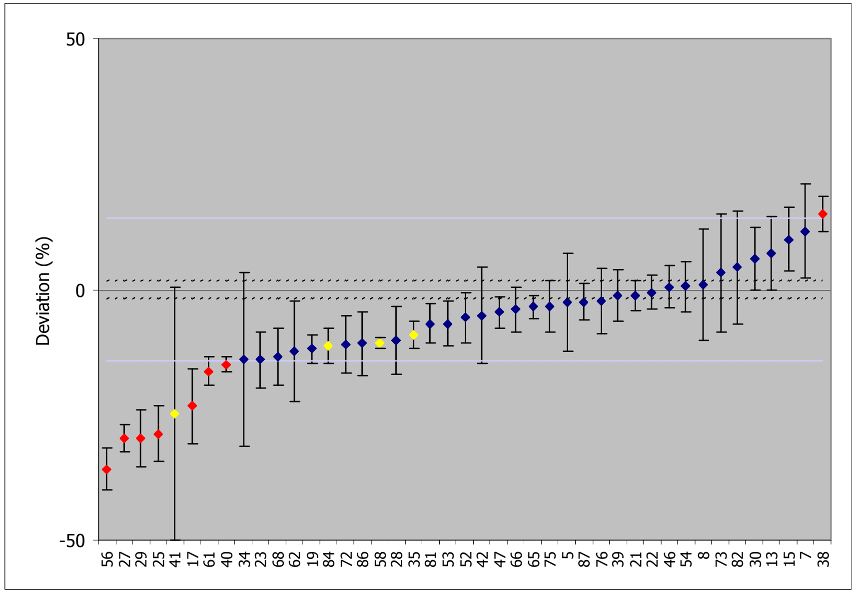
S samples 2009



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20 January 2010

Radionuclides in S

6 nuclides:

^{55}Fe, ^{90}Sr, ^{133}Ba, ^{134}Cs, ^{137}Cs and ^{152}Eu

Participants: 26

S composition (Bq g^{-1})

	NPL	Labs	*t*-test	Ratio
^{55}Fe	**4.38(13)**	4.09(21)	-1.16	0.93
^{90}Sr	**0.810(20)**	0.757(17)	-2.01	0.93
^{133}Ba	**4.91(13)**	4.55(3)	-2.79 D	0.93
^{134}Cs	**11.8(3)**	11.05(5)	-2.38	0.94
^{137}Cs	**5.05(13)**	5.15(4)	0.78	1.02
^{152}Eu	**4.84(13)**	4.67(4)	-1.25	0.97

Homogeneity uncertainty

Measure each samples once:

$$u_{bb} = \sqrt{\frac{\sum_i (x_i - \bar{x}_i)^2}{n-1}} \left(\frac{100}{\bar{x}_i}\right)$$

Measure one sample *m* times:

$$u_{\text{meas}} = \sqrt{\frac{\sum_j (x_j - \bar{x}_j)^2}{m-1}} \left(\frac{100}{\bar{x}_j}\right)$$

Internal (counting) uncertainty:

$$u_{\text{int}} = 100\, mean\left(\frac{u_i}{x_i}\right)$$

Homogeneity uncertainty:

$$u_{\text{hom}}^2 = u_{bb}^2 - u_{meas}^2$$

or

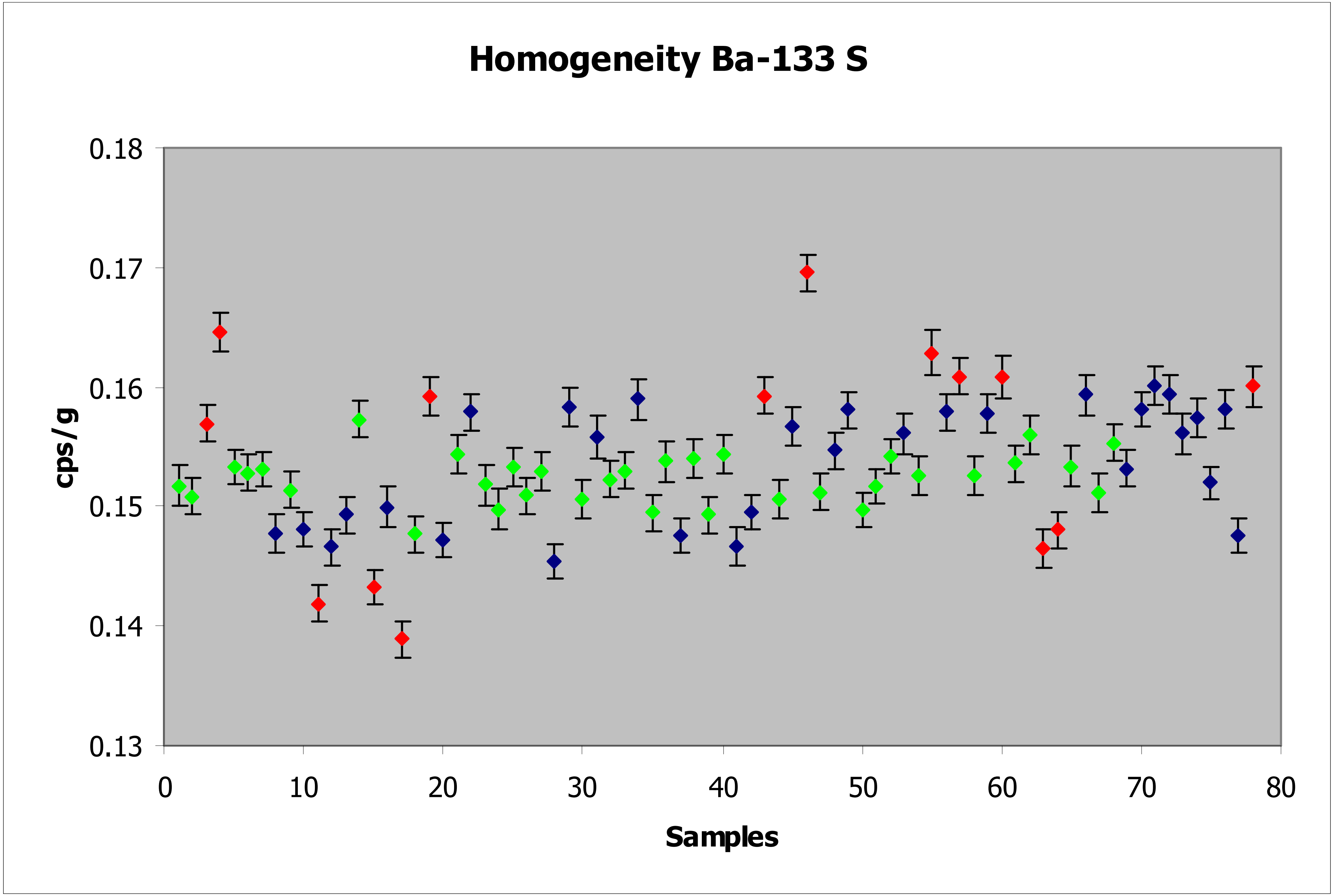
$$u_{\text{hom}}^2 = u_{bb}^2 - u_{\text{int}}^2$$

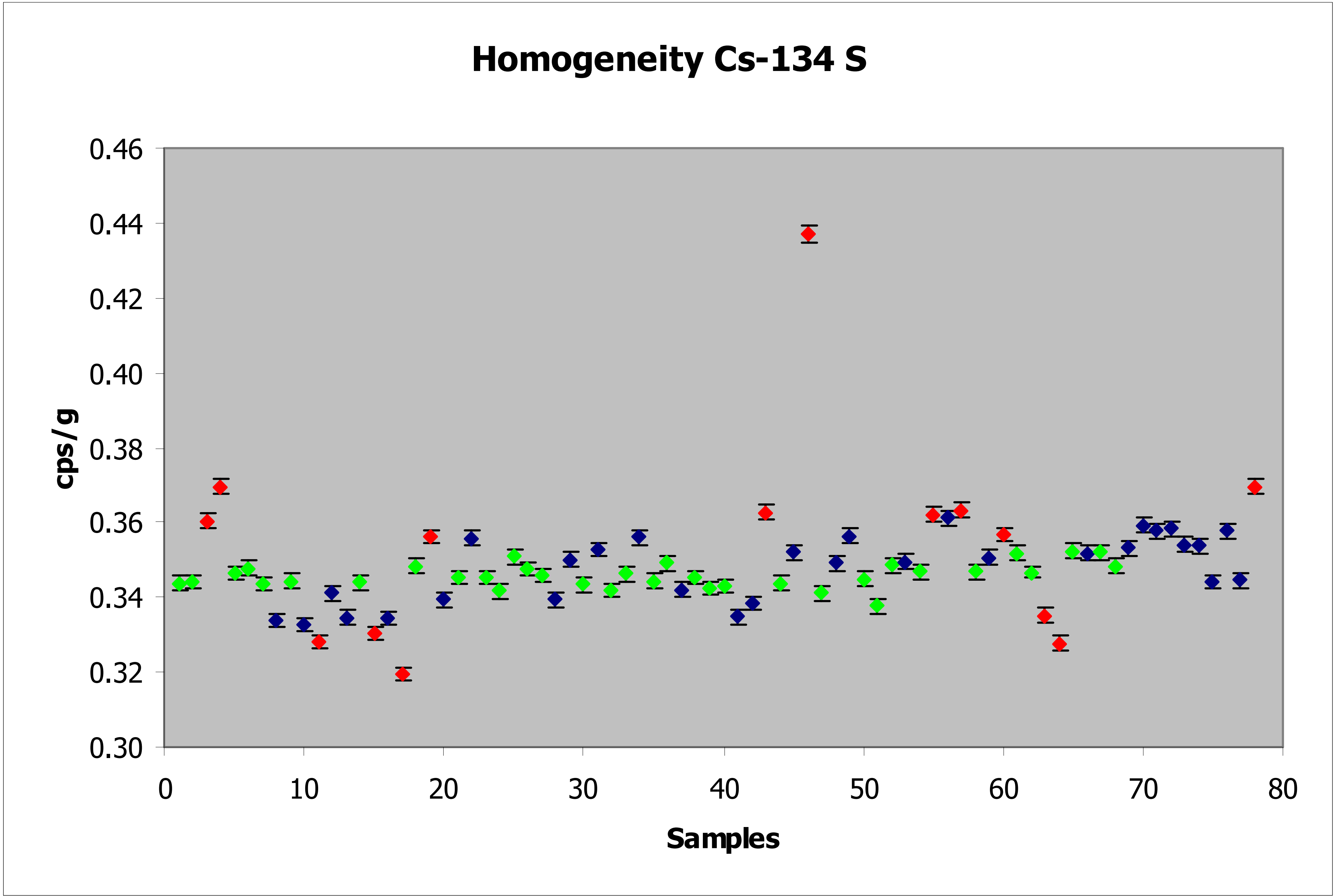
whichever gives the lowest result

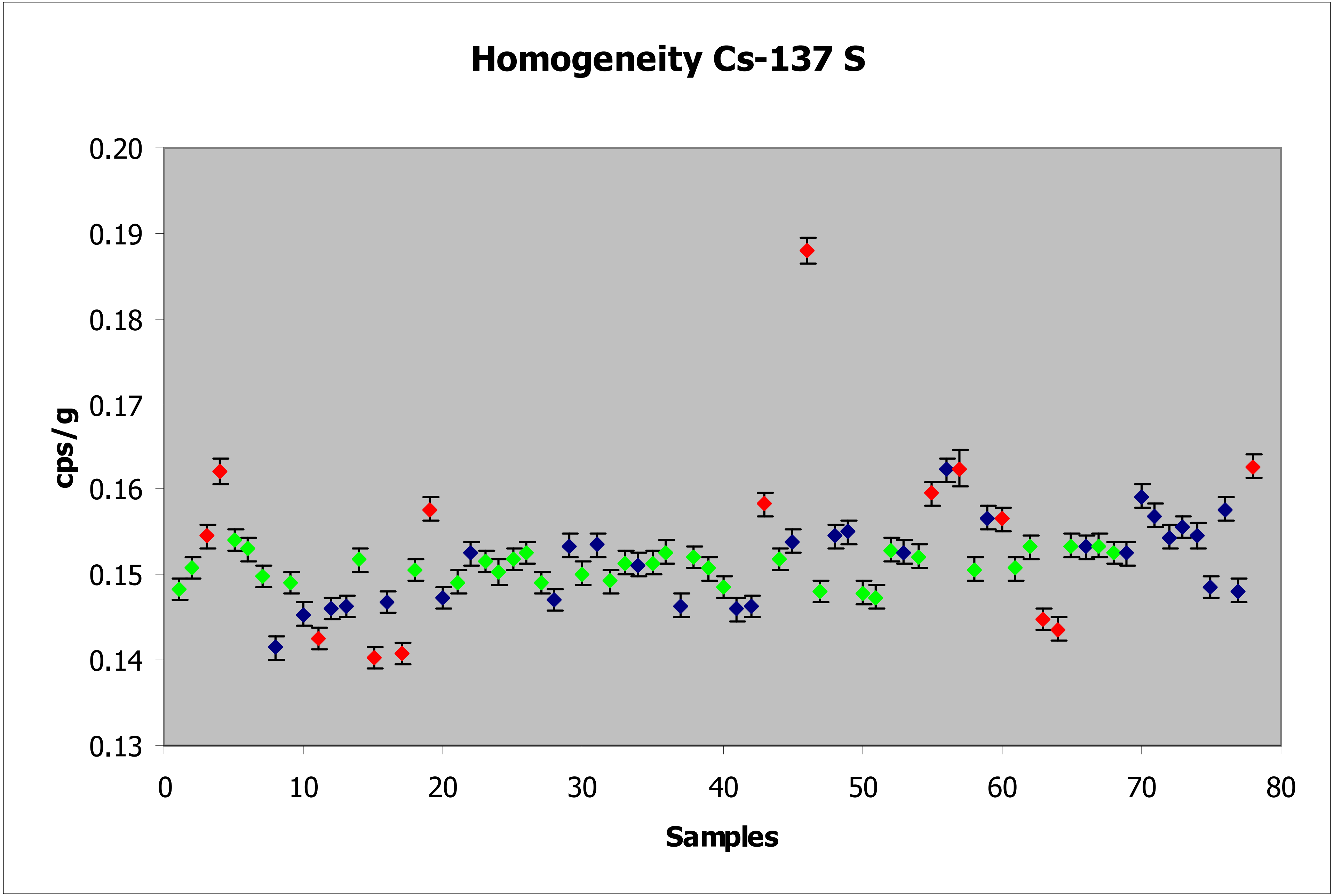
Homogeneity uncertainty

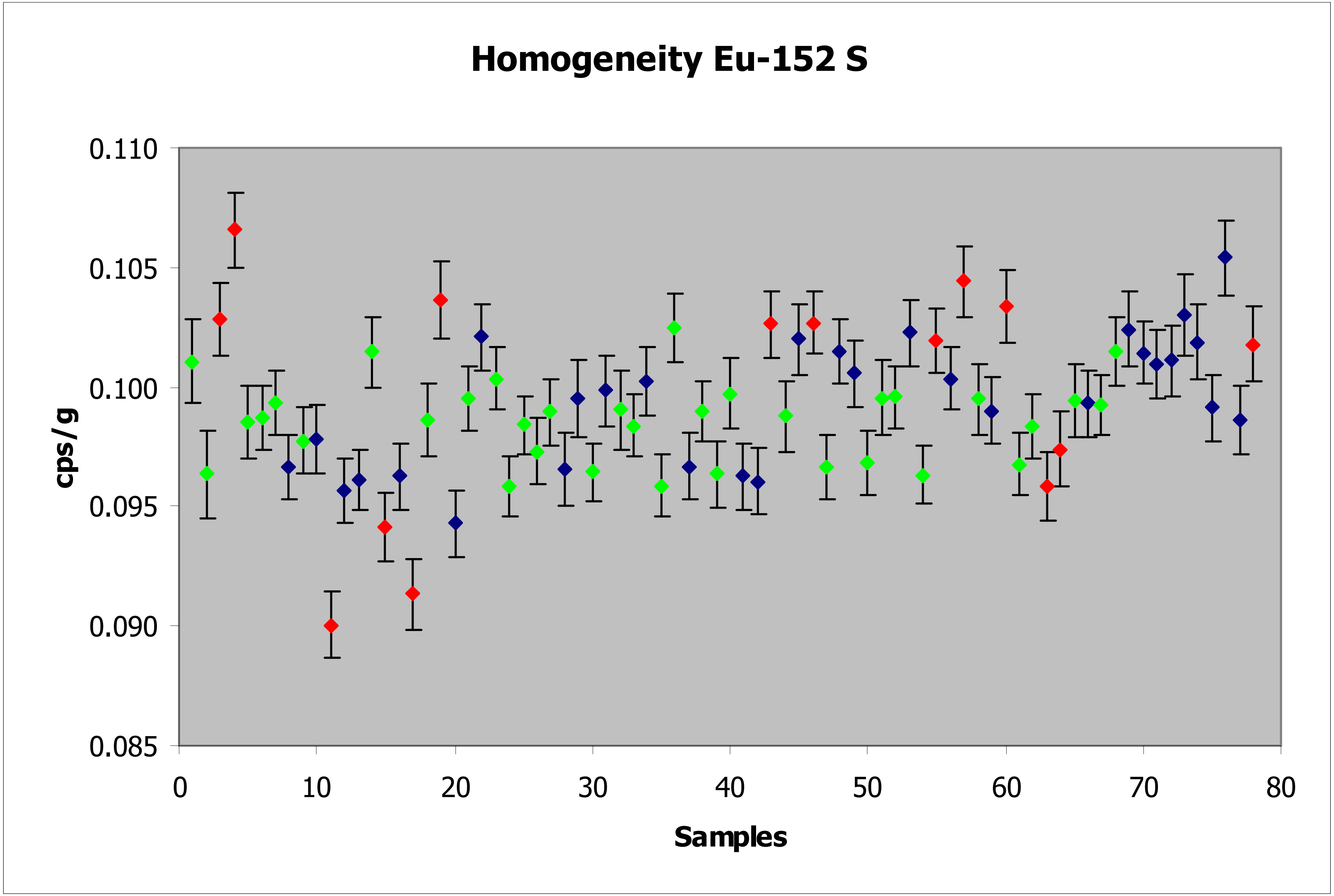
	u_{bb} (%)	u_{meas} (%)	u_{int} (%)	u_{hom} (%)
^{133}Ba	1.34	1.61*	1.03	**0**
^{134}Cs	0.95	1.41*	0.55	**0**
^{137}Cs	1.19	1.73*	0.89	**0**
^{152}Eu	1.73	2.42*	1.42	**0**

	u_{N^*} (%)	u_{hom} (%)	u_{stab} (%)	u_N (%)
^{55}Fe	1.73	-	2.5	**3.04**
^{90}Sr	0.20	-	2.5	**2.51**
^{133}Ba	0.71	0	2.5	**2.60**
^{134}Cs	0.68	0	2.5	**2.59**
^{137}Cs	0.69	0	2.5	**2.59**
^{152}Eu	0.68	0	2.5	**2.59**


Homogeneity Ba-133 S
0.18
0.17
0.16
0.15
0.14
0.13
cps/g
0
10
20
30
40
50
60
70
80
Samples


Homogeneity Cs-134 S
cps/g
0.46
0.44
0.42
0.40
0.38
0.36
0.34
0.32
0.30
0
10
20
30
40
50
60
70
80
Samples


Homogeneity Cs-137 S
0.20
0.19
0.18
0.17
0.16
0.15
0.14
0.13
cps/g
0
10
20
30
40
50
60
70
80
Samples

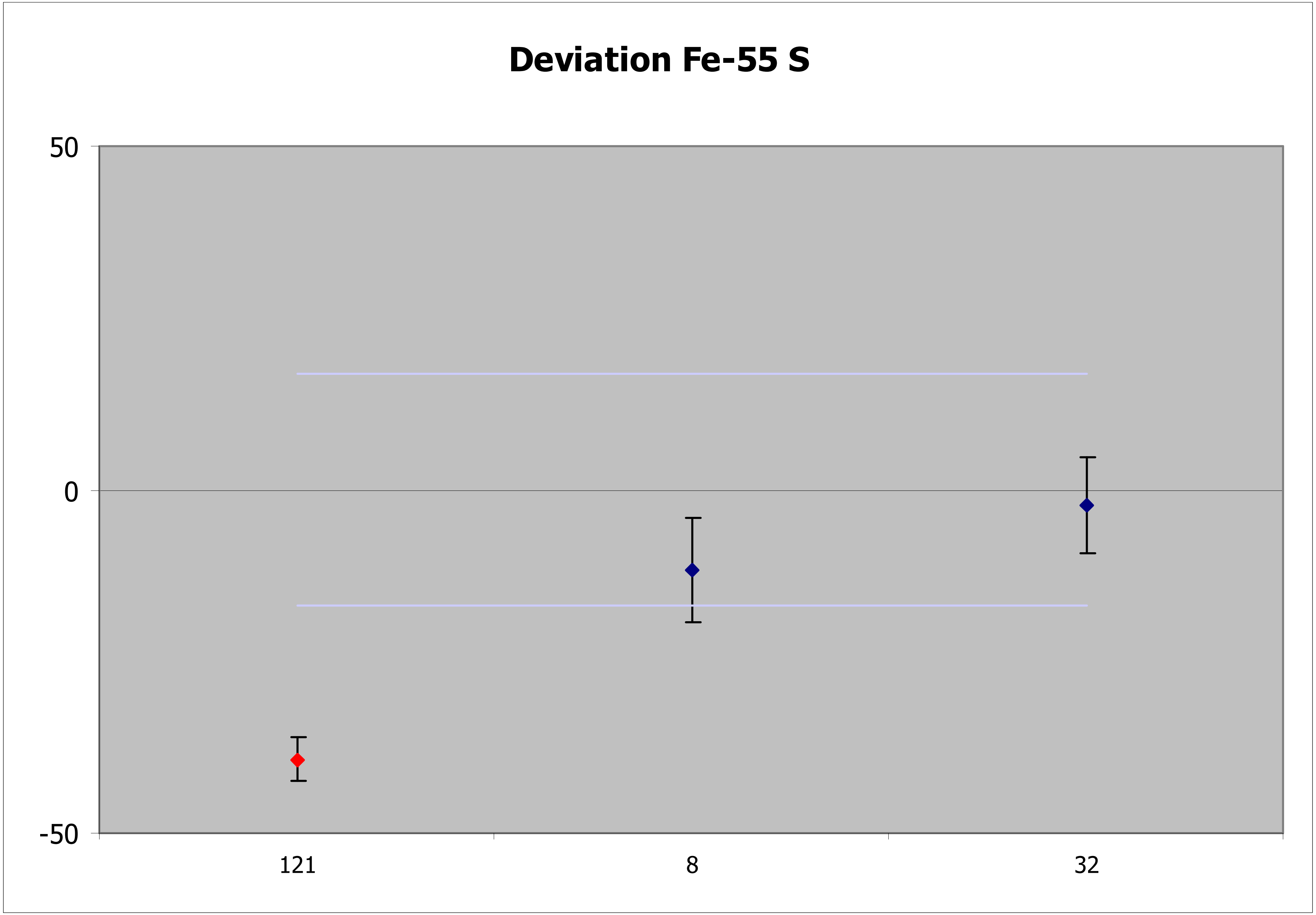

Homogeneity Eu-152 S
cps/g
0.110
0.105
0.100
0.095
0.090
0.085
0
10
20
30
40
50
60
70
80
Samples

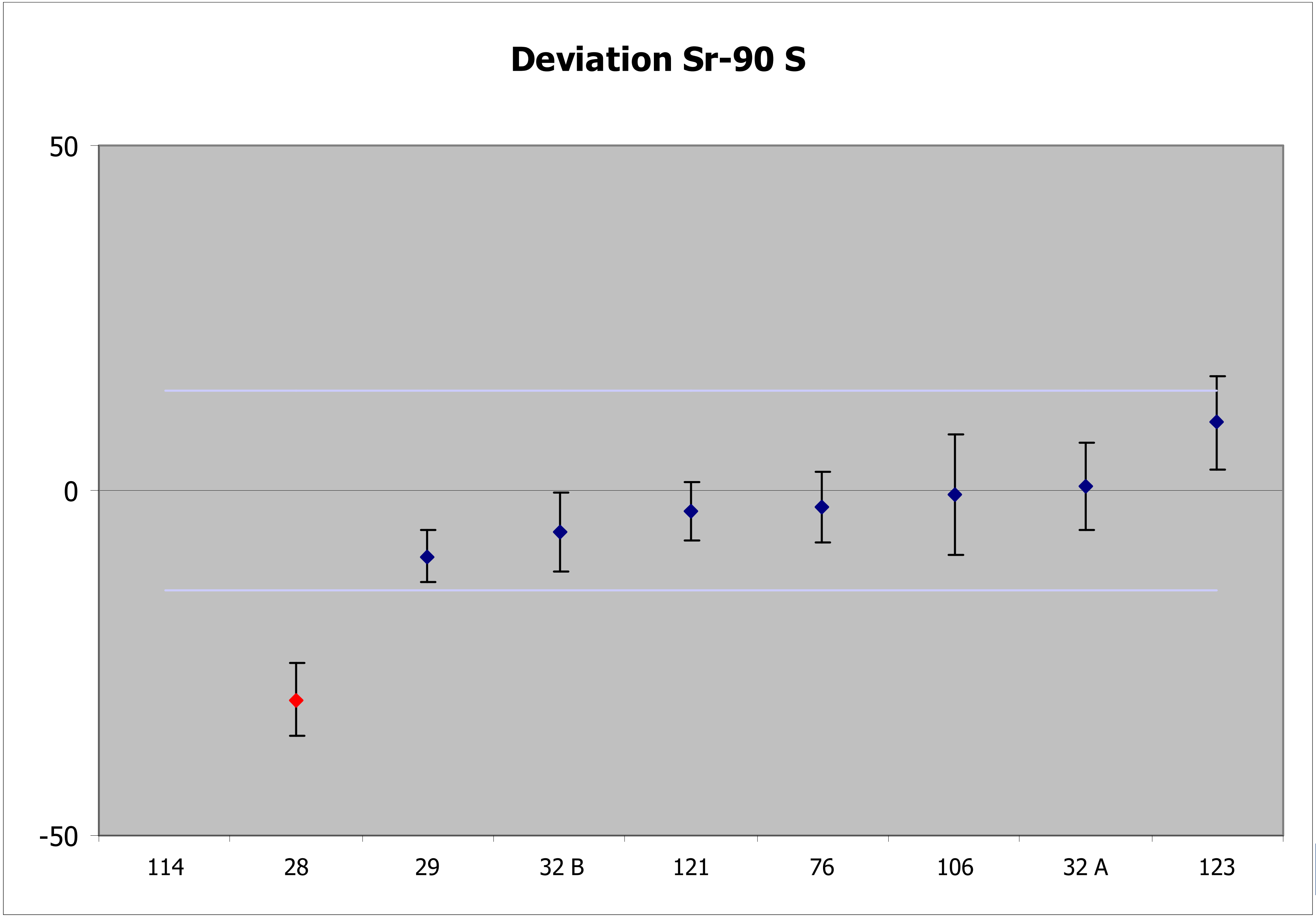
S in agreement (%)

		(size of LCS)
^{55}Fe	**67**	(67%)
^{90}Sr	**78**	(78%)
^{133}Ba	**64**	(82%)
^{134}Cs	**70**	(78%)
^{137}Cs	**88**	(88%)
^{152}Eu	**68**	(82%)

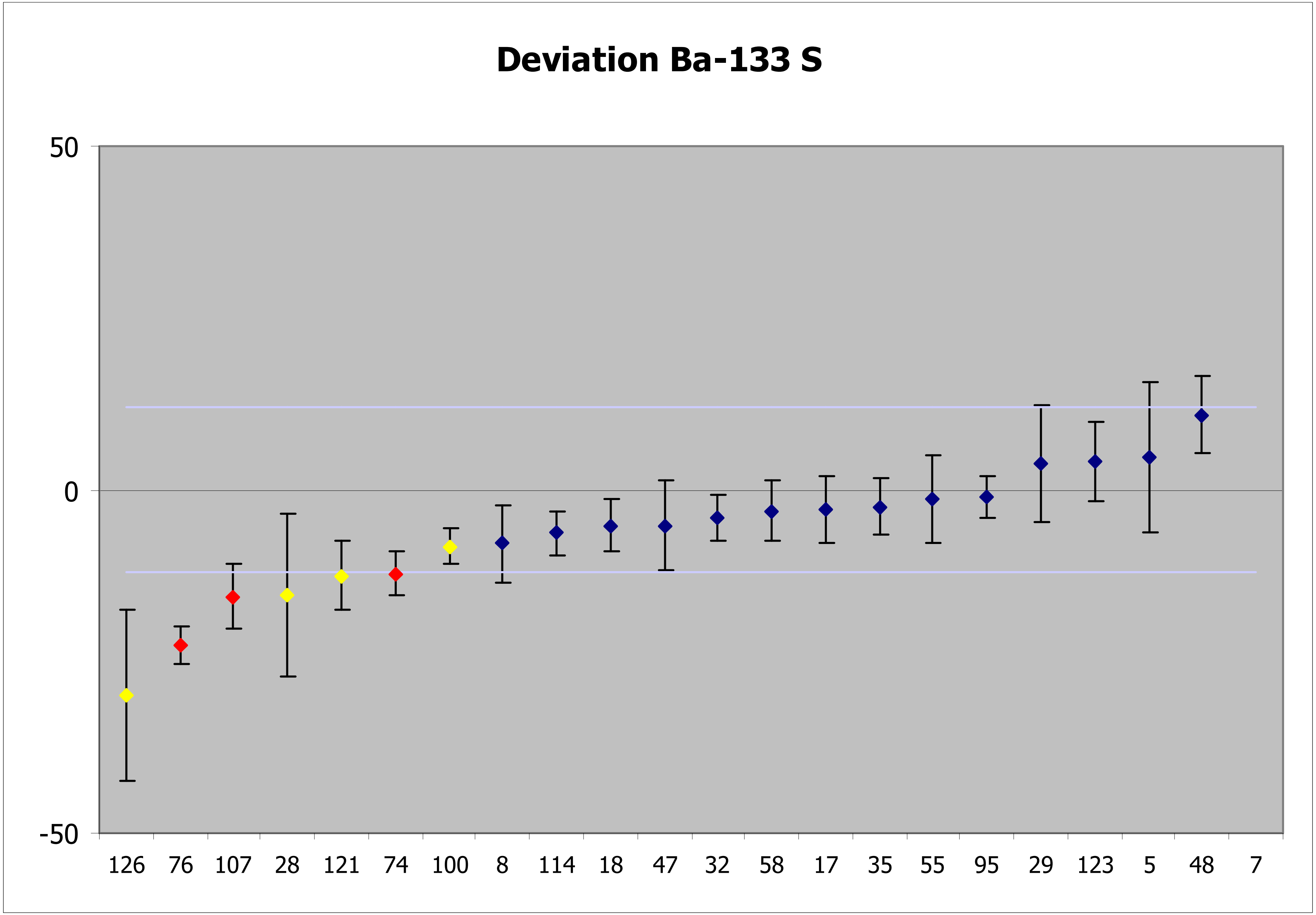
Results S

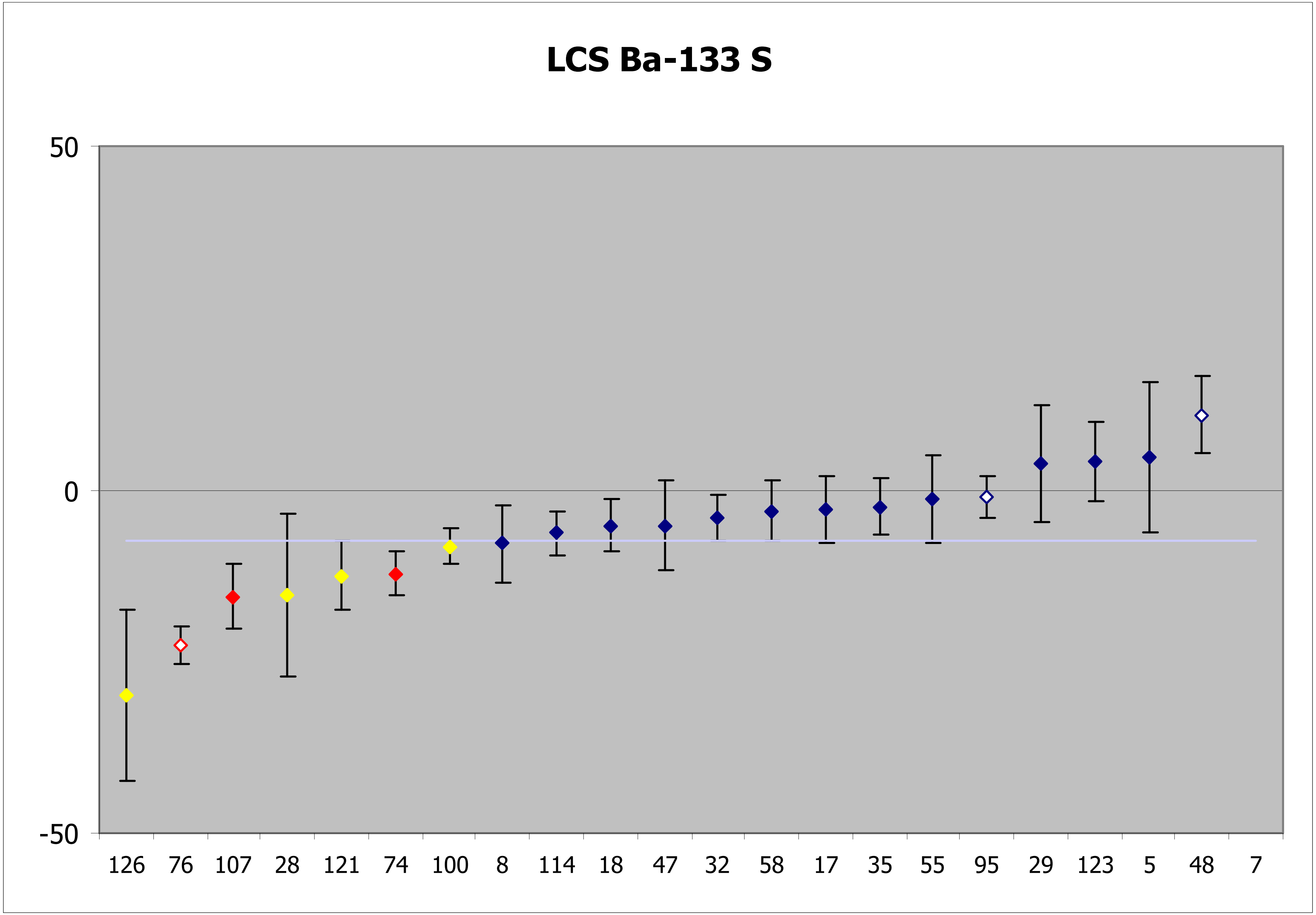
73%	‘in agreement’
12%	‘questionable’
16%	‘discrepant’


Deviation Fe-55 S
50
0
-50
121
8
32

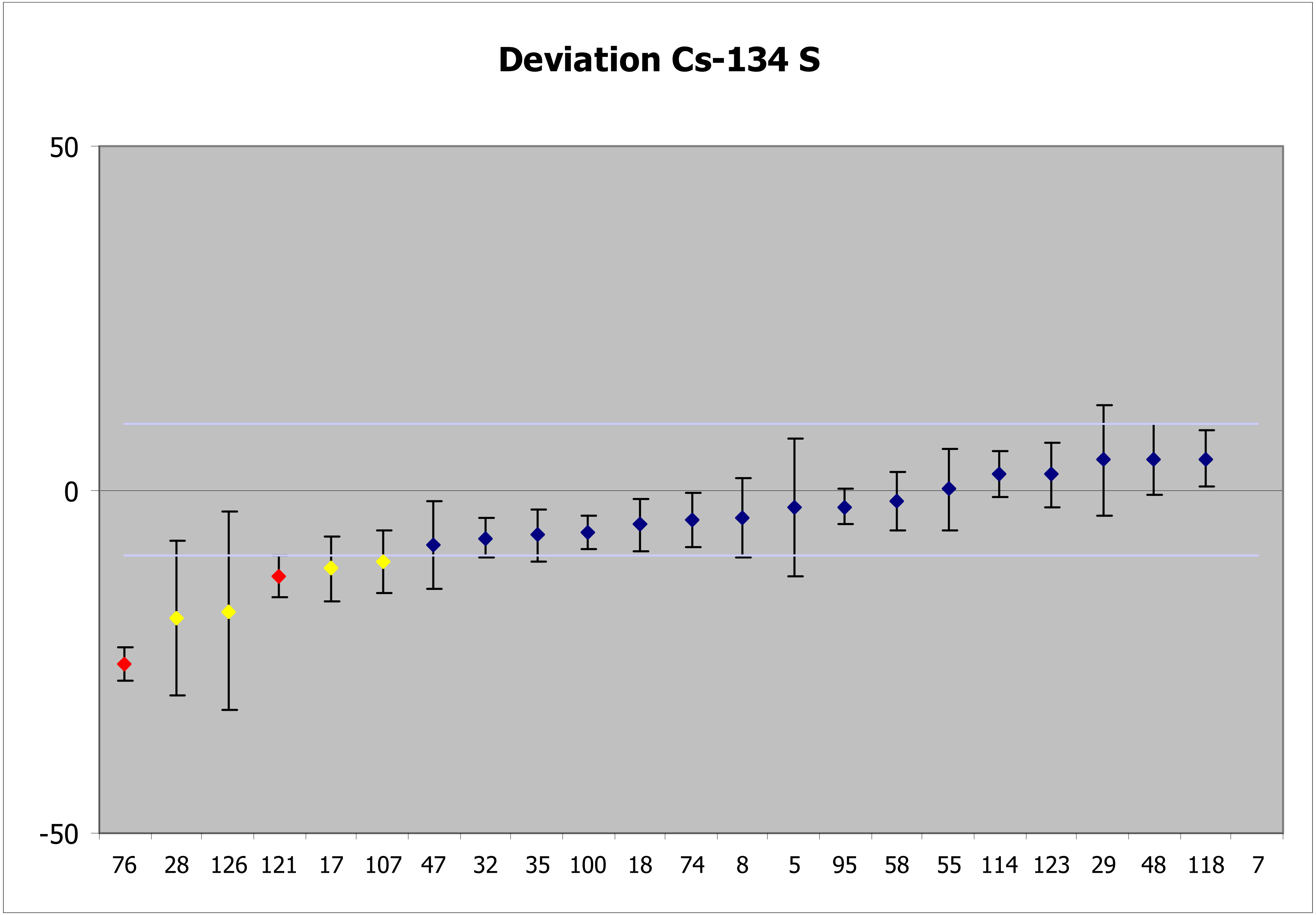


Deviation Sr-90 S
50
0
-50
114
28
29
32 B
121
76
106
32 A
123

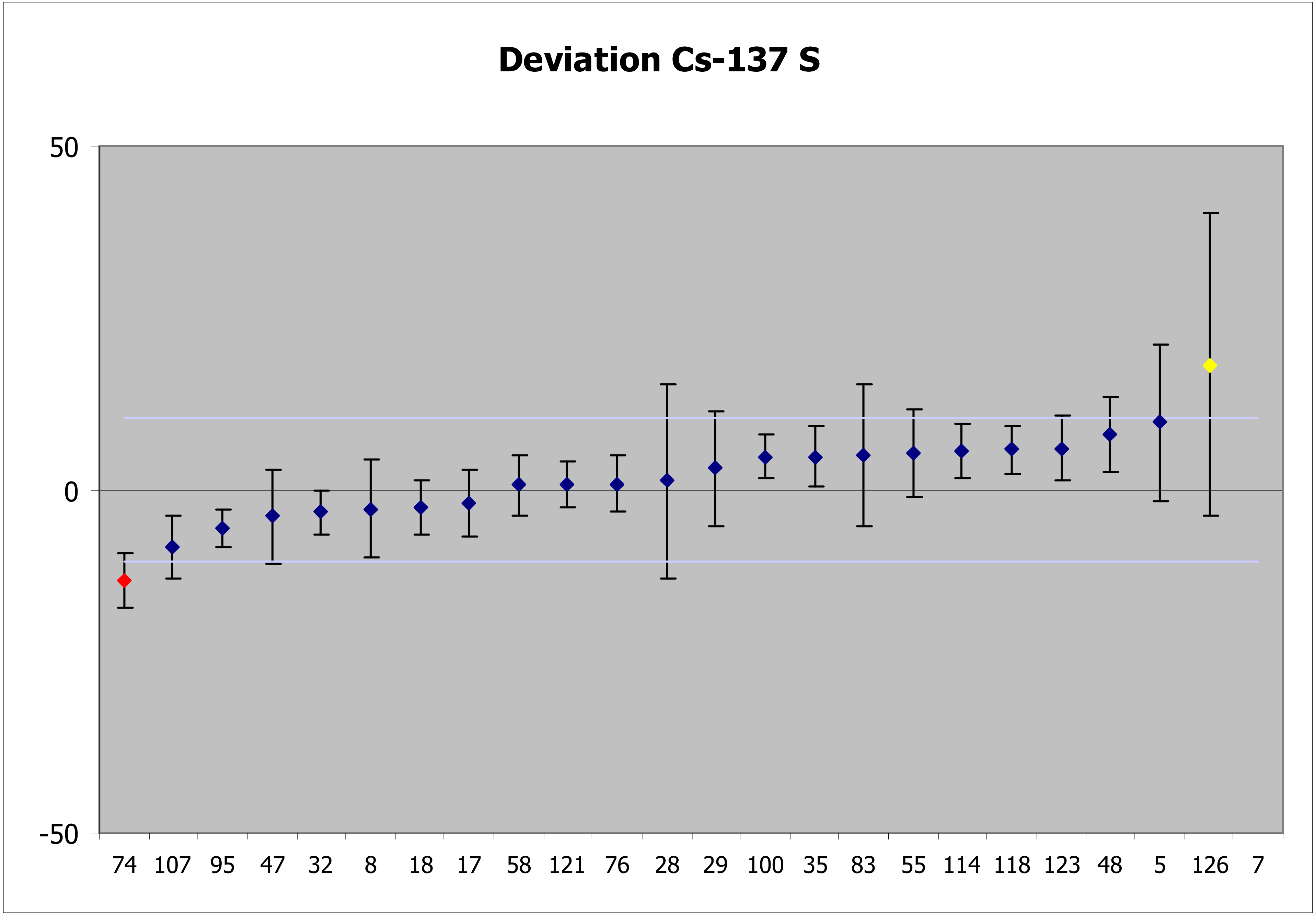

Deviation Ba-133 S
50
0
-50
126
76
107
28
121
74
100
8
114
18
47
32
58
17
35
55
95
29
123
5
48
7



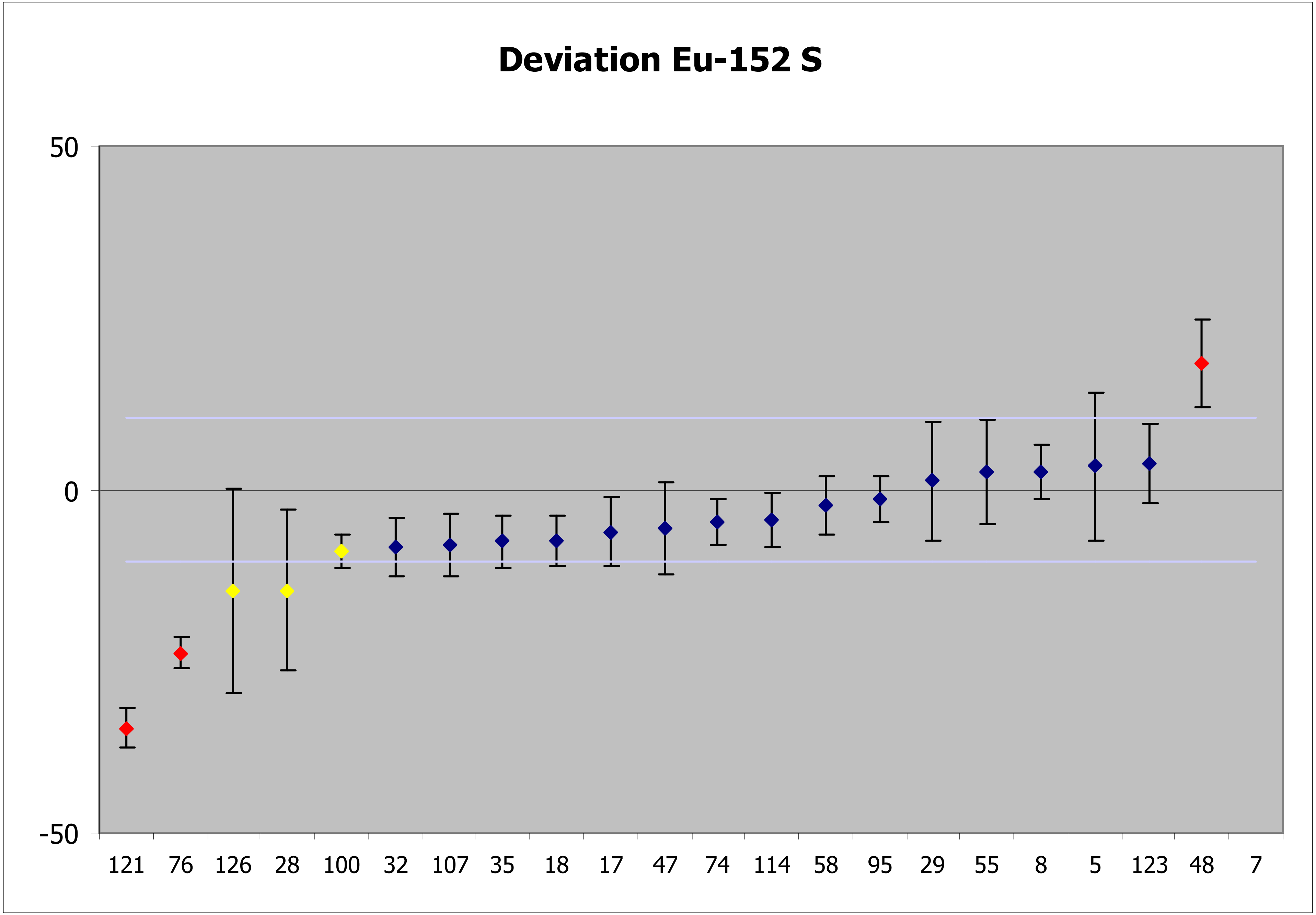
LCS Ba-133 S
50
0
-50
126
76
107
28
121
74
100
8
114
18
47
32
58
17
35
55
95
29
123
5
48
7



Deviation Cs-134 S
50
0
-50
76
28
126
121
17
107
47
32
35
100
18
74
8
5
95
58
55
114
123
29
48
118
7



Deviation Cs-137 S
50
0
-50
74
107
95
47
32
8
18
17
58
121
76
28
29
100
35
83
55
114
118
123
48
5
126
7



Deviation Eu-152 S
50
0
-50
121
76
126
28
100
32
107
35
18
17
47
74
114
58
95
29
55
8
5
123
48
7

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Contents lists available at ScienceDirect

Applied Radiation and Isotopes

journal homepage: www.elsevier.com/locate/apradiso

Development of a neutron-activated concrete powder reference material

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ARTICLE INFO

Keywords:
Reference material
Neutron-activated concrete powder
Gamma-emitting radionuclides
Comparison
Homogeneity
Stability

ABSTRACT

In this paper, the development of a neutron-activated concrete powder reference material is described. The material originated from core samples taken from a concrete bioshield of a decommissioned nuclear reactor which ceased operation almost 30 years ago after approximately 20 years of operation. The assigned values, which were in the Bq g^{-1} range, for the radionuclides in the material were determined by a 'consensus' method from measurements made by 33 organisations from 15 countries. The measurements were made within a wider test exercise (the NPL Environmental Radioactivity Proficiency Test Exercise 2008). Assigned specific activity values were obtained for ^{60}Co, ^{133}Ba, ^{152}Eu and ^{154}Eu and indicative values were obtained for ^{3}H (total), ^{3}H (leachable), ^{14}C, ^{40}K, ^{55}Fe and ^{63}Ni.

1. Introduction

Large volumes of construction materials, such as concrete, are a significant part of the low level and intermediate level radioactive waste streams generated by decommissioning nuclear facilities (Hou, 2007; Hong et al., 2009). In order to assign materials to the correct waste stream, there is a general need for certified reference materials to serve as quality control materials to achieve traceability, method validation and instrument calibration.

In this paper, the development of a neutron-activated concrete powder reference material is described. The material originated from core samples taken from the concrete bioshield of a decommissioned nuclear reactor which ceased operation almost 30 years ago after approximately 20 years of operation. Apart from natural activity, the concrete was thought to contain the following radionuclides formed by neutron activation of concrete components: ^{3}H (both fixed and mobile), ^{14}C, ^{36}Cl, ^{41}Ca, ^{55}Fe, ^{60}Co, ^{63}Ni, ^{133}Ba, ^{152}Eu and ^{154}Eu (Suzuki et al., 2001; Kinno et al, 2002; Hou, 2007; Kim et al., 2008). The assigned values for the radionuclides in the material were determined by a 'consensus' method from measurements made by 33 organisations from 15 countries. The measurements were made within a wider test exercise (the NPL Environmental Radioactivity Proficiency Test Exercise 2008) (Harms and Gilligan, 2009). The assigned values, which were in the Bq g^{-1} range, were calculated as the weighted mean of the largest consistent subset (LCS), a technique that takes the measurement uncertainties of the submitted results into account (Cox, 2007). Robust means (as described by ISO 13528, 2005), which ignore the measurement uncertainties of the submitted results, were calculated in cases where the largest consistent subset contained <75% of the results in the data set. Assigned specific activity values were obtained for ^{60}Co, ^{133}Ba, ^{152}Eu and ^{154}Eu (all gamma-emitting radionuclides) and indicative specific activity values were obtained for ^{3}H (both total ^{3}H and leachable ^{3}H), ^{14}C, ^{40}K, ^{55}Fe and ^{63}Ni. The samples were likely to contain ^{36}Cl and ^{41}Ca as well, but not enough information was received to obtain indicative values for these radionuclides.

2. Material and methods

2.1. Sample preparation and composition

Concrete core samples from a bioshield of a decommissioned nuclear reactor were crushed, mixed and sieved to <0.25 mm to form 6.3 kg of powder. The powder was heated overnight at 110 °C to remove some of the tritium present in the sample as mobile tritiated water. Subsequently, 62 samples (each 100 g nominal in HDPE bottles) were prepared. All samples were measured by gamma spectrometry (high purity Ge detector) at NPL in order to check the homogeneity throughout the entire batch of material.

One sample was analysed for its elemental content by instrumental neutron activation analysis. In total, 35 elements were detected. Three elements (Al, Ca and Si) were found to be present at levels of at least 1 wt%. Eight elements, including Fe [0.649(7) wt%] and Ba [160(16) ppm] were found to be present at levels between 100 ppm and 1 wt%. Nineteen elements, including Co [2.52(8) ppm], were found to be present at levels between 1 ppm and 100 ppm. Five elements, including Eu [0.378(11) ppm], were found to be present at levels lower than 1 ppm. Nickel was not detected and it is likely to be present at levels below its

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doi:10.1016/j.apradiso.2009.11.031

Please cite this article as: Harms, A., Gilligan, C., Development of a neutron-activated concrete powder reference material. Appl. Radiat. Isotopes (2009), doi:10.1016/j.apradiso.2009.11.031

Thank you

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