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Risø-R-1467(EN)

An Intercomparison on Radionuclides in Environmental Samples

Baltic-Danish Co-operation Project on
Radiation Protection 2001-2003

Sven P. Nielsen

Risø National Laboratory
Roskilde
Denmark
July 2004

Risø-R-Report

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Introduction

The Danish Emergency Management Agency and Risø National Laboratory agreed in 2001 on a project covering co-operation with Poland, Estonia, Latvia and Lithuania in the field of radiation protection with emphasis on environmental monitoring and radioecological studies (Baltic-Danish Co-operation Project on Radiation Protection, 2001-2003). In connection with this co-operation, Risø National Laboratory organised an intercomparison exercise on laboratory analyses of radionuclides in environmental samples.

Participants

Fifteen organisations from Germany, Denmark, Estonia, Finland, Lithuania, Latvia and Poland participated in the intercomparison as listed in Table 1. Each organisation participated with one laboratory except for one organisation, which participated with two laboratories.

Table 1. Organisations participating in the intercomparison exercise.

Institute	Country
Federal Maritime and Hydrographic Agency, Hamburg	DE
Risø National Laboratory, Roskilde	DK
Estonian Radiation Protection Centre, Tallinn	EE
Institute of Physics, University of Tartu, Tartu	EE
Radiation and Nuclear Safety Authority, STUK, Helsinki	FI
Ignalina Nuclear Power Plant, Visaginas	LT
Institute of Physics, Vilnius	LT
Ministry of Environment, Radiological Laboratory of Joint Research Centre, Vilnius	LT
Nuclear Hydrophysics Laboratory, Vilnius Gediminas Technical University	LT
Radiation Protection Centre, Vilnius	LT
Latvian Environment Agency, Laboratory Department, Jurmala	LV
Radiation Safety Centre, Riga	LV
State Limited Liability Company "RAPA", Salaspils	LV
Central Laboratory for Radiological Protection Warsaw	PL
Institute of Meteorology and Water Mangement, Maritime Branch, Gdynia	PL

Sample Materials

The sample types involved seawater, seaweed, lake water, soil and dry milk. Sample materials of dry milk, soil, seaweed and seawater were collected from the Danish environment. The lake water was obtained from a location in Sweden with Chernobyl fallout significantly higher than in Denmark.

The seaweed and soil materials were dried and mixed thoroughly. A number of aliquots were selected from the different materials and analysed by gamma spectrometry to test the homogeneity. For all sample types the gamma emitting radionuclides detected were homogeneously distributed within the statistical counting uncertainty, which ranged from 1 to 6%.

Samples were distributed to the participants in spring 2003 as specified in Table 2, which also lists the radionuclides suggested for analysis. Results of laboratory analyses were received by June 2003.

Table 2. Samples distributed for the intercomparison exercise.

Sample types	Sample amounts	Radionuclides suggested for analysis
Seawater	50 L	^{137}Cs , ^{90}Sr , ^{99}Tc
Lake water	25 L	^{137}Cs , ^{90}Sr
Soil	0.2 kg	^{137}Cs , ^{90}Sr , ^{232}Th -series, ^{238}U -series, ^{40}K
Dry milk	1.8 kg	^{137}Cs , ^{90}Sr , ^{40}K
Seaweed	0.3 kg	^{137}Cs , ^{90}Sr , ^{99}Tc , ^{40}K

Analytical Results

All laboratories receiving samples except for one submitted analytical results. Results were received on more than 10 different radionuclides. For 8 radionuclides (^{137}Cs , ^{90}Sr , ^{60}Co , $^{239,240}\text{Pu}$, ^{241}Am , ^{226}Ra , ^{232}Th and ^{40}K) results were received from two or more participants per nuclide and sample type and this was considered sufficient for the intercomparison purpose. Each participant was assigned a code number to ensure anonymity. The analytical results and uncertainties are shown in the Tables 3-7, which also give the median values of the radionuclide concentrations for each sample. The results are furthermore shown in column charts by radionuclide and sample type in Appendix A with error bars showing reported uncertainties at one standard deviation.

Table 3. Results reported on radionuclides in dry milk.

Lab No.	^{137}Cs		^{90}Sr		^{40}K	
	Bq/kg	1 SD	Bq/kg	1 SD	Bq/kg	1 SD
1			0.41	0.025		
2	0.38	0.08			357	20
3	0.35	0.02	0.26	0.03	400	40
4	0.35	0.07	0.40	0.26	441.5	82.8
5	0.36	0.05	0.31	0.09	370	8
6			0.38	0.06		
7	0.35	0.06	0.16	0.01	351	62
13	0.88	0.20			443.4	2.6
14	0.36	0.054	0.27	0.014	370	22.2
16	0.47	0.06			423.2	11.8
17	0.434	0.071			461	15
Median	0.36		0.31		400	

Table 4. Results reported on radionuclides in soil.

Lab No.	¹³⁷ Cs		⁹⁰ Sr		⁴⁰ K		²²⁶ Ra		²³² Th		^{239,240} Pu		²⁴¹ Am	
	Bq/kg	1 SD	Bq/kg	1 SD	Bq/kg	1 SD	Bq/kg	1 SD	Bq/kg	1 SD	Bq/kg	1 SD	Bq/kg	1 SD
1			2.3	0.16										
2	13.0	0.9			436	25	16.8	0.9	17.5	1.8				
3	13.2	0.8	1.28	0.13	454	45	17.5	1.8	18.2	1.8	0.22	0.03	0.082	0.01
4	10.9	1.2	1.57	0.69	374	64								
5	13.1	0.9	1.29	0.21	455	23	17.3	1.3	17.2	1.5				
6	13.4	1.2	1.39	0.17	566	33								
7	12.1	1.3	0.97	0.10	428	55	35	7	16.1	1.6				
10	11.2	0.3			384	23	13.3	0.9	13.8	0.8				
11	12.6	1.0	1.6	0.3							0.16	0.03	0.10	0.03
12	13.1	0.6												
13	16.6	0.93			750.9	10.5								
14	13.1	0.52			440	22	16.3	1.63	17.4	1.74				
16	13.0	0.27			483	21.5	16.7	0.66	14.1	0.58				
17	12.6	0.65			407	13								
Me-dian	13.0		1.39		440		16.8		17.2		0.19		0.091	

Table 5. Results reported on radionuclides in seaweed.

Lab No.	¹³⁷ Cs		⁹⁰ Sr		⁴⁰ K		⁶⁰ Co		²²⁶ Ra		²³² Th	
	Bq/kg	1 SD	Bq/kg	1 SD	Bq/kg	1 SD	Bq/kg	1 SD	Bq/kg	1 SD	Bq/kg	1 SD
1			2.4	0.17								
2	7.6	1.0			917	22			8.1	1.0	10.4	1.9
3	7.37	0.37	1.36	0.14	990	99	0.46	0.05	7.8	0.8	10.8	1.1
4	5.73	0.83	1.56	0.69	657	115.3						
5	8.09	0.40	0.95	0.16	983	32						
6	9.45	1.70	5.65	0.55								
7	7.0	1.0			960	113						
10	6.1	0.2			848	51			8	0.6	7.3	0.4
11	6.9	0.6	1.3	0.3								
12	7.8	0.4										
13	8.4	0.6			862	50			5.2	0.6	9.44	0.4
14	7.40	0.37	1.43	0.11	880	44						
15	7.00	0.15			856	5.4	0.50	0.15				
17	7.45	0.45			935	30						
Median	7.43		1.50		899		0.48		7.9		9.9	

Table 6. Results reported on radionuclides in seawater.

Lab No.	¹³⁷ Cs		⁹⁰ Sr	
	Bq/m ³	1 SD	Bq/m ³	1 SD
1	52	2	12.6	0.50
3	55.4	2.8	12.2	1.2
4	71	25	16.8	14.0
5	67.4	2.8		
6	76.5	5.0	10.6	1.7
7	56.0	7.0	2	0.2
9	57.4	0.6	12.1	0.6
11	59.4	5.3	10.5	2.6
12	50	5	17	3
13	53.99	4.00		
15	58.2	0.94	11.9	0.27
16	58	0.64		
17	52	3.4		
Median	56.7		12.2	

Table 7. Results reported on radionuclides in lake water.

Lab No.	¹³⁷ Cs		⁹⁰ Sr	
	Bq/m ³	1 SD	Bq/m ³	1 SD
1	147	4	17.8	0.7
3	169	8	14.9	1.5
4	150	30	17	14
5	90.2	3.1	9.18	0.35
6	249	13	18	2
7	110	10	12	1
9	167.3	1.8	44.9	3.3
11	163	14.6	12.5	3.2
12	164	16	25	4
13	124.2	2.5		
15	156.7	1.8	15.3	0.35
16	159.7	6.5	11.1	0.27
17	156.9	10.4		
Median	157		16.2	

Evaluation

The evaluation procedure involves a comparison of the result x from each participant (radionuclide activity concentration) for a single radionuclide and sample type with the median of the results x_m across laboratories. From this a z-score value is calculated according to $z = (x - x_m) / \sigma$, where σ is a target standard deviation, for which a value of 10% has been assumed throughout for this intercomparison. Values of z-

scores are combined across sample types into rescaled sums of z-scores according to $RSZ = (\Sigma z)/n^{1/2}$, where n is the number of sample types. The RSZ-value is an indicator of analytical bias for the radionuclide in question and used for a test in the normal distribution. When tested at a 99% level of significance, values of RSZ lower than -2.6 indicate analytical bias on the low side exceeding the target standard deviation of 10% and RSZ values higher than 2.6 indicate analytical bias on the high side larger than 10%.

Furthermore, values of z-scores are combined across sample types into sums of squares of z-scores according to $SSZ = \Sigma z^2$. The SSZ-value is an indicator of analytical accuracy and used for a test in the chi-square distribution. If tested at the 99% significance level and in case that a participant has submitted results on concentrations of a certain radionuclide in 4 sample types, SSZ values in the range from 0.2 to 14.9 indicate that the analytical accuracy is consistent with the assumption of an analytical uncertainty corresponding to a 10% standard deviation. If the SSZ-value is lower than 0.2, this indicates that the accuracy corresponds to an analytical uncertainty smaller than 10%. Correspondingly, if the SSZ-value is higher than 14.9, this indicates that the accuracy corresponds to an analytical uncertainty, which is greater than 10%.

The results of the tests are summarised by radionuclide across sample types in Table 8 listing the number of participants submitting analytical results, the number of those whose performance matches the target standard deviation or better, and the number of those who do not. The tests were made at the 99% significance level.

Table 8. Summary of the performance tests for the participants by radionuclide across sample types.

Radionuclide	Number of participants submitting results	Number of participants for which the results passed the tests	Number of participants for which the results did not pass the tests
¹³⁷ Cs	16	10	6
⁹⁰ Sr	12	5	7
⁶⁰ Co	2	2	0
^{239,240} Pu	2	2	0
²⁴¹ Am	2	2	0
²²⁶ Ra	8	6	2
²³² Th	7	7	0
⁴⁰ K	12	10	2
Radionuclides combined	16	6	10

The detailed results of the evaluation are shown in Appendix B, which lists the test parameters and significance levels for the radionuclides ¹³⁷Cs, ⁹⁰Sr, ⁶⁰Co, ^{239,240}Pu, ²⁴¹Am, ²²⁶Ra, ²³²Th and ⁴⁰K across sample types for each laboratory. In addition graphs show column charts for each radionuclide of the sums of z-scores for each participant with information on contributions from each sample type. Test parameters and significance levels are also given for all radionuclides combined.

Conclusions

Sixteen laboratories participated in an intercomparison exercise carried out in 2003 on laboratory analyses of radionuclides in environmental samples. The sample types included seawater, lake water, soil, dry milk and seaweed and the exercise involved the radionuclides ^{137}Cs , ^{90}Sr , ^{60}Co , $^{239,240}\text{Pu}$, ^{241}Am , ^{226}Ra , ^{232}Th and ^{40}K .

The evaluation of analytical performance was based on comparison with median values, a 10% target standard deviation and statistical tests at the 99% level. For ^{137}Cs the results from 10 out of 16 laboratories passed the evaluation tests. For ^{90}Sr the results from 5 out of 12 laboratories passed the evaluation tests. For ^{60}Co , $^{239,240}\text{Pu}$ and ^{241}Am two laboratories submitted results and both passed the tests. For the natural radionuclides ^{226}Ra , ^{232}Th and ^{40}K , only a few laboratories did not pass the tests. For all radionuclides combined, the results from 6 out of 16 laboratories passed the evaluation tests. The results indicate that for several of the laboratories there is room to improve the analytical quality on radionuclides in environmental samples to match an uncertainty corresponding to a relative standard deviation of 10%.

It is important, however, to recognise two subjective components of the evaluation:

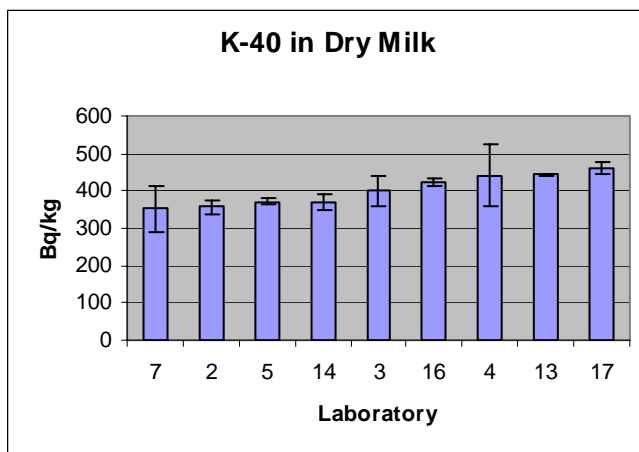
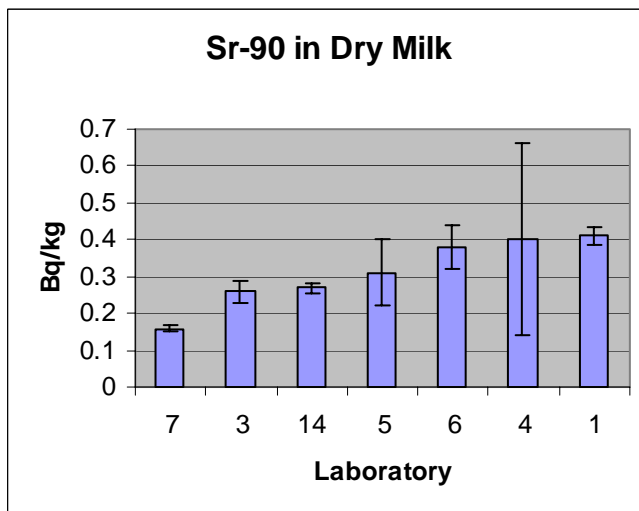
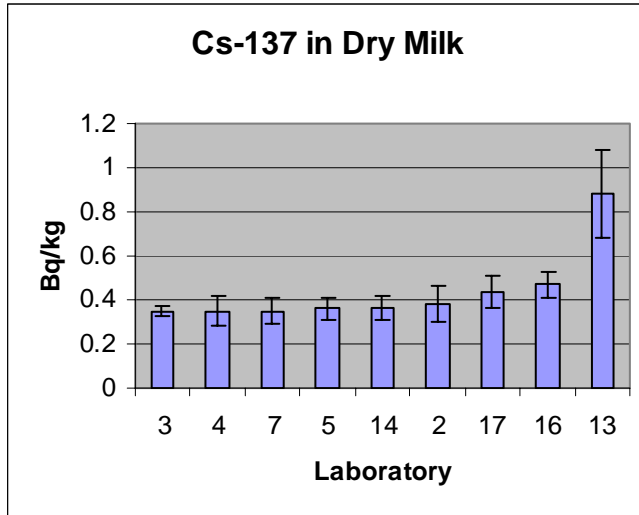
- the choice of a target standard deviation of 10%
- the choice of using median values to represent the true values.

Acknowledgement

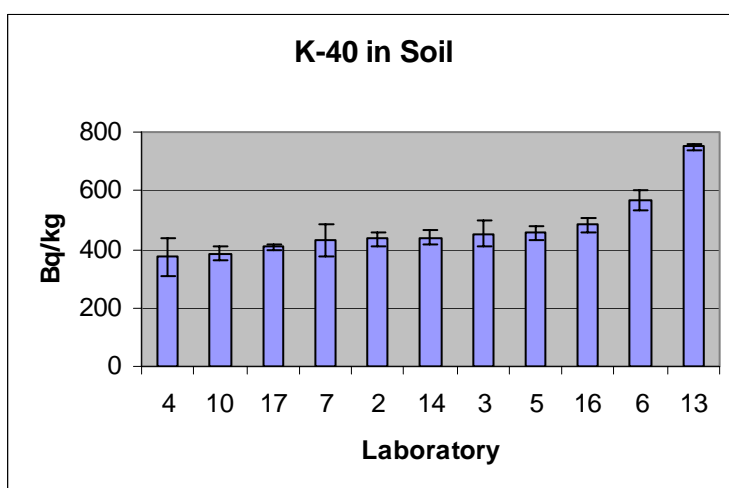
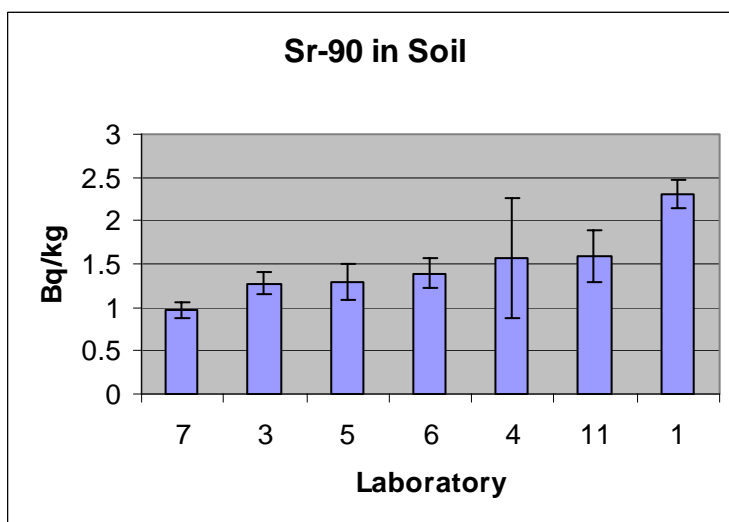
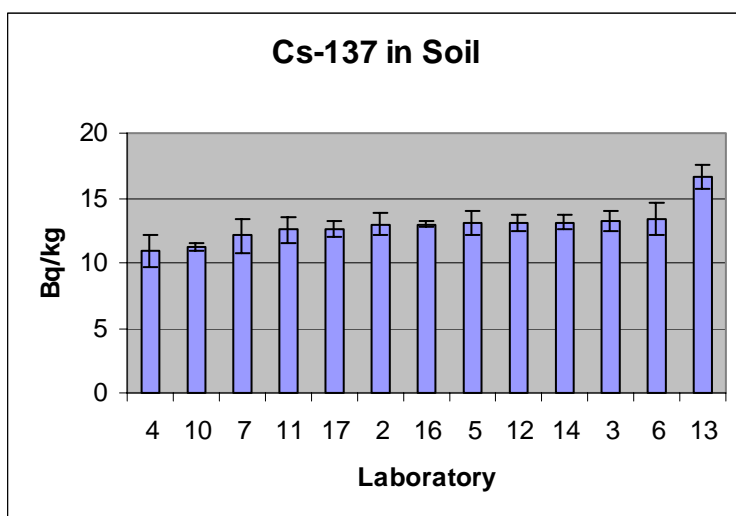
This work was sponsored by the Danish Emergency Management Agency.

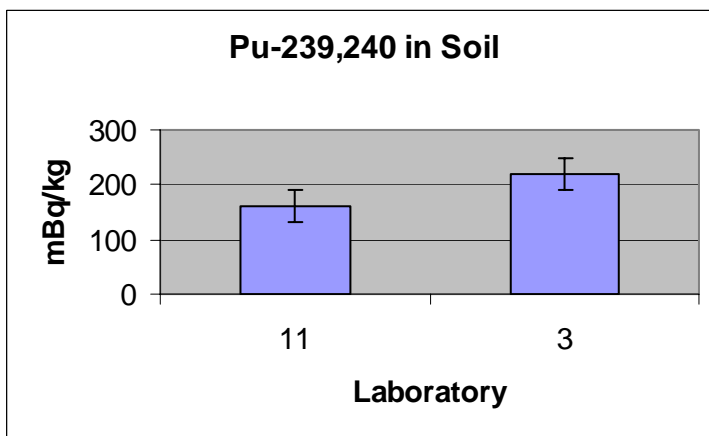
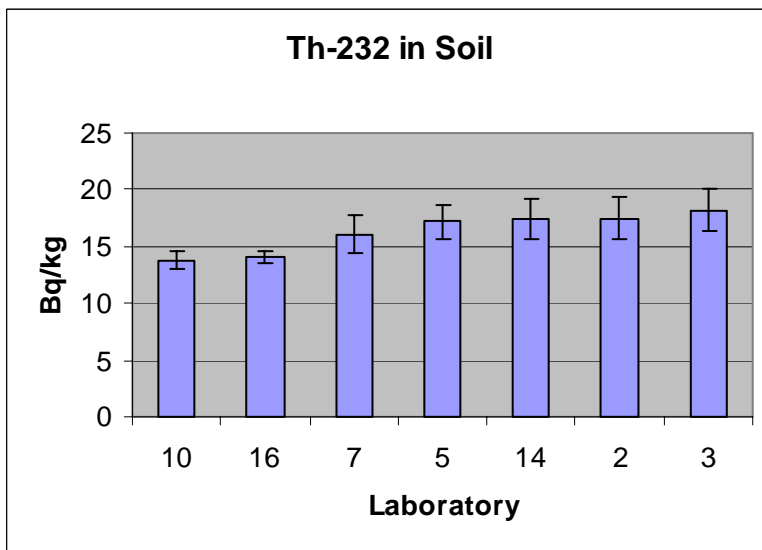
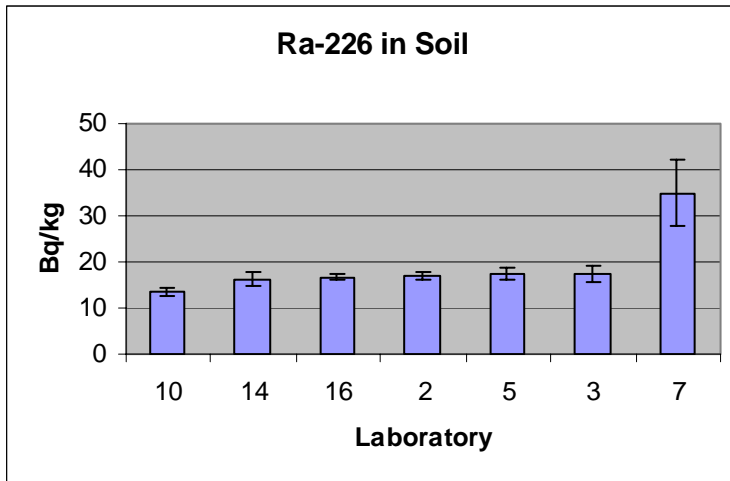
Appendix A. Graphical presentation of analytical results

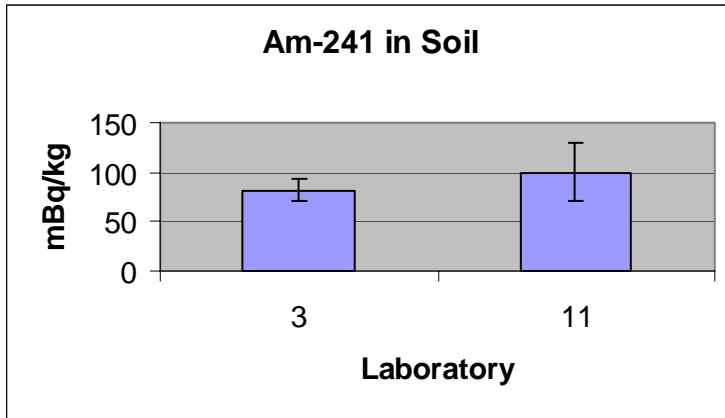
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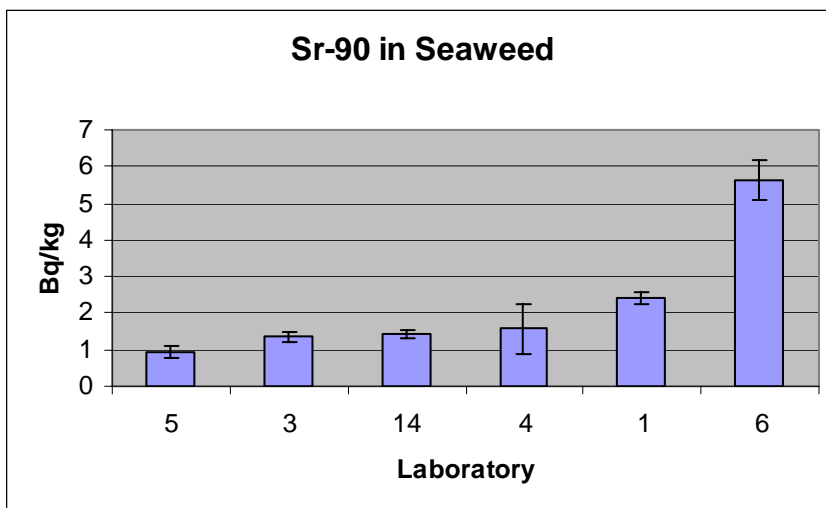
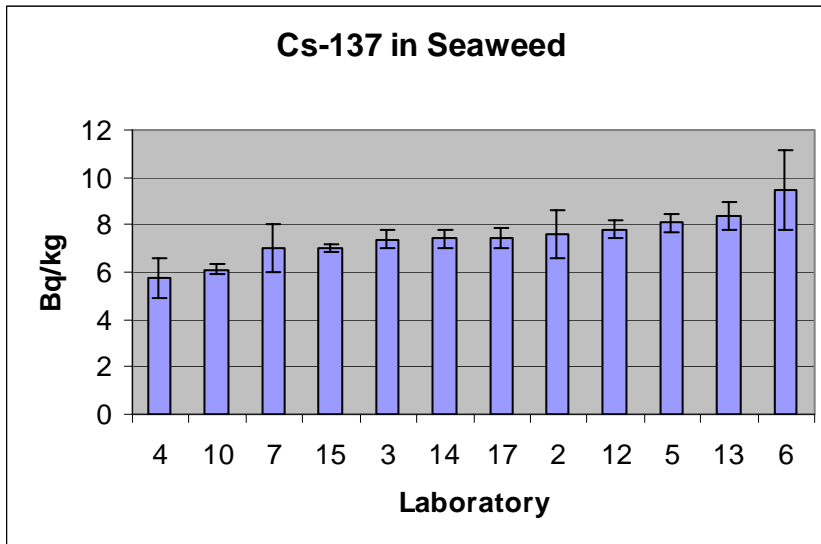
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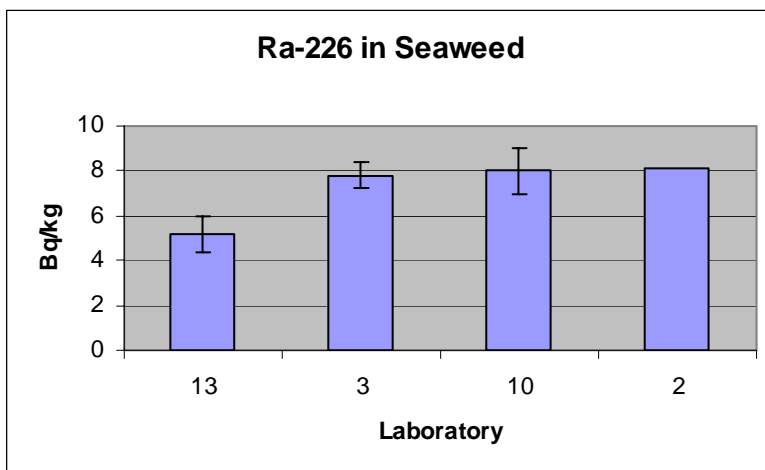
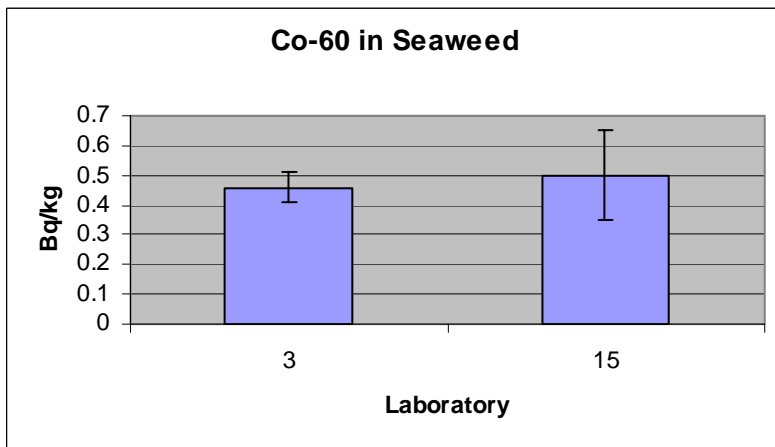
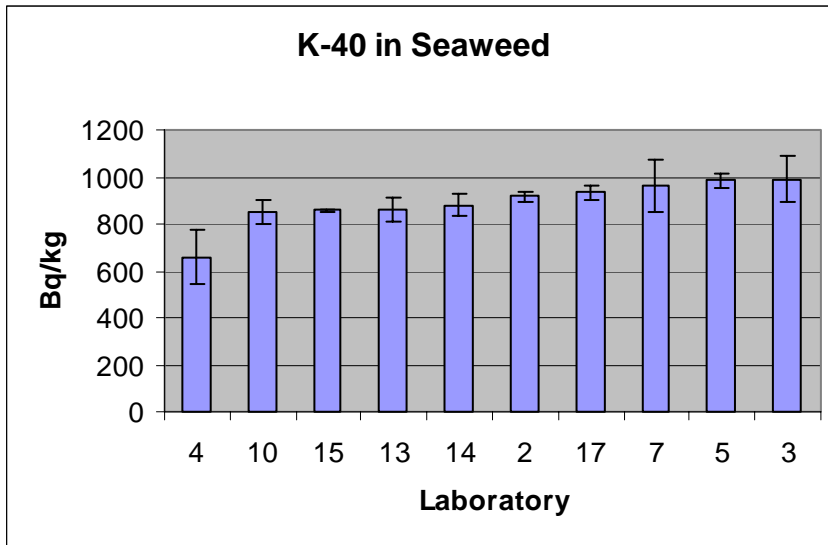


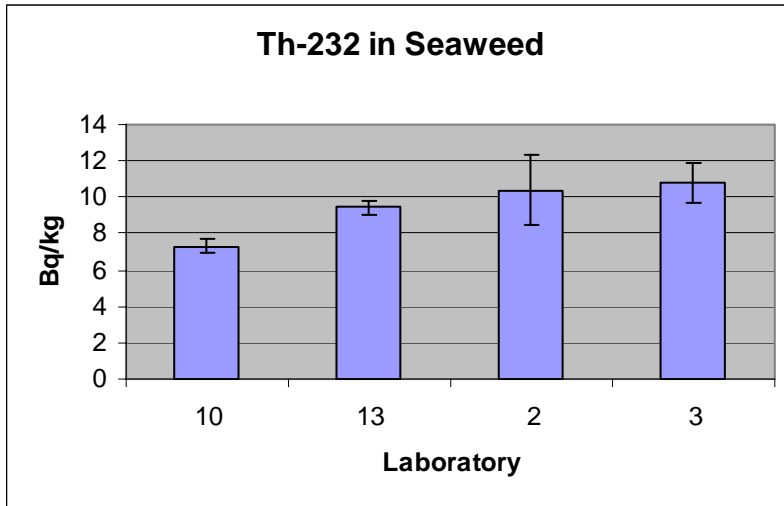




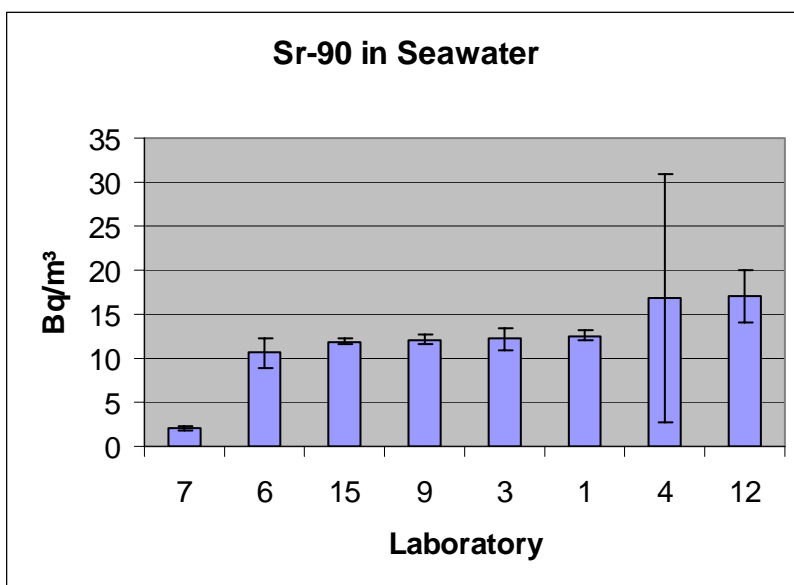
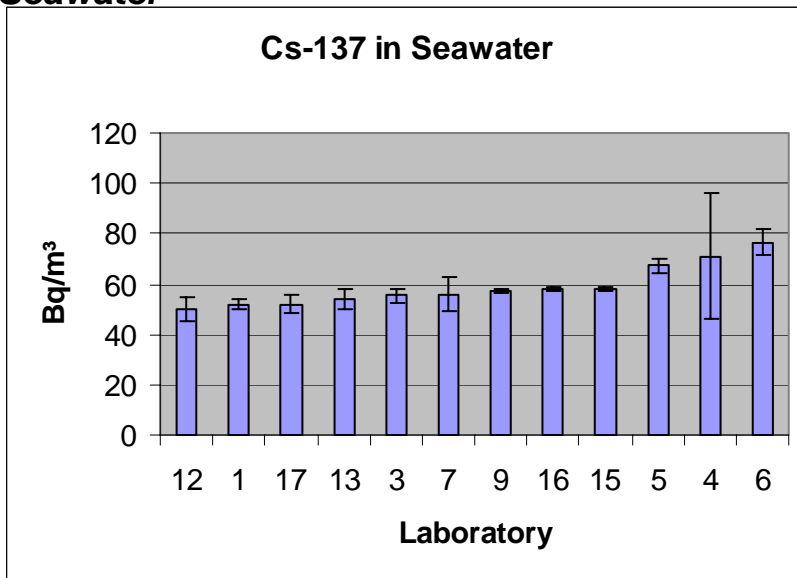
Seaweed



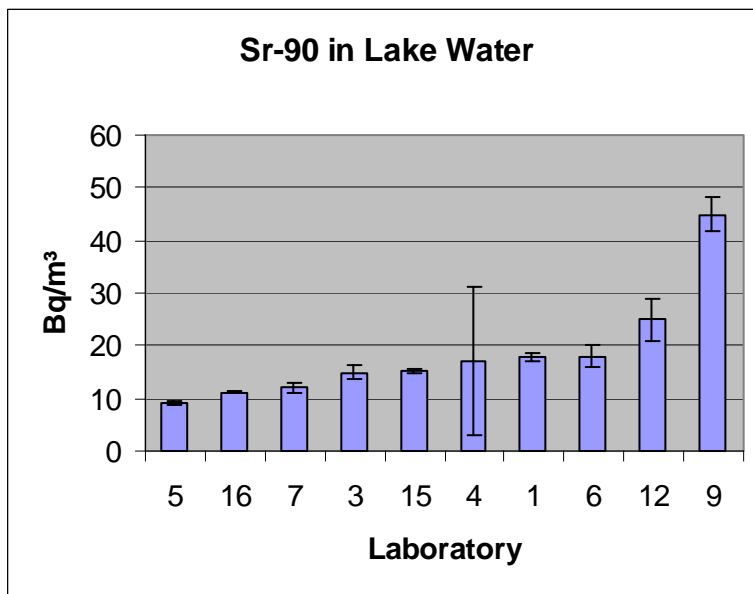
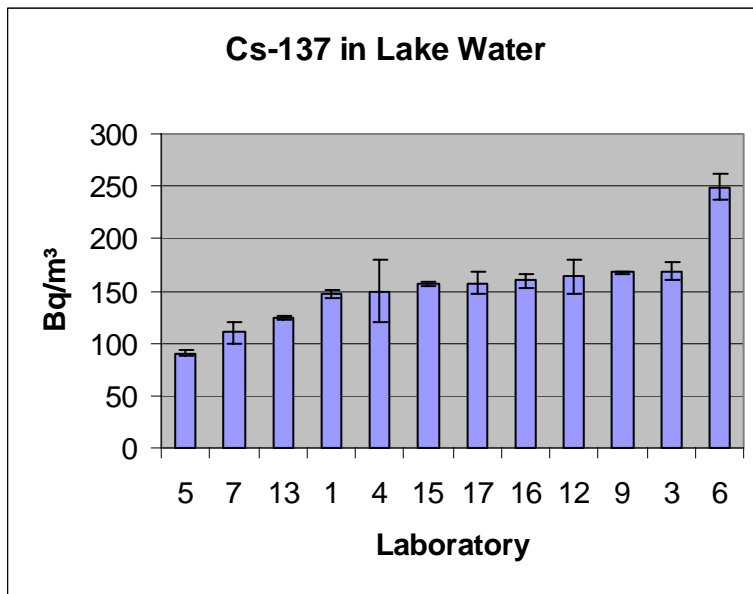




Seawater



Lake Water



Appendix B. Statistical evaluation

The evaluation of the results from each participant is shown in the following tables and graphs for individual radionuclides across samples and for all radionuclides combined. The evaluation is based on the methods described previously, where *n* indicates the number of results submitted, *RSZ* the rescaled sum of z-scores, *SSZ* the sum of squares of z-scores, *Perc* the percentiles from the statistical tests, and *Sign* the associated levels of significance (“ns” denotes not significant, “*” significant at the 95% level, “**” significant at the 99% level, and “***” significant at the 99.9% level).

Caesium-137

Table B1. Statistical evaluation of results for ¹³⁷Cs.

Lab No.	n	RSZ	Perc	Sign	SSZ	Perc	Sign
1	2	-1.1	0.1363	ns	1.2	0.5415	ns
2	3	0.4	0.6689	ns	0.3	0.9543	*
3	5	0.1	0.5527	ns	0.7	0.9841	*
4	5	-1.6	0.0539	ns	16.9	0.0048	**
5	5	-2.2	0.0145	*	57.8	0.0000	***
6	4	4.4	1.0000	***	25.2	0.0000	***
7	5	-2.7	0.0037	**	19.1	0.0018	**
9	2	0.5	0.6978	ns	0.4	0.8167	ns
10	2	-2.7	0.0038	**	7.3	0.0260	ns
11	1	-0.3	0.3754	ns	0.1	0.7509	ns
12	4	-0.2	0.4357	ns	2.2	0.7041	ns
13	5	2.7	0.9969	**	48.1	0.0000	***
14	3	0.0	0.5098	ns	0.0	0.9998	***
15	3	-0.2	0.4225	ns	0.4	0.9310	ns
16	4	1.4	0.9165	ns	5.6	0.2337	ns
17	5	0.2	0.5959	ns	3.8	0.5798	ns

Nuclide|Cs137

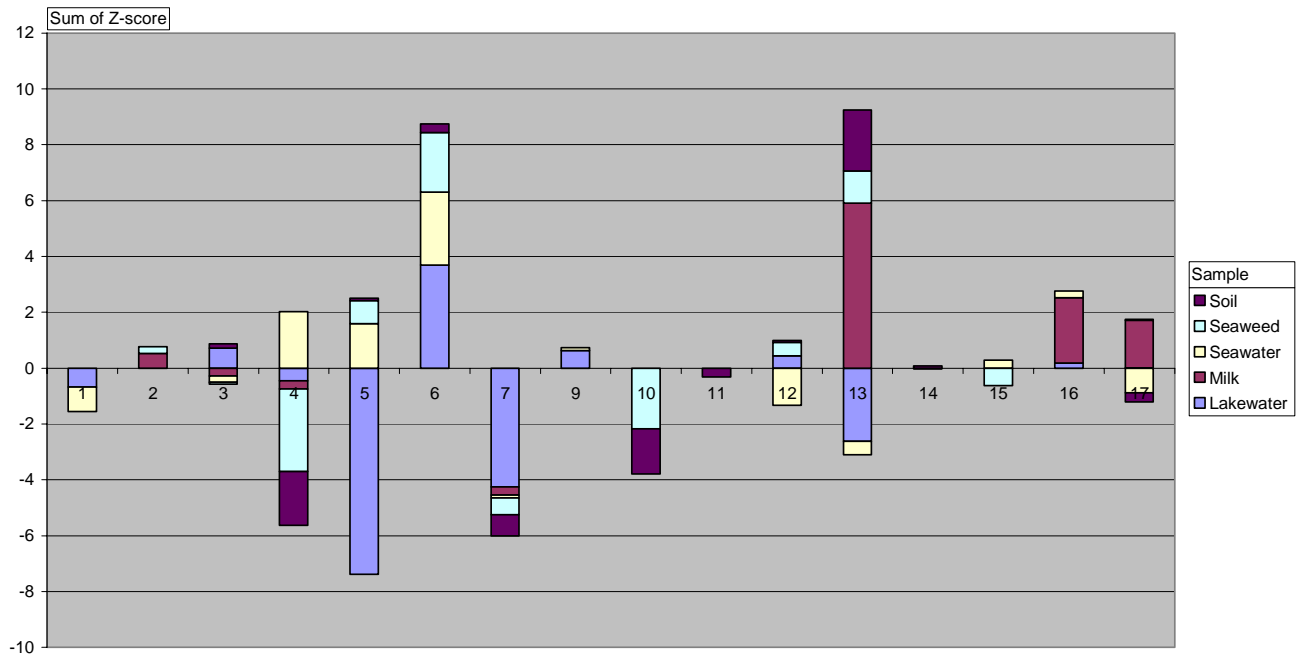


Fig. B1. Column diagram of sum of z-scores for ¹³⁷Cs results showing contributions from individual samples.

Strontium-90

Table B2. Statistical evaluation of results for ^{90}Sr .

Lab No.	n	RSZ	Perc	Sign	SSZ	Perc	Sign
1	5	3.3	0.99945	**	22.7	0.0004	***
3	5	-1.5	0.0637	ns	5.0	0.4115	ns
4	5	3.0	0.9985	**	14.3	0.0139	*
5	4	-3.9	0.0000	***	58.6	0.0000	***
6	5	0.3	0.6182	ns	7.1	0.2112	ns
7	4	-34.0	0.0000	***	2694.2	0.0000	***
9	2	4.5	1.0000	***	41.0	0.0000	***
11	1	1.3	0.9053	ns	1.7	0.1894	ns
12	2	4.5	1.0000	***	20.7	0.0000	***
14	2	-1.0	0.1550	ns	2.2	0.3334	ns
15	2	-0.5	0.2941	ns	0.4	0.8383	Ns
16	1	-4.5	0.0000	***	20.7	0.0000	***

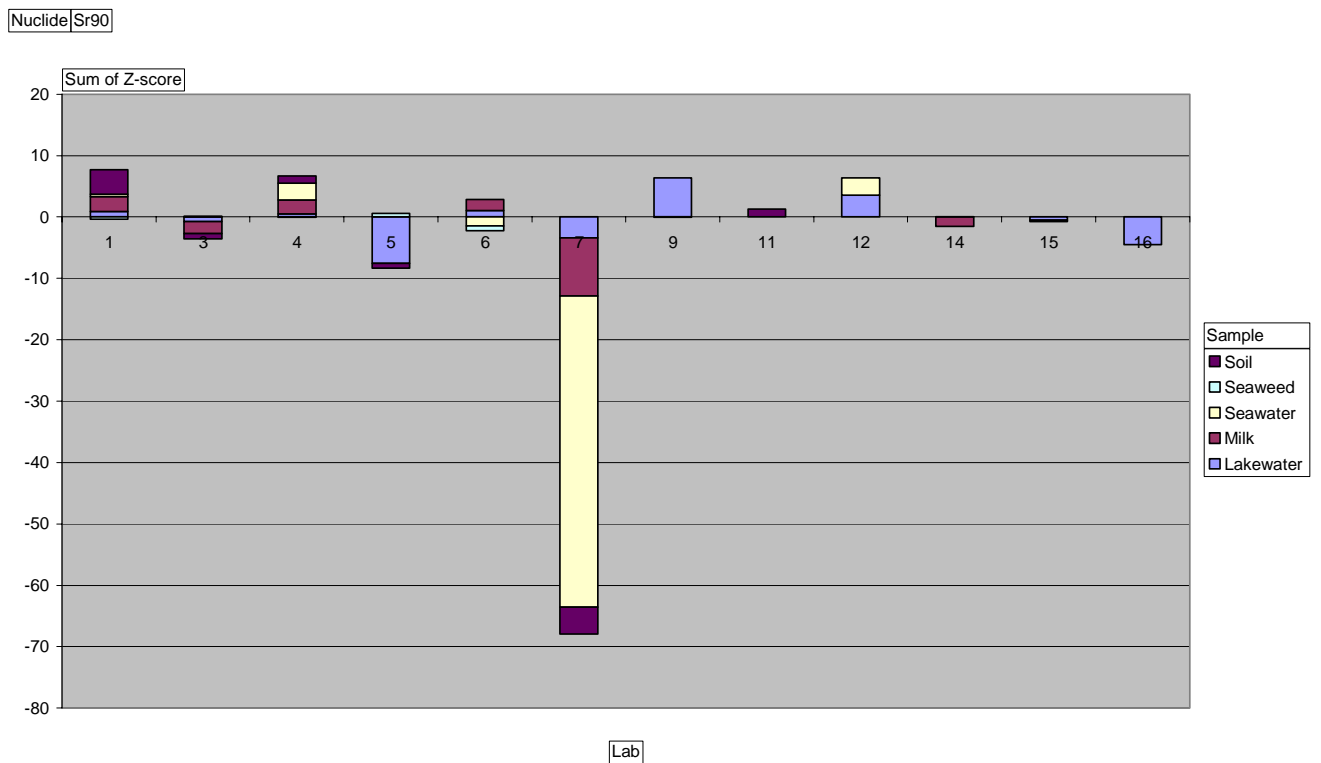


Fig B2. Column diagram of sum of z-scores for ^{90}Sr results showing contributions from individual samples.

Cobalt-60

Table B3. Statistical evaluation of results for ^{60}Co .

Lab No.	n	RSZ	Perc	Sign	SSZ	Perc	Sign
3	1	-0.4	0.3319	ns	0.2	0.6637	Ns
15	1	0.4	0.6554	ns	0.2	0.6892	Ns

Nuclide Co60

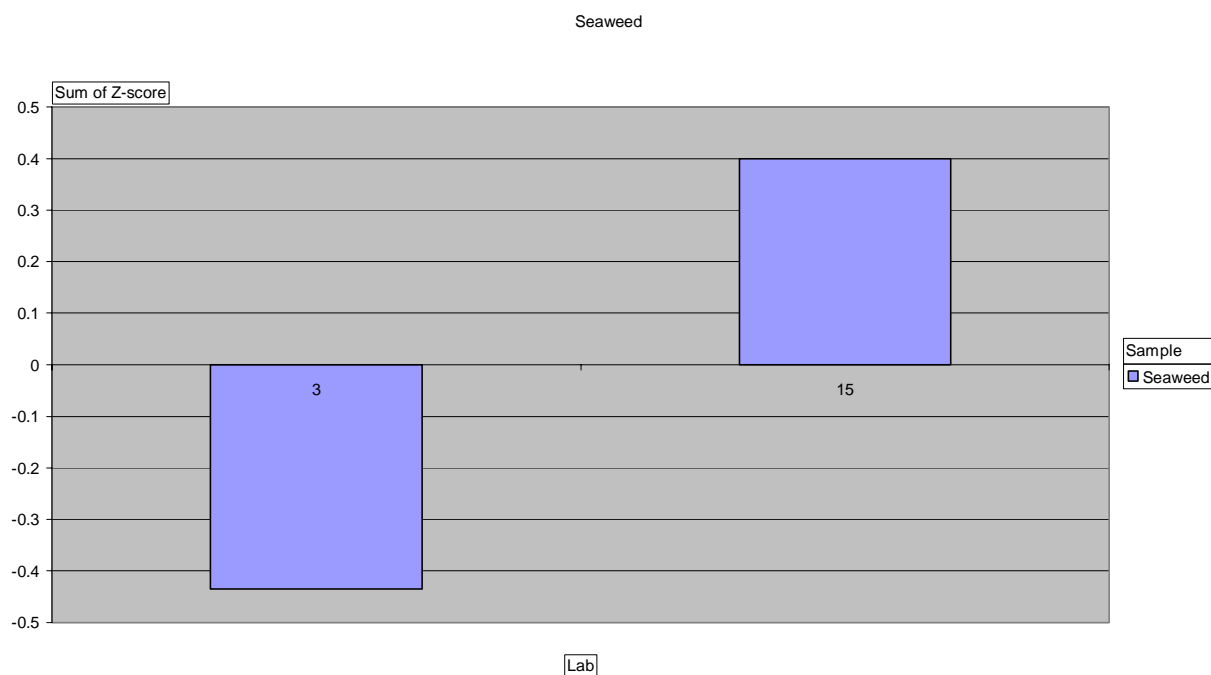


Fig B3. Column diagram of sum of z-scores for ^{60}Co results.

Plutonium-239,240

Table B4. Statistical evaluation of results for $^{239,240}\text{Pu}$.

Lab No.	n	RSZ	Perc	Sign	SSZ	Perc	Sign
3	1	1.4	0.9137	ns	1.9	0.1727	ns
11	1	-1.9	0.0304	ns	3.5	0.0608	ns

Nuclide Pu239,240

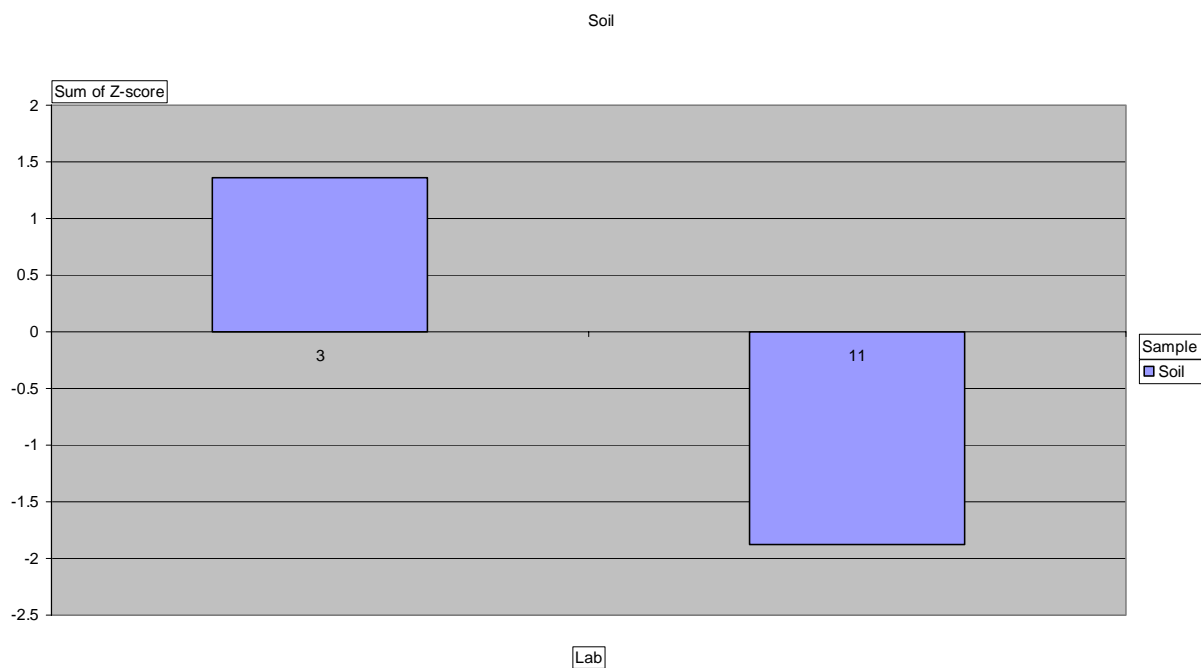


Fig B4. Column diagram of sum of z-scores for $^{239,240}\text{Pu}$ results.

Americium-241

Table B5. Statistical evaluation of results for ²⁴¹Am.

Lab No.	n	RSZ	Perc	Sign	SSZ	Perc	Sign
3	1	-1.1	0.1362	ns	1.2	0.2724	ns
11	1	0.9	0.8159	ns	0.8	0.3681	ns

Nuclide Am241

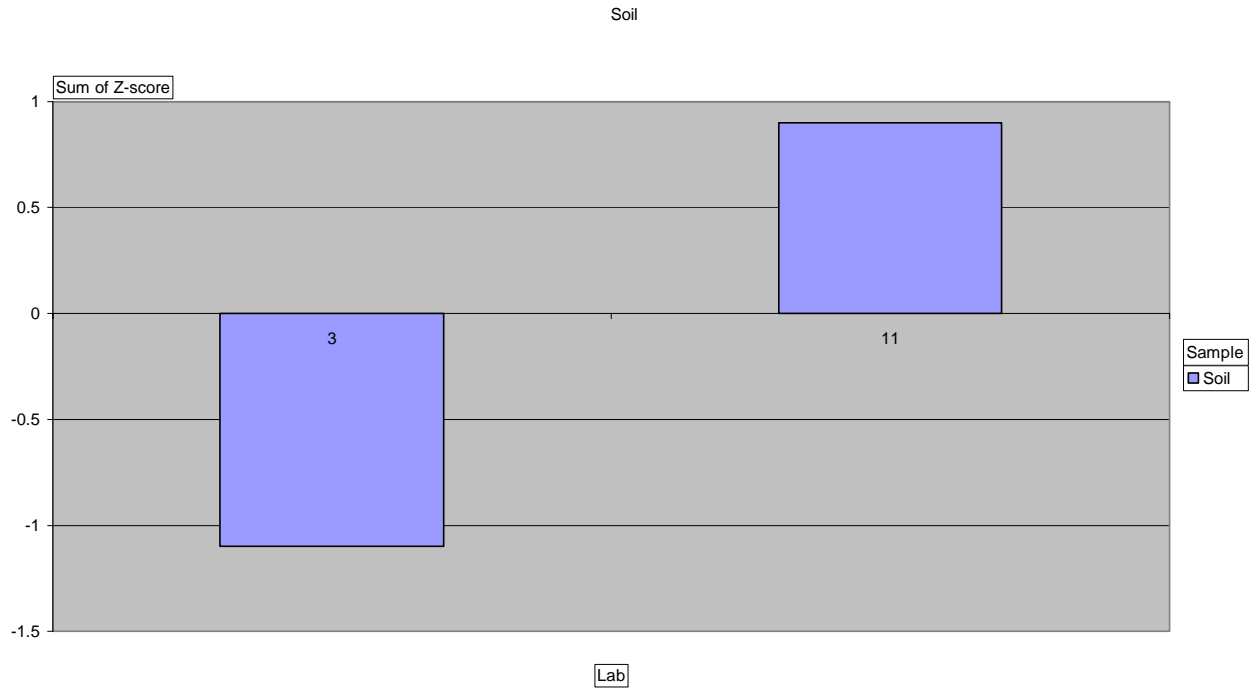


Fig B5. Column diagram of sum of z-scores for ²⁴¹Am results.

Radium-226

Table B6. Statistical evaluation of results for ^{226}Ra .

Lab No.	n	RSZ	Perc	Sign	SSZ	Perc	Sign
2	2	0.2	0.5693	ns	0.1	0.9700	ns
3	2	0.2	0.5762	ns	0.2	0.9156	ns
5	1	0.3	0.6137	ns	0.1	0.7726	ns
7	1	5.2	1.0000	***	27.0	0.0000	***
10	2	-1.8	0.0382	ns	6.9	0.0311	ns
13	1	-5.2	0.0000	***	27.0	0.0000	***
14	1	-0.3	0.3795	ns	0.1	0.7590	ns
16	1	-0.1	0.4761	ns	0.0	0.9523	ns

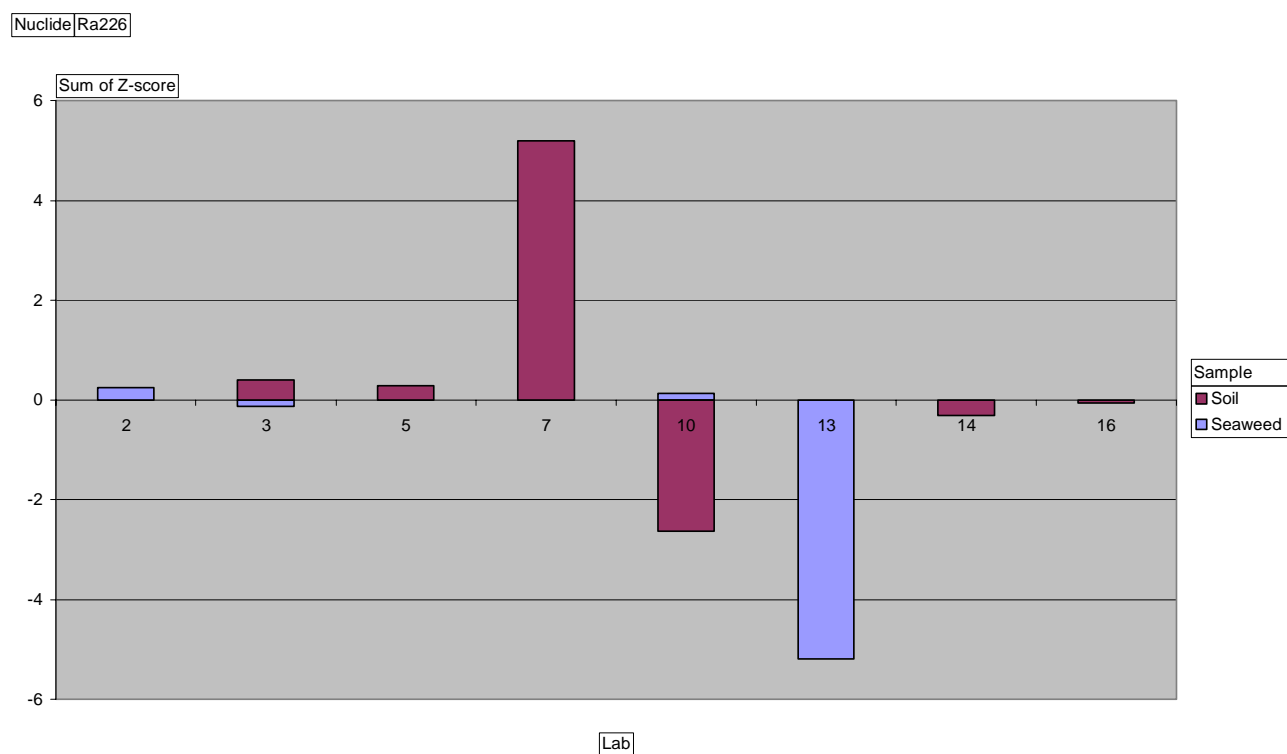


Fig B6. Column diagram of sum of z-scores for ^{226}Ra results showing contributions from individual samples.

Thorium-232

Table B7. Statistical evaluation of results for ^{232}Th .

Lab No.	n	RSZ	Perc	Sign	SSZ	Perc	Sign
2	1	0.2	0.5681	ns	0.0	0.8639	Ns
3	1	0.5	0.7087	ns	0.3	0.5827	Ns
5	1	0.0	0.5000	ns	0.0	1.0000	***
7	1	-0.7	0.2472	ns	0.5	0.4945	Ns
10	1	-2.5	0.0069	*	6.1	0.0137	*
14	1	0.1	0.5458	ns	0.0	0.9085	Ns
16	1	-2.2	0.0140	*	4.8	0.0279	Ns

Nuclide Th232

Soil

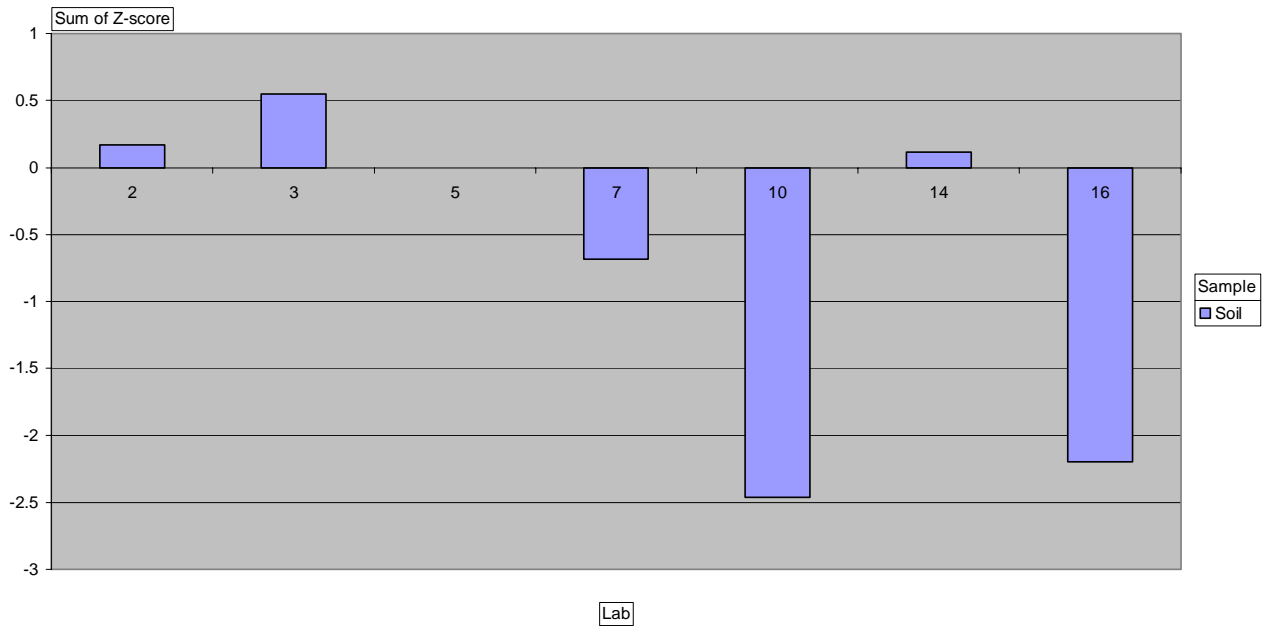


Fig B7. Column diagram of sum of z-scores for ^{232}Th results showing contributions from individual samples.

Potassium-40

Table B8. Statistical evaluation of results for ^{40}K .

Lab No.	n	RSZ	Perc	Sign	SSZ	Perc	Sign
2	3	-0.6	0.2637	ns	1.5	0.6823	Ns
3	3	0.7	0.7617	ns	0.9	0.8135	Ns
4	3	-2.6	0.0047	**	17.5	0.0006	**
5	3	0.2	0.5865	ns	1.5	0.6811	Ns
6	1	2.2	0.9870	*	5.0	0.0260	Ns
7	3	-0.6	0.2749	ns	2.4	0.4866	Ns
10	2	-1.5	0.0732	ns	2.5	0.2892	Ns
13	3	2.7	0.9966	**	18.3	0.0004	***
14	3	-0.6	0.2778	ns	0.7	0.8728	Ns
15	1	-0.5	0.3106	ns	0.2	0.6213	Ns
16	2	1.0	0.8455	ns	1.1	0.5789	Ns
17	3	0.5	0.6989	ns	2.6	0.4644	Ns

Nuclide|K40

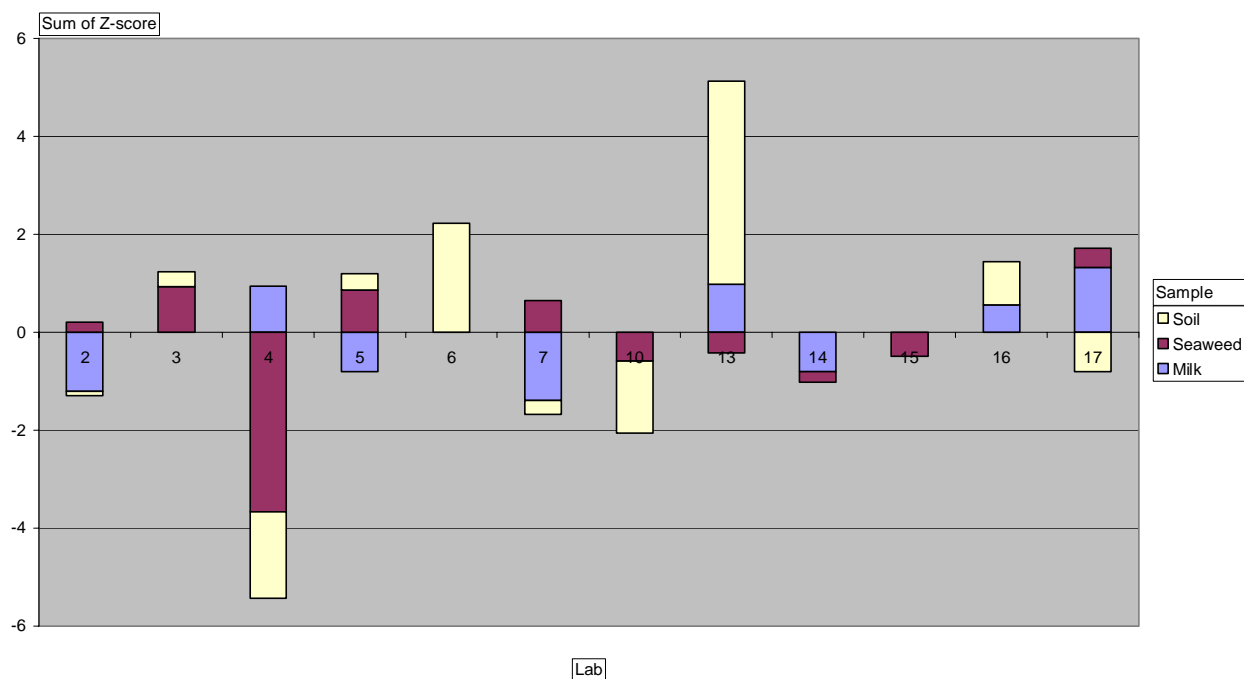


Fig B8. Column diagram of sum of z-scores for ^{40}K results showing contributions from individual samples.

Radionuclides combined

Table B9. Statistical evaluation of results for all radionuclides combined.

Lab No.	n	RSZ	Perc	Sign	SSZ	Perc	Sign
1	7	2.6	0.9949	*	24.0	0.0012	**
2	9	0.0	0.5185	ns	1.9	0.9927	*
3	19	-0.5	0.2916	ns	10.4	0.9425	Ns
4	13	-0.7	0.2559	ns	48.6	0.0000	***
5	14	-5.4	0.0000	***	117.9	0.0000	***
6	10	5.2	1.0000	***	37.2	0.0001	***
7	14	-31.5	0.0000	***	2743.2	0.0000	***
9	4	5.0	1.0000	***	41.4	0.0000	***
10	7	-7.6	0.0000	***	22.8	0.0019	**
11	4	0.0	0.5080	ns	6.1	0.1883	Ns
12	6	3.0	0.9988	**	22.8	0.0009	***
13	9	2.5	0.9941	*	93.3	0.0000	***
14	10	-1.5	0.0662	ns	3.0	0.9811	*
15	7	-0.7	0.2445	ns	1.2	0.9909	*
16	9	-1.3	0.0963	ns	32.2	0.0002	***
17	8	0.6	0.7410	ns	6.4	0.6078	Ns

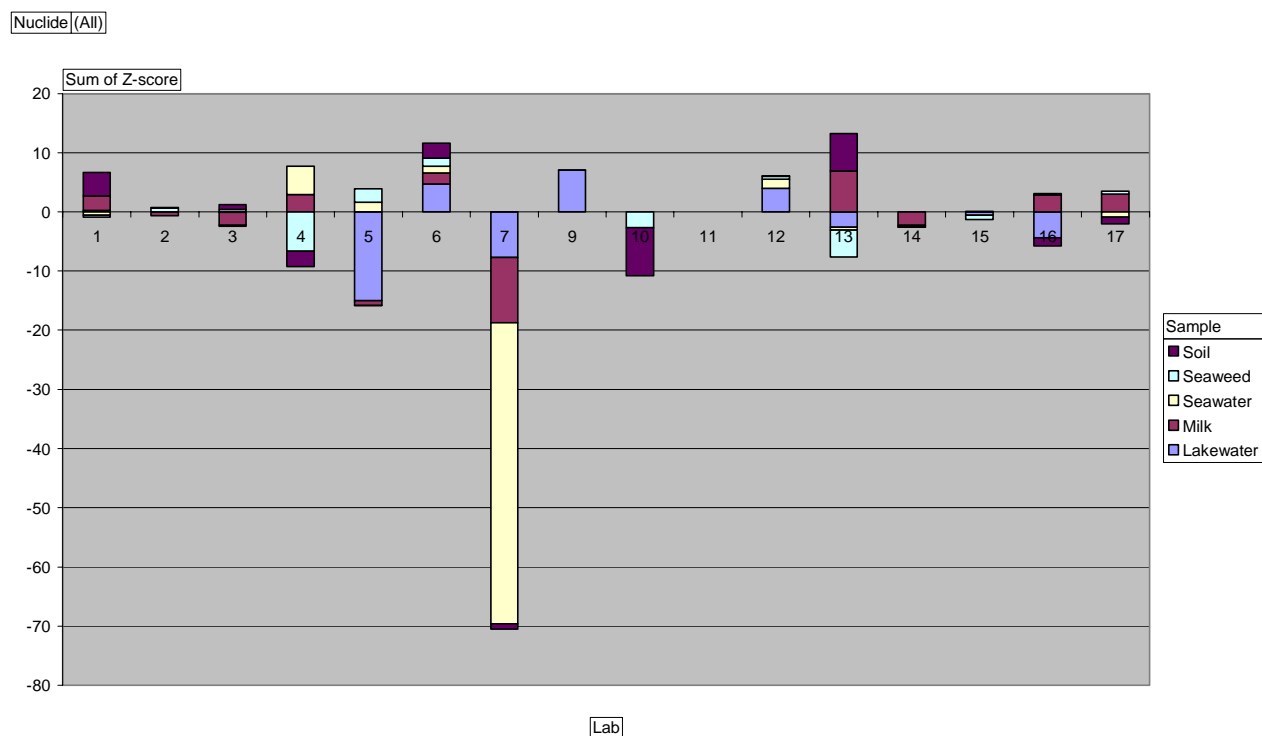


Fig B9. Column diagram of sum of z-scores for results from all radionuclides combined showing contributions from individual samples.

Mission

To promote an innovative and environmentally sustainable technological development within the areas of energy, industrial technology and bioproduction through research, innovation and advisory services.

Vision

Risø's research **shall extend the boundaries** for the understanding of nature's processes and interactions right down to the molecular nanoscale.

The results obtained shall **set new trends** for the development of sustainable technologies within the fields of energy, industrial technology and biotechnology.

The efforts made **shall benefit** Danish society and lead to the development of new multi-billion industries.