	1350550.20	476872.35	5.8	0.31
PBC9-1	1350550.20	476872.35	7.4	0.31
PBC9-1	1350550.20	476872.35	6.5	1
PBC9-1	1350550.20	476872.35	7.3	1
PBC10-1	1347673.91	478029.91	80.6	0.31
PBC10-1	1347673.91	478029.91	81.2	0.31
PBC10-1	1347673.91	478029.91	73.7	1
PBC10-1	1347673.91	478029.91	74.3	1

Det. Ht. = Detector Height (meters)

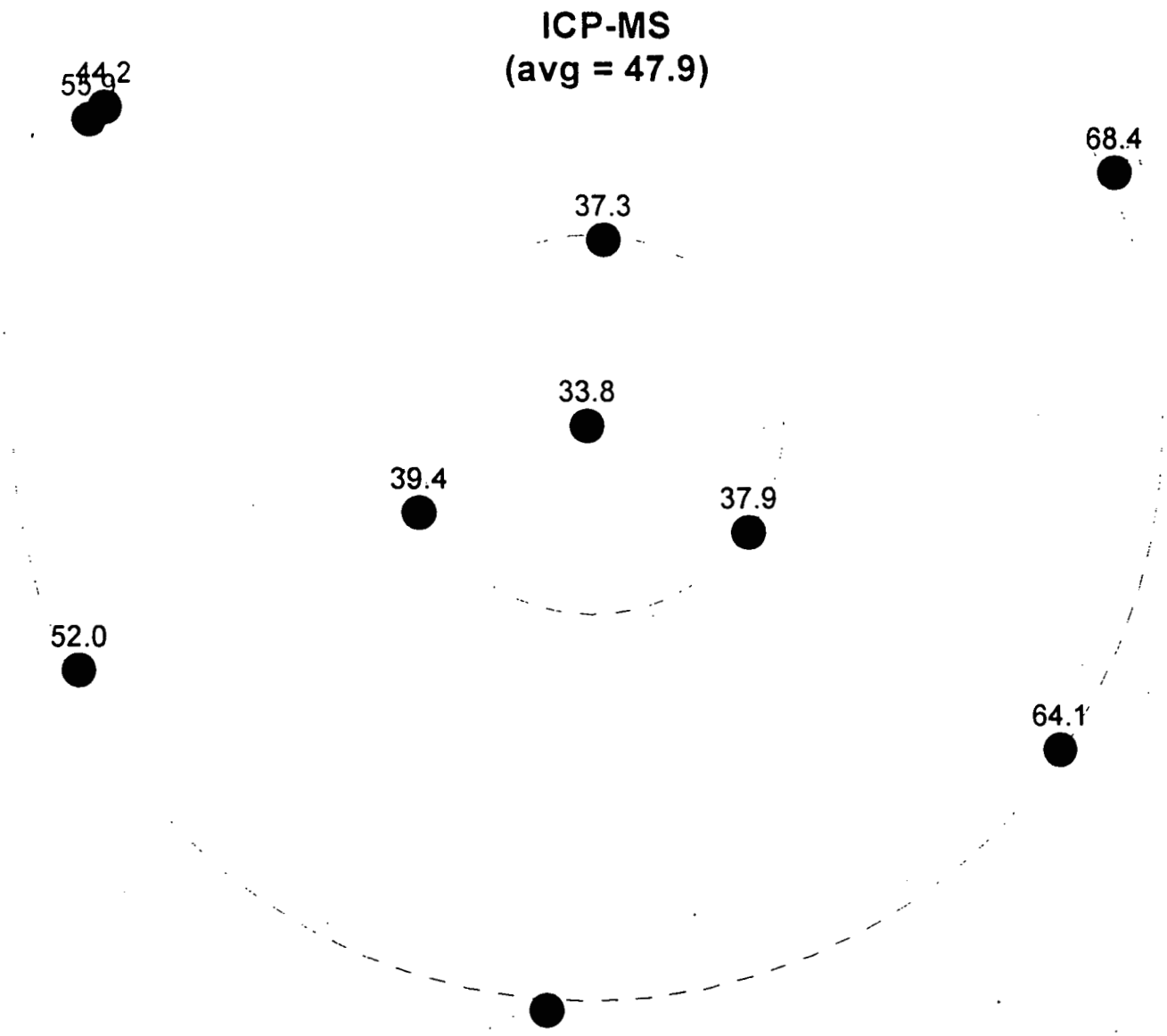
PBC 5 - Total Uranium



HPGe Measurements	
1 Foot	1 Meter
41.6	46.7
42.8	49.1

NOTES:
Results are reported in wet weight (ppm).
Outer circle is approximately 20 feet in diameter.
Inner circle is approximately 6 feet 8 inches in diameter.
There were two repeated HPGe measurements at each detector height.
Average was calculated as a non-spatially weighted average.
Duplicate readings (approximate same location) were averaged before overall average was calculated.

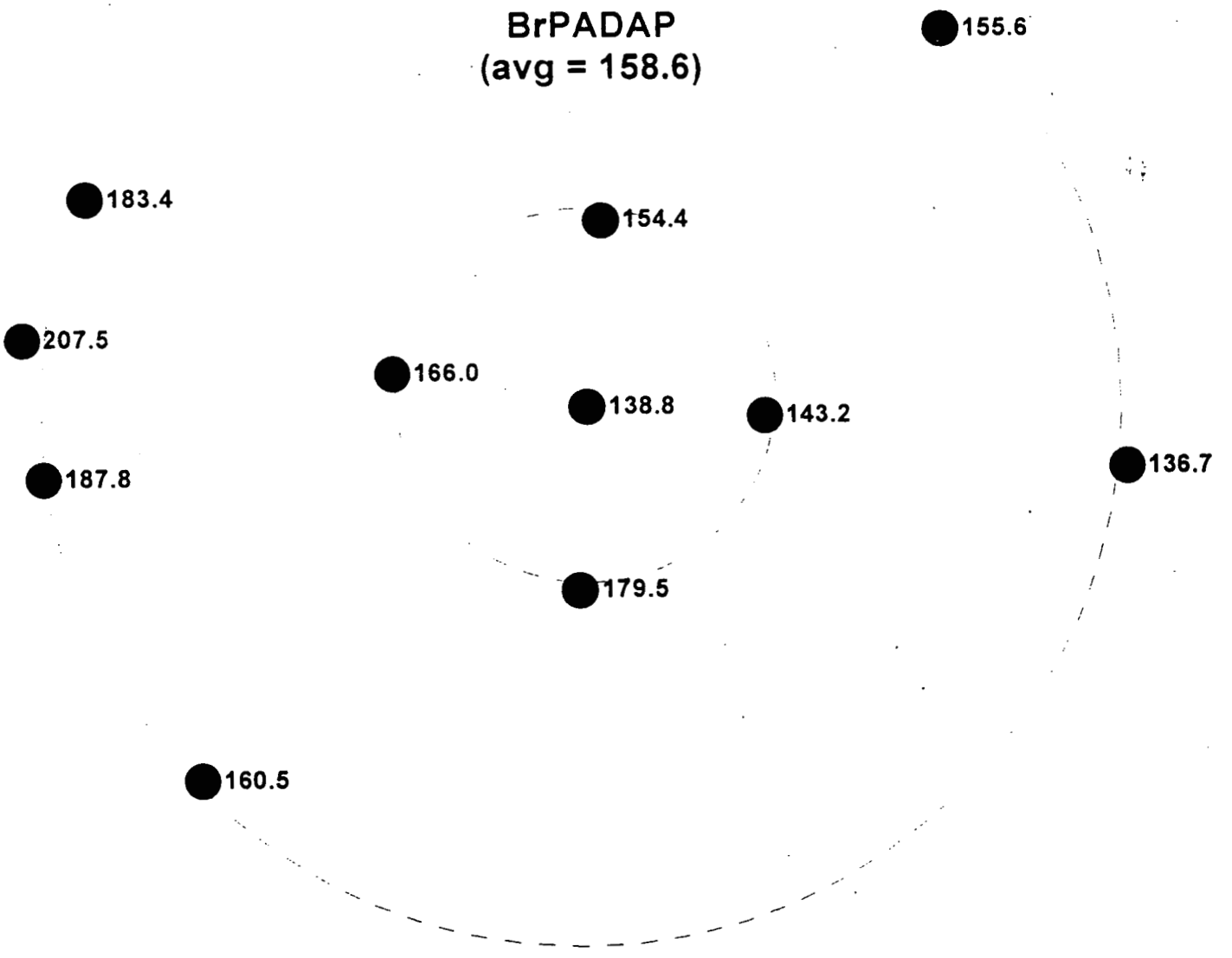
PBC 5 - Total Uranium



HPGe Measurements	
1 Foot	1 Meter
41.6	46.7
42.8	49.1

NOTES:
Results are reported in wet weight (ppm).
Outer circle is approximately 20 feet in diameter.
Inner circle is approximately 6 feet 8 inches in diameter.
There were two repeated HPGe measurements at each detector height.
Average was calculated as a non-spatially weighted average.
Duplicate readings (approximate same location) were averaged before overall average was calculated.

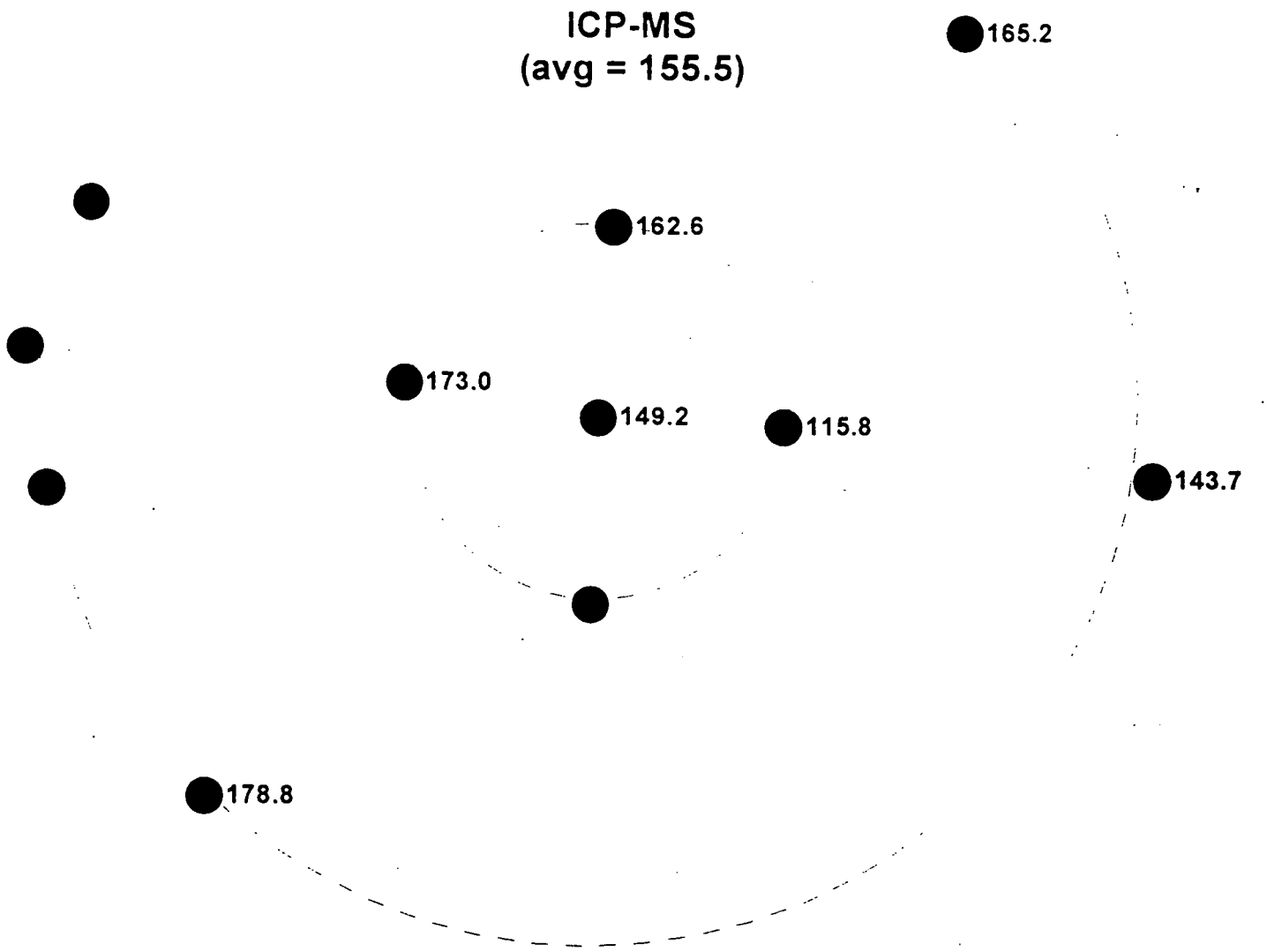
PBC 7 - Total Uranium



HPGe Measurements	
1 Foot	1 Meter
140.3	138.9
142.1	141.8

NOTES:
Results are reported in wet weight (ppm).
Outer circle is approximately 20 feet in diameter.
Inner circle is approximately 6 feet 8 inches in diameter.
There were two repeated HPGe measurements at each detector height.
Average was calculated as a non-spatially weighted average.
Duplicate readings (approximate same location) were averaged before overall average was calculated.

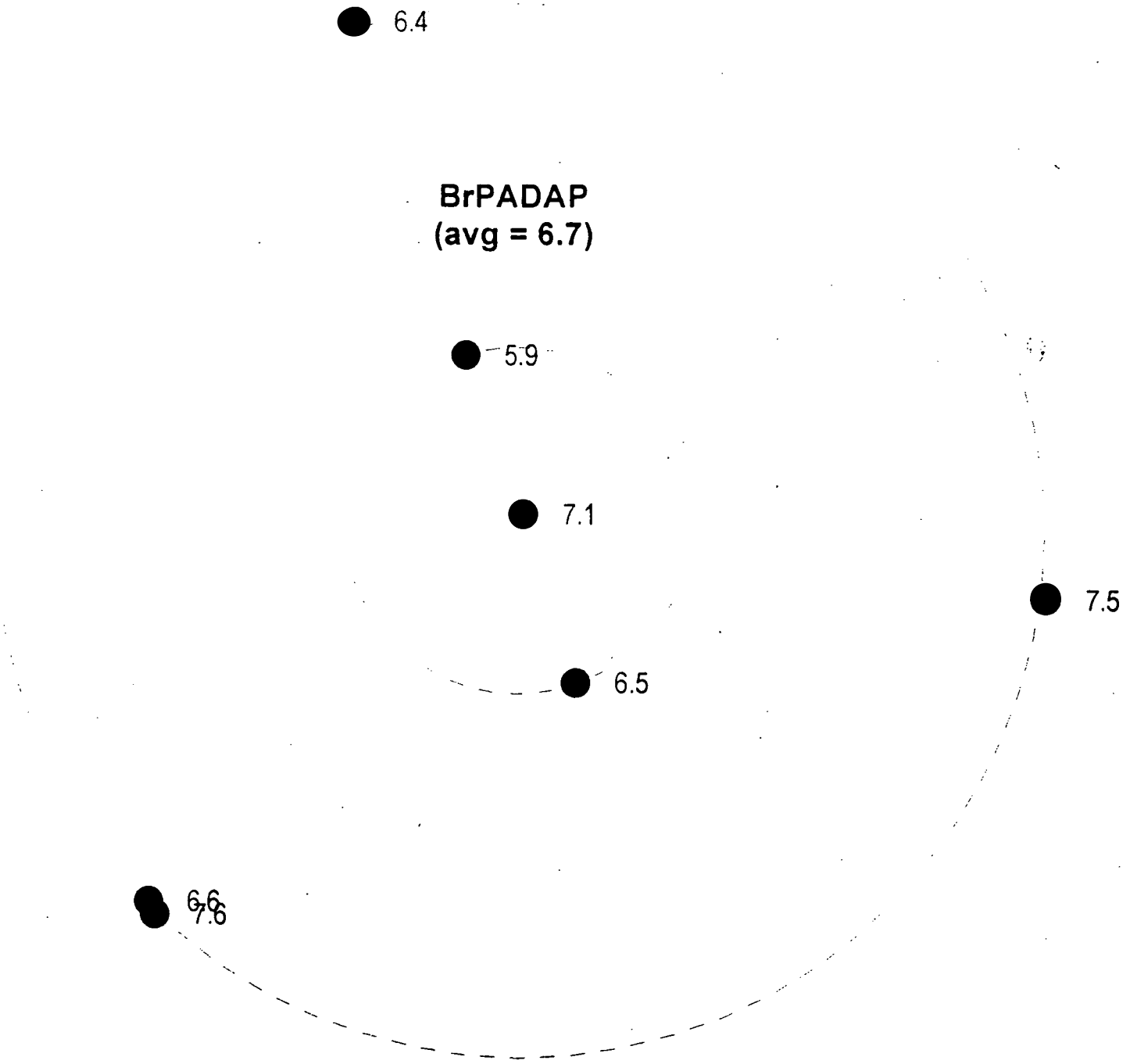
PBC 7 - Total Uranium



HPGe Measurements	
1 Foot	1 Meter
140.3	138.9
142.1	141.8

NOTES:
Results are reported in wet weight (ppm).
Outer circle is approximately 20 feet in diameter.
Inner circle is approximately 6 feet 8 inches in diameter.
There were two repeated HPGe measurements at each detector height.
Average was calculated as a non-spatially weighted average.
Duplicate readings (approximate same location) were averaged before overall average was calculated.

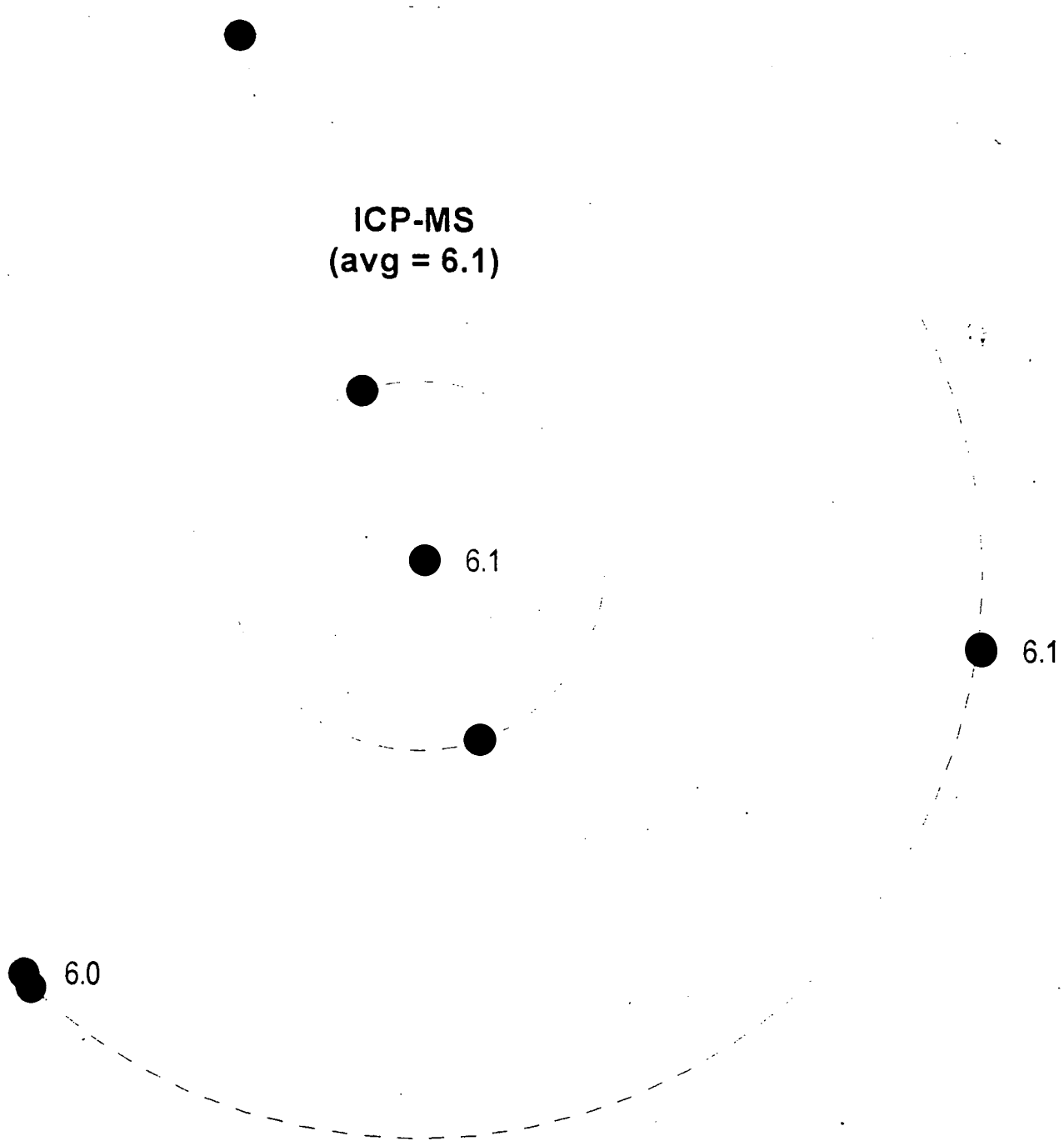
PBC 9 - Total Uranium



HPGe Measurements	
1 Foot	1 Meter
5.8	6.5
7.4	7.3

NOTES:
Results are reported in wet weight (ppm).
Outer circle is approximately 20 feet in diameter.
Inner circle is approximately 6 feet 8 inches in diameter.
There were two repeated HPGe measurements at each detector height.
Average was calculated as a non-spatially weighted average.
Duplicate readings (approximate same location) were averaged before overall average was calculated.

PBC 9 - Total Uranium



HPGe Measurements	
1 Foot	1 Meter
5.8	6.5
7.4	7.3

NOTES:
Results are reported in wet weight (ppm).
Outer circle is approximately 20 feet in diameter.
Inner circle is approximately 6 feet 8 inches in diameter.
There were two repeated HPGe measurements at each detector height.
Average was calculated as a non-spatially weighted average.
Duplicate readings (approximate same location) were averaged before overall average was calculated.

RTRAK Results from Comparability Study Areas: URANIUM

		Raw Counts - U-238 (cps)			Calculated U-238 (pCi/g)			Estimated Total Uranium (ppm)*		
PBC	N	Minimum	Maximum	Average	Minimum	Maximum	Average	Minimum	Maximum	Average
1-S	140	11.2	45.9	27.3	-21.1	48.0	4.3	-63.2	144.1	12.9
2-S	128	20.1	58.3	36.8	-34.2	42.0	4.4	-102.5	126.0	13.2
4-R	38	23.5	56.1	40.0	-19.3	55.7	20.7	-57.9	167.2	62.1
4-S	138	14.8	54.1	37.7	-32.3	53.4	13.5	-96.8	160.1	40.5
5-R	37	25.3	58.3	40.8	-10.7	50.8	20.5	-32.0	152.5	61.5
5-S	136	14.4	60.8	38.6	-24.7	76.2	18.3	-74.1	228.6	55.0
6-R	37	19.9	60.8	40.0	-8.9	55.3	20.3	-26.8	165.8	60.8
6-S	138	26.8	71.1	45.2	-17.8	71.9	26.7	-53.3	215.7	80.0
7-S	137	28.4	76.8	55.8	-4.0	91.2	40.9	-12.0	273.7	122.7
8-R	40	9.2	38.8	25.4	-19.3	49.1	10.4	-57.9	147.2	31.2
8-S	140	5.6	43.9	25.3	-40.5	46.0	3.4	-121.6	138.1	10.3
9-R	36	16.3	38.3	28.1	-44.2	36.5	3.4	-132.6	109.6	10.2
9-S	146	10.7	43.4	28.4	-25.3	44.4	5.5	-75.8	133.3	16.5
10C-S	121	129.3	200.5	163.0	-50.9	95.9	17.1	-152.6	287.6	51.3
10E-S	131	104.3	188.3	150.3	-83.0	93.2	5.7	-248.9	279.5	17.2
10W-S	121	416.7	554.6	488.9	-208.1	162.3	-7.5	-624.2	487.0	-22.4

NOTES:
PBC = Part B Comparability
S = Static or stationary read
R = Roving or mobile read
* = Total Uranium estimated as 3 times the U-238(pCi/g) result

COMPARABILITY STUDY: RTRAK - PBC 5

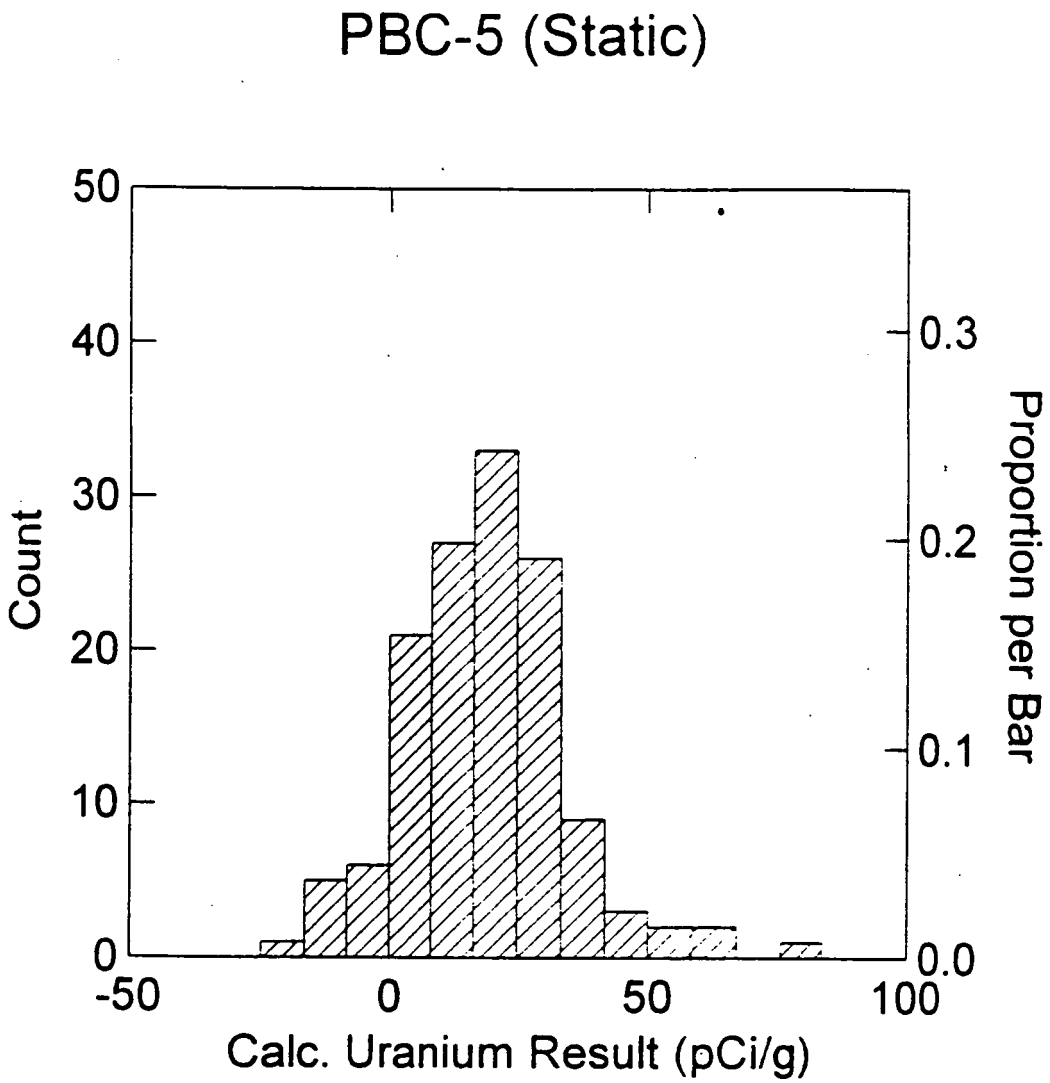
ROVING		Raw Counts - U-238 (cps)	Calculated U-238 (pCi/g)	Estimated Total Uranium (ppm)*
	Aquisitions	37	37	37
	Min	25.3	-10.68	-32.0
	Max	58.3	50.83	152.5
	Range	33.0	61.51	184.5
	Average	40.8	20.49	61.5
	Std. Dev.	7.2	14.05	42.1
On Mean:	Lower C.I.	38.1	15.32	45.9
(95 %)	Upper C.I.	43.5	25.67	77.0
Percetiles	Lower (.05)	29.5	-4.95	-14.9
	Upper (.95)	51.2	41.06	123.2
Quartiles	Lower (.25)	36.6	11.47	34.4
	Upper (.75)	45.9	30.28	90.8

STATIC		Raw Counts - U-238 (cps)	Calculated U-238 (pCi/g)	Estimated Total Uranium (ppm)*
	Aquisitions	136	136	136
	Min	14.4	-24.71	-74.1
	Max	60.8	76.20	228.6
	Range	46.4	100.91	302.7
	Average	38.6	18.34	55.0
	Std. Dev.	7.3	15.29	45.9
On Mean:	Lower C.I.	37.2	15.40	46.2
(95 %)	Upper C.I.	40.0	21.28	63.8
Percetiles	Lower (.05)	28.0	-7.48	-22.4
	Upper (.95)	50.6	42.63	127.9
Quartiles	Lower (.25)	34.4	8.60	25.8
	Upper (.75)	42.8	27.38	82.1

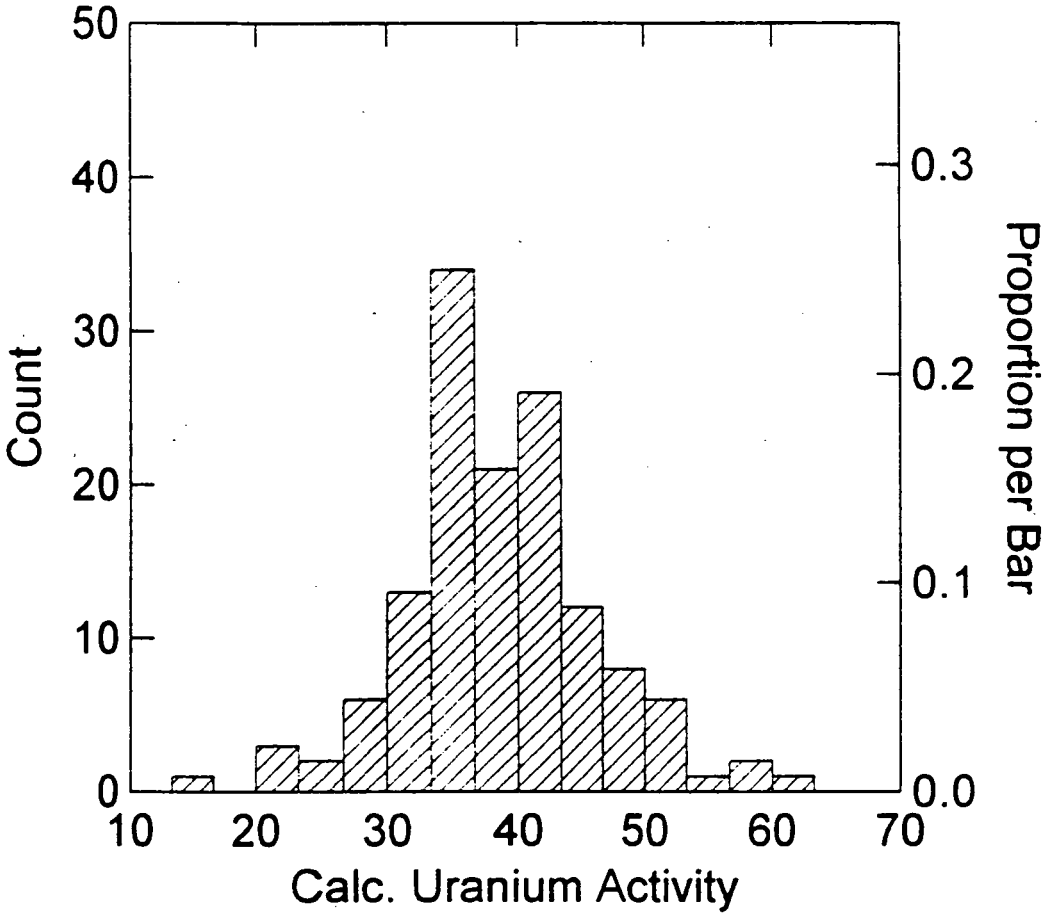
COMPARABILITY STUDY: RTRAK - PBC 9

ROVING		Raw Counts (cps)		Calculated Level (pCi/g)		Estimated Total Uranium (ppm)*
		Th-232	U-238	Th-232	U-238	
	Aquisitions	36	36	36	36	36
	Min	-1.5	16.3	-0.2	-44.2	-132.6
	Max	21.4	38.3	1.2	36.5	109.6
	Range	22.9	22.0	1.4	80.8	242.3
	Average	11.1	28.1	0.5	3.4	10.2
	Std. Dev.	4.4	5.0	0.3	15.0	44.9
On Mean:	Lower C.I.	6.1	15.4	0.3	0.8	2.5
(95 %)	Upper C.I.	16.0	40.7	0.8	6.0	17.9
Percetiles	Lower (.05)	2.2	19.0	0.0	-19.0	-57.1
	Upper (.95)	16.9	36.7	0.9	28.1	84.3
Percetiles	Lower (.25)	9.1	0.4	25.9	-4.8	-14.3
	Upper (.75)	13.8	0.7	31.1	11.7	35.0

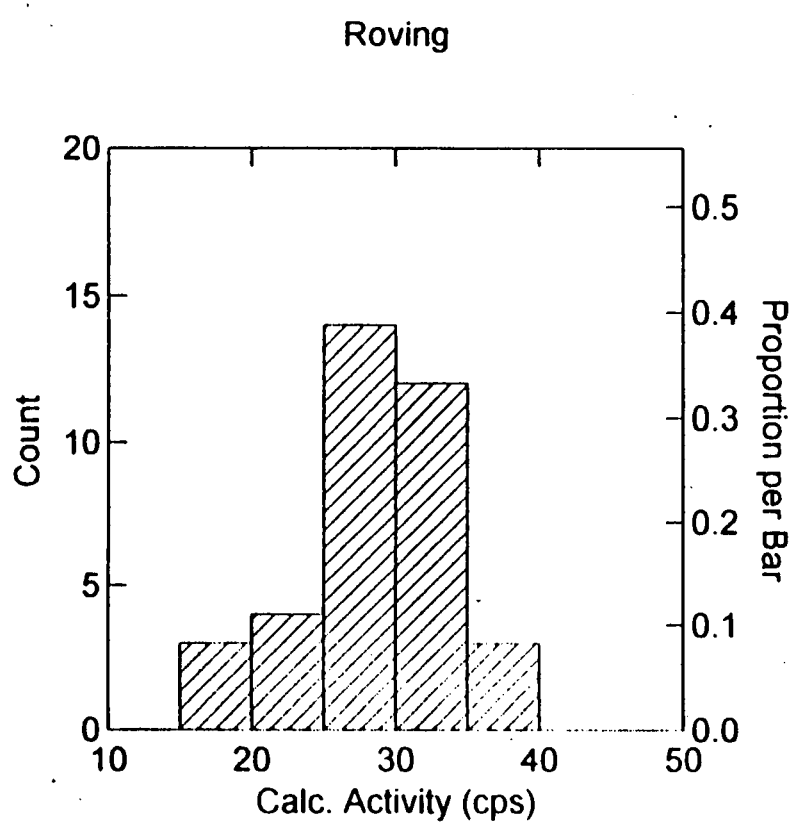
STATIC		Raw Counts (cps)		Calculated Level (pCi/g)		Estimated Total Uranium (ppm)*
		Th-232	U-238	Th-232	U-238	
	Aquisitions	146	146	146	146	146
	Min	-7.7	10.7	-0.6	-25.3	-75.8
	Max	21.4	43.4	1.2	44.4	133.3
	Range	29.1	32.7	1.8	69.7	209.2
	Average	10.5	28.4	0.5	5.5	16.5
	Std. Dev.	4.5	6.4	0.3	14.7	44.1
On Mean:	Lower C.I.	8.2	22.7	0.4	4.3	12.9
(95 %)	Upper C.I.	12.8	34.2	0.6	6.7	20.0
Percetiles	Lower (.05)	2.7	18.5	0.0	-16.5	-49.6
	Upper (.95)	16.7	37.8	0.9	28.6	85.7
Percetiles	Lower (.25)	8.3	0.4	23.5	-5.7	-17.1
	Upper (.75)	13.3	0.7	33.2	15.4	46.2

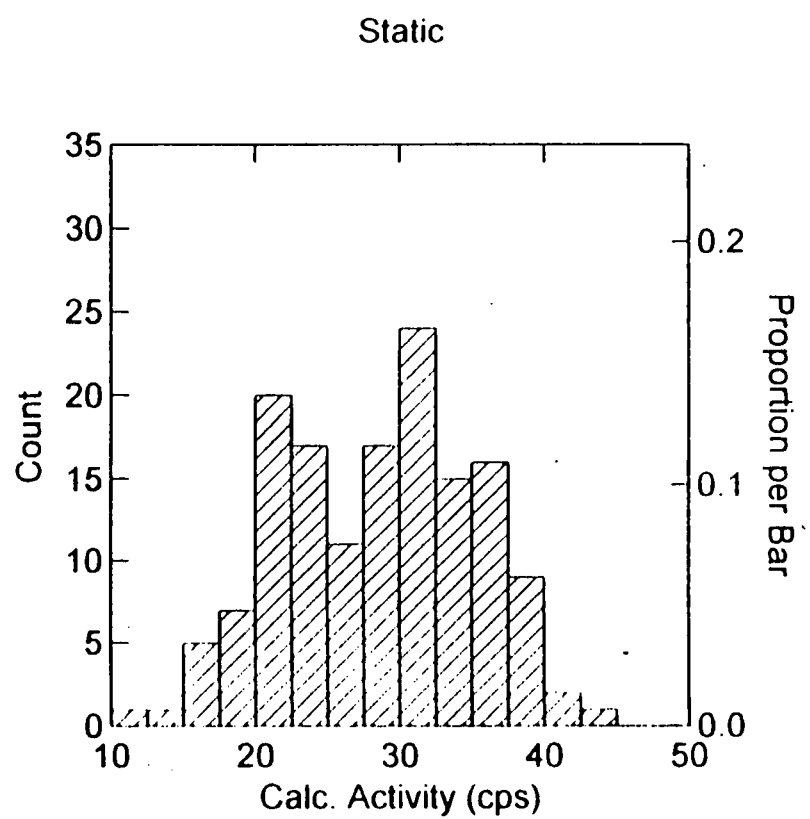

PBC-5 (Static)
Count
Proportion per Bar
Calc. Uranium Result (pCi/g)
50
40
30
20
10
0
0.3
0.2
0.1
0.0
-50
0
50
100

PBC-5 (Static)

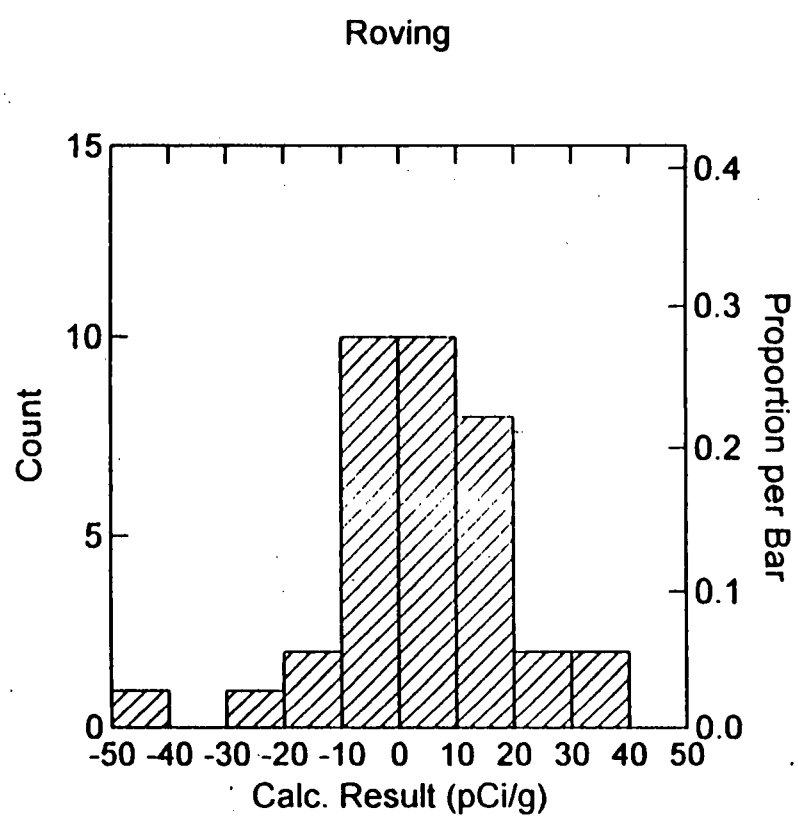

50
40
30
20
10
0
Count
0.3
0.2
0.1
0.0
Proportion per Bar
10
20
30
40
50
60
70
Calc. Uranium Activity

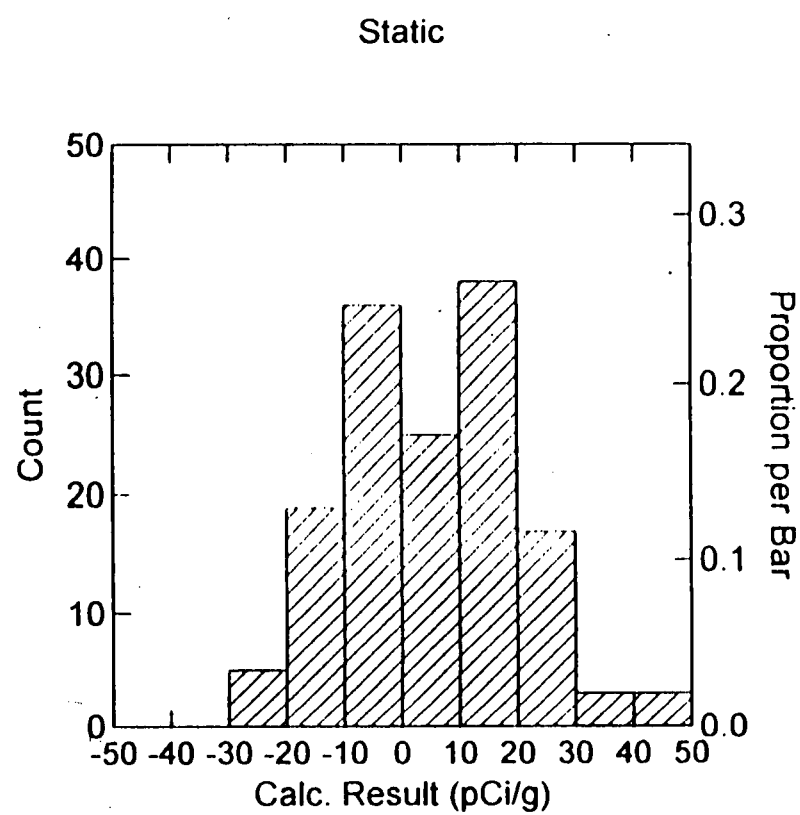
RTRAK - PBC-9


Roving
Count
Proportion per Bar
Calc. Activity (cps)
0
5
10
15
20
0.0
0.1
0.2
0.3
0.4
0.5
10
20
30
40
50


Static
Count
Proportion per Bar
Calc. Activity (cps)
0
5
10
15
20
25
30
35
0.0
0.1
0.2
10
20
30
40
50

RTRAK - PBC-9


Roving
Count
Proportion per Bar
Calc. Result (pCi/g)
0
5
10
15
0.0
0.1
0.2
0.3
0.4
-50 -40 -30 -20 -10 0 10 20 30 40 50


Static
Count
Proportion per Bar
Calc. Result (pCi/g)
0
10
20
30
40
50
0.0
0.1
0.2
0.3
-50 -40 -30 -20 -10 0 10 20 30 40 50

WAC Attainment Data: RT - 34

Method	Total U (ppm) (dry)	% Moisture	Total U (ppm) (wet)
BrPADAP	49	26.2	36.2
HPGe	- - -	- - -	52.1

RT-34 (within 15 ft.)		Raw Counts - U-238 (cps)	Calculated U-238 (pCi/g)	Estimated Total U (ppm)*
	Min	26.3	-12.7	-38.0
	Max	59.3	40.3	120.8
	Range	33.0	52.9	158.7
	Average	40.1	12.8	38.3
	Std. Dev.	7.4	16.4	49.3
On Mean:	Lower C.I.	36.4	4.5	13.5
(95 %)	Upper C.I.	43.8	21.0	63.0
Percetiles	Lower (.05)	29.2	-11.0	-33.0
	Upper (.95)	50.5	36.3	108.9
Percetiles	Lower (.25)	35.3	-2.6	-7.7
	Upper (.75)	45.0	27.5	82.5

WAC Attainment Data: RT - 26 / 28

Method	Total U (ppm) (dry)	% Moisture	Total U (ppm) (wet)	Station ID
BrPADAP	47	37.1	29.6	RT-28
	45	23.7	34.3	RT-26
HPGe	- - -	- - -	48.4	RT-28
	- - -	- - -	53.2	RT-26

RT - 26 / 28 (within 15 ft.)		Raw Counts - U-238 (cps)	Calculated U-238 (pCi/g)	Estimated Total U (ppm)*
	Min	30.7	-20.3	-60.9
	Max	68.2	54.9	164.8
	Range	37.5	75.2	225.7
	Average	47.4	20.0	60.1
	Std. Dev.	10.4	20.2	60.5
On Mean:	Lower C.I.	42.3	10.2	30.5
(95 %)	Upper C.I.	52.4	29.9	89.7
Percetiles	Lower (.05)	34.0	-10.0	-30.1
	Upper (.95)	64.6	54.3	162.9
Percetiles	Lower (.25)	39.7	7.5	22.6
	Upper (.75)	50.5	35.9	107.7

COMPARABILITY STUDY: PART B

IN SITU GAMMA vs PHYSICAL SAMPLES FOR FEMP SOIL CERTIFICATION

PURPOSE:

The Purpose of the FDF comparability Study (Part B) is to demonstrate certification of FEMP soil areas can be accomplished using either the in situ Gamma Spectrometry system or physical samples analyzed in the laboratory. Data obtained from the In situ system is to be statistically analyzed for comparability to laboratory data obtained from discrete physical samples.

R-TRAK

The R-TRAK is a mobile sodium iodide gamma detection system with associated data acquisition and software systems. The system has a dedicated global positioning link and acquires gamma data in specified intervals as it moves slowly over the certification area. R-TRAK data is downloaded into a mapping system for evaluation, and positioning if the HPGe. The measuring gamma spectrometry system uses principles and equipment similar to the laboratory system as described above. The R-Trak allows for complete coverage of a certification unit and identifies areas of relatively increased activity for gamma discernable radionuclides.

Field HPGe

The Field HPGe detection system is similar to laboratory systems described above and consists of a HPGe detector mounted on a tripod 1 meter above the test point. The HPGe is capable of detecting the radioactive materials below the FEMP FRL in the read circle approximately 30 feet in diameter. The HPGe Detection system has associated data acquisition system and software which converts counts to isotope activity capable of being downloaded into the site data base. Isotopes of uranium, thorium, radium, cesium, potassium, and other gamma discernable materials can be detected.

PHYSICAL SAMPLES:

For Part B of the Comparability Study physical samples were collected in 4 inch deep by 2 or 3 inch diameter cores, from areas within the area of coverage of the HPGe Detector readings. Samples submitted to the laboratory were dried, and ground to pass a 1 mm sieve and homogenized in a consistent manner prior to analysis.

The following analysis were preformed at the on site laboratories: % moisture, total uranium by BromoPADAP, uranium by ICP-MS, isotopic uranium, activities by gamma spectrophotometry, isotopic uranium, by alpha spectrophotometry for comparison to the in situ system. Isotopic thorium and Radium 226, & 228 will be analyzed on the physical samples, to determine if these isotopes have influenced the in situ measurements.

Summary of each of the analytical methods used in the comparability study is given below.

Total Uranium by BromoPADAP

The Uranium by BromoPADAP is a classical colorimetric procedure for the determination of uranium. In this procedure the sample is digested with acid to solubilize the uranium. Once solubilized the uranium is reacted with 2-(5-bromo-2-pyridylazo)-5-diethylaminophenol (BromoPADAP) to produce a colored complex. The color of the solution is proportional to the concentration of uranium present. A standard uranium solution is used for calibration of absorbance for this method. The analysis gives a total uranium result in that all uranium isotopes will be measured. Since Uranium 238 represents the most abundant uranium isotope the result most represents the mass of Uranium 238 in the sample. Uranium 235 which is 0.71% by mass in natural Uranium is the next most abundant uranium isotope.

Uranium, by ICP-MS

Uranium measured using Inductively Coupled Plasma - Mass Spectrometry (ICP-MS) is a relatively new application of a recently developed technology for the direct measurement of metals. This procedure is an adaptation of the SW-846 procedure 6020 Inductively Coupled Plasma - Mass Spectrometry. ICP-MS is a very sensitive and selective technique for the measurement of metals. Digested sample is aspirated into the plasma torch where ionization takes place. The ions are then guided through a vacuum chamber where they are separated by mass and detected. This technique will detect uranium isotopes which are present in sufficient concentration in the sample, and is calibrated using certified standards containing uranium. The technique is capable of detecting uranium 238, and 235 isotope masses. Total uranium is based derived by summing the individual uranium isotopic masses. In addition Thorium 232 and 230 can be analyzed via ICP-MS.

Isotopic Uranium measurements by Gamma Spectrometry

The technique of gamma spectrometry can be to accurately quantify the important uranium isotopes. Since gamma rays are highly penetrating radiation, samples can be analyzed by this technique with no chemical preparation other than homogenizing the samples. Samples are placed in a standardized geometry on the gamma detector and energy spectrum of the gamma emissions from the sample is collected in a device called a multichannel pulse height

analyzer. From peaks in the accumulated spectrum, the energy of the various gamma emissions detected indicates which isotopes are present and the number of gamma rays counted at a particular energy (IE. The area under the peak) is used to calculate the amount of that particular isotope that is present in the sample. U-238, and U-235 can be quantified in this manner, as can any other isotope that emits characteristic gamma rays. The accuracy of the method is reduced if a sample contains two or more radionuclides which emit characteristic gamma rays of within 2 or 3 Kev of one another. This is especially true if the activity of one of the interfering radionuclides is more than 5 times greater than that of the other nuclide. Also a high environmental background or presence of many radionuclides in a sample can elevate the minimal detectable activity for a given sample even though they are not analytes of concern. Gamma spectrometry is applicable for the analysis of isotopes of uranium, thorium, radium potassium 40, cesium 137 and other radionuclides.

Isotopic Uranium, Alpha Spectrometry

Like gamma rays, alpha emissions from radioactive nuclei are expelled with energies that are characteristic of the isotope emitting them. The technique of alpha spectrometry makes use of this property to identify and quantify the isotopes which are present. Since alpha particles are very weakly penetrating and can be absorbed by the sample material itself, a chemical sample preparation must be performed to separate the analyte of interest from the sample matrix. This involves a mineral acid digestion of the sample and separation of the element of interest from other elements using ion exchange resins. Extreme care must be exercised to prepare a uniform and very thin sample for counting in an alpha spectrometer. A known quantity of a radioactive isotope of the element being analyzed is added to the sample at the start of the chemical preparation process to provide a quantitative measure of the losses that occur during sample preparation. This radio tracer must be an isotope of the analyte element which is not present in the samples.

The alpha spectrometry technique offers double assurance that only the isotope of interest are being counted. First of all, the chemical preparation process is designed to separate the element of interest from all others, and then the spectrometric technique assures that you are counting only the desired isotopes. If the chemical separation is not complete for some reason, or the chemical losses are greater than normal, the sample spectrum will reveal evidence of these conditions. The chemical processing required for his technique is labor intensive and time consuming. While alpha spectrometry is highly selective and extremely sensitive, sample turnaround times are longer than other laboratory analysis techniques. It generally takes 4 to 5 days to complete the analysis of a batch of 20 soil samples. Alpha Spectrometry is applicable for the analysis of isotopic Uranium, Isotopic Thorium, Radium 226, 228, and others.

ISOTOPIC AND URANIUM ANALYSIS METHODS

ANALYSIS METHOD	ANALYTE	MATRIX	SAMPLE SIZE	SAMPLE PREP. METHOD	UPPER/LOWER DETECTION LIMITS	ANALYSIS TIME PER BATCH	METHOD STRENGTHS/WEAKNESSES
EDXRF	Total U & Th	Soil	20g	Crush & ball bill, then press into pellet	U: 11-320ppm Th: 18-128ppm	3 hours for 24 samples. Does not include sample prep time.	A: Rapid simultaneous analysis for U & Th. Fully automated. Minimal waste created. D: Calibration is matrix dependant. Soils must be matrix matched with calibration standards. Few certified standards available.
EDXRF	Qualitative scan for any element of atomic number greater than 10.	Any	Solids: varies from small dips or powders to as large as chamber will fit. Liquids: one drop to 10 ml	Separate into components if desired. Pour liquids or powders into sample cups. Mount powder on films (tapes)	For any given element, varies according to matrix. Generally from low ppm to 100% LDL near 1% for light elements.	Per sample: Major components - seconds Trace components - minutes Full identification in about 10 minutes	A: Can positively determine presence or absence of most elements in seconds. Few spectral interferences. Non-destructive. No reference standards needed. Can work with very small samples (< 1 gram). Elements detected can be quantified using an appropriate technique. Analyzes total amount of element present (isotopes are not differentiated). D: Cannot detect elements lighter than Na. Relatively high detection limits in liquids or for light elements (1-2%). Only the surface of a sample can be analyzed. (This can also be an advantage.) Significant extra work may be required to quantify detected elements! Not all combinations of elements can be confirmed present or absent. (Example: Sulfur cannot be determined when lead is also present.)

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ANALYSIS METHOD	ANALYTE	MATRIX	SAMPLE SIZE	SAMPLE PREP. METHOD	UPPER/LOWER DETECTION LIMITS	ANALYSIS TIME PER BATCH	METHOD STRENGTHS/WEAKNESSES
BrPADAP	Uranium	Soil	2.0 gram	$HNO_3/HClO_4$ to near dryness	1.0ppm-10,000 ppm	1 - 1.5 hours	Advantages/Disadvantages Sample analysis is automated and has a quick turn-around time (TAT). Sample prep is fast, usually 45 minutes for a batch of twenty samples. Detection limit 1ppm. High organic content in the sample may pose a problem when using HCLO4 acid. This may be averted by muffling the sample however this will slow the TAT. An organic waste stream is produced.
ICP-MS	Uranium	Soil	2.0 grams	$HNO_3/HClO_4$ to near dryness	1.00ppm to 1000 ppm	5-6 hours	Advantages/Disadvantages Low detection limits. More than one element can be scanned from the one sample digest. Uranium isotopic ratios can be determined from this digest.
Alpha Spec	U-234 U235/6 U-238	Solids eg. soil sediment	0.5 to 5g	Acid digest + ion exchange	LLD = 0.1pCl/g ULD = Infinite with dilution	4 days/20 samples	Strength Chemical separation plus spectrometric evidence when interferences present. Tracer to monitor losses. U-234 activity easier to measure than very small U-234 mass. Weakness Can't always clearly resolve U-235/6 or U-233/4. Labor intensive. Takes long time to complete analyses.

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ANALYSIS METHOD	ANALYTE	MATRIX	SAMPLE SIZE	SAMPLE PREP. METHOD	UPPER/LOWER DETECTION LIMITS	ANALYSIS TIME PER BATCH	METHOD STRENGTHS/WEAKNESSES
Alpha Spec	U-235 U-235/6 U-238	Water	0.2 to 1L	Acid digest + ion exchange	LLD = 0.2 pCi/L ULD = Infinite with dilution	4 days/20 samples	<u>Strength</u> Chemical separation plus spectrometric evidence when interferences present. Tracer to monitor losses. U-234 activity easier to measure than very small U-234 mass. <u>Weakness</u> Can't always clearly resolve U-235/6 or U-233/4. Labor intensive. Takes long time to complete analyses.
High resolution gamma spec (HPGe)	U-235 U-238	Solids eg. soil sediment	50-500g	Dry, grind, homogenize	U-238 LLD ~3 pCi/g U-235 LLD ~0.5 pCi/g ULD - Infinite with dilution	2 days/20 samples	<u>Strengths</u> Spectrometric technique gives positive ID of isotopes present in sample, minimal sample prep. Rapid turn around. Can attain lower MDC with longer count, larger sample. <u>Weakness</u> Technique uses U-238 daughter gamma ray. Presence of Th in a sample elevates MDC.

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