

Concrete C samples

**NPL Environmental Radioactivity Proficiency Test
Exercise 2007 Workshop**

Arvic Harms

19 September 2007

Not a proficiency test but a comparison (with 27 participants)

Assigned values are the medians of the participant values

C samples originate from samples taken from a concrete bioshield of a decommissioned nuclear reactor which ceased operation ~25 years ago after ~20 years of operation.

The concrete is thought to contain (at least) the following 9 nuclides formed by neutron activation of concrete components:

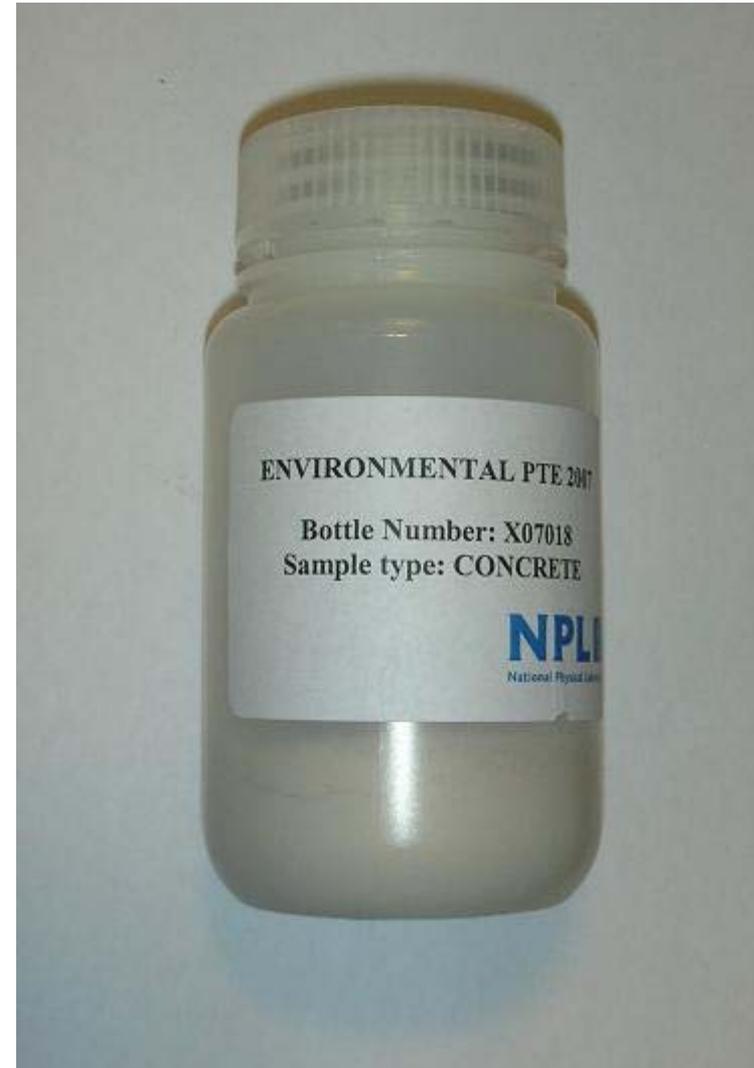
^3H , ^{14}C , ^{41}Ca , ^{55}Fe , ^{60}Co , ^{63}Ni , ^{133}Ba , ^{152}Eu and ^{154}Eu

Sample preparation

The concrete core samples were crushed, mixed and sieved to <0.5 mm to form a homogeneous powder (~ 2.7 kg)

The powder was then heated overnight to 150 °C to remove ^3H present as mobile tritiated water

52 samples (50 g each) were prepared



C composition as determined with neutron activation analysis

| | | |
|----|---------------|-------------------|
| Ca | 26.0(3) % | Ca-41 |
| Si | 15(3) % | |
| Al | 1.21(3) % | |
| Fe | 0.680(8) % | Fe-55 |
| K | 0.53(2) % | K-40 |
| Mg | 0.44(3) % | |
| Na | 0.222(3) % | |
| Ba | 192(12) ppm | Ba-133 |
| Co | 2.70(6) ppm | Co-60 |
| Eu | 0.372(11) ppm | Eu-152 and Eu-154 |

and 26 other elements (including Au, Cs, Th and U)

Homogeneity testing (Co-60, Ba-133, Eu-152 and Eu-154)

15 randomly selected bottles (29% of the bottles) were kept at NPL

14 bottles were measured once

s_{bb} (%)

1 bottle was measured five times

s_{meas} (%)

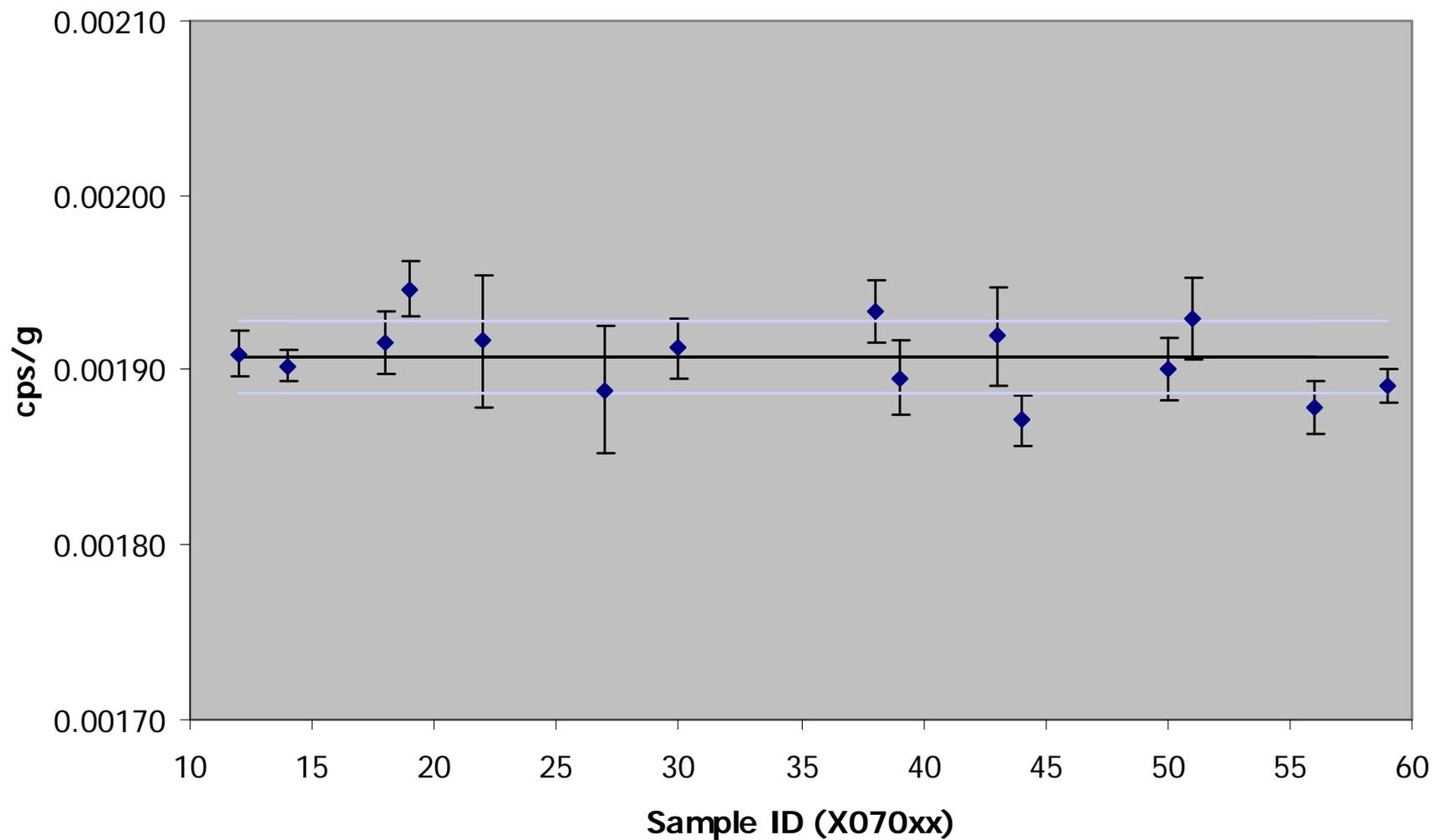
$$u_{bb}^2 = u_{meas}^2 + u_{incho}^2$$

$$u_{incho}^2 = u_{bb}^2 - u_{meas}^2 = s_{bb}^2 - \frac{s_{meas}^2}{5}$$

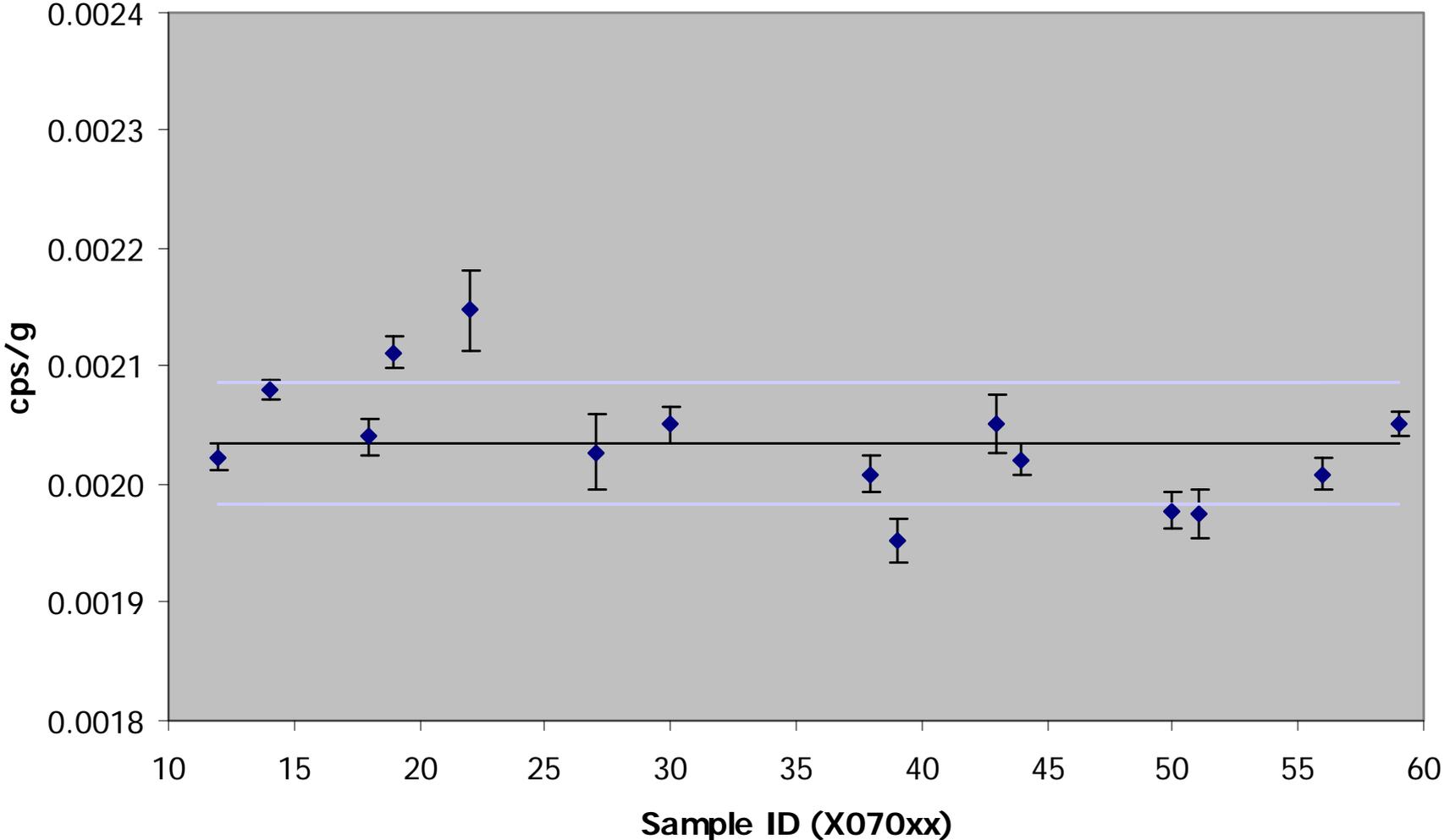
(ideally $u_{incho} = 0$)

$$u_N^2 = u_{med}^2 + u_{incho}^2 = u_{med}^2 + s_{bb}^2 - \frac{s_{meas}^2}{5}$$

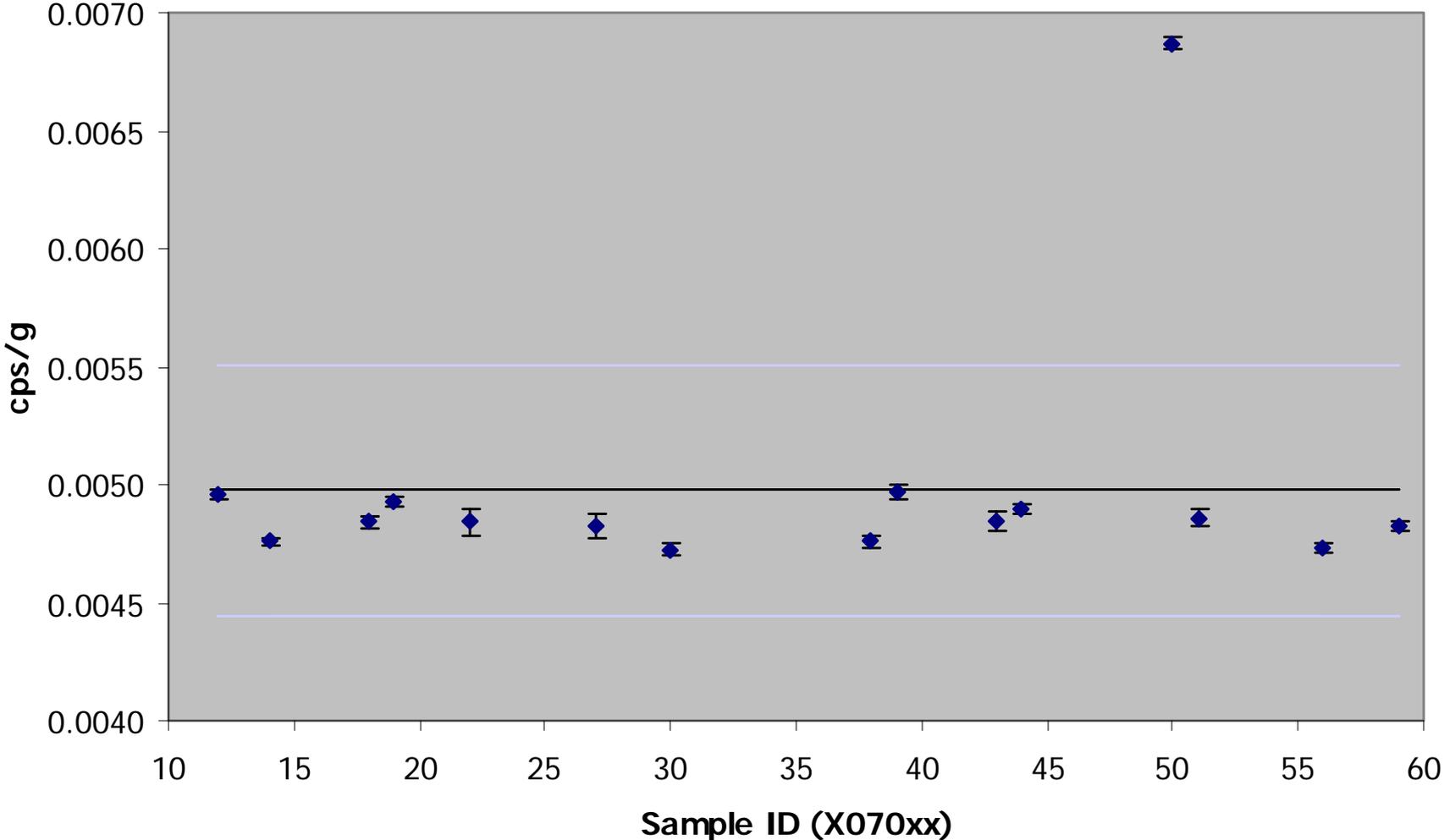
Co-60 homogeneity



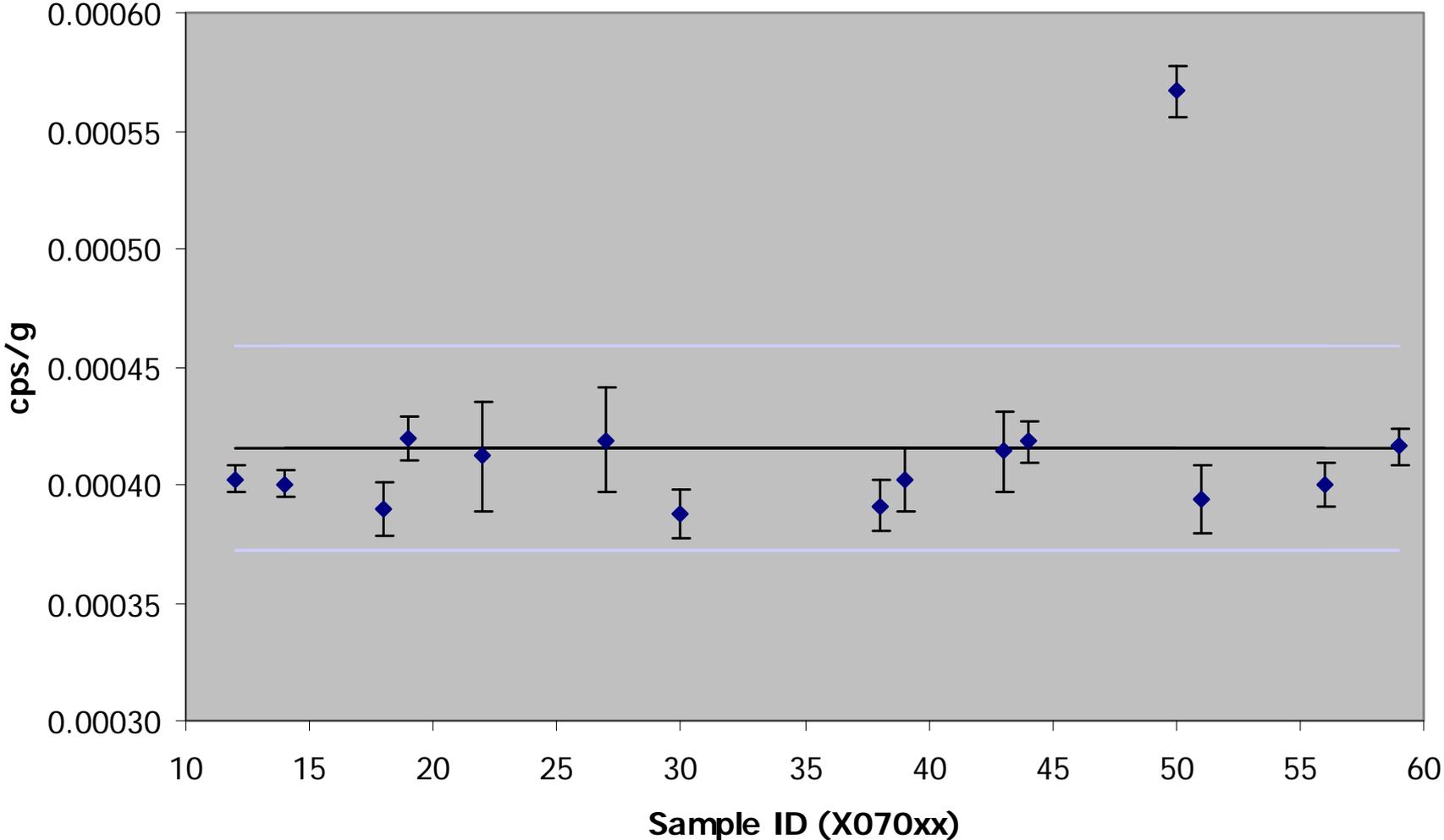
Ba-133 homogeneity



Eu-152 homogeneity



Eu-154 homogeneity



Between-bottle uncertainty (in %)

| | U_{bb} | U_{meas} | U_{inho} | U_{med} | U_N |
|--------|----------|------------|------------|-----------|-------|
| Co-60 | 1.09 | 0.92 | 0.59 | 2.48 | 2.55 |
| Ba-133 | 2.52 | 0.79 | 2.40 | 4.41 | 5.02 |
| Eu-152 | 10.64 | 0.58 | 10.62 | 4.62 | 11.59 |
| Eu-154 | 10.41 | 1.96 | 10.22 | 2.67 | 10.57 |

(without X07050)

| | U_{bb} | U_{meas} | U_{inho} | U_{med} | U_N |
|--------|----------|------------|------------|-----------|-------|
| Co-60 | 1.12 | 0.92 | 0.65 | 2.48 | 2.56 |
| Ba-133 | 2.48 | 0.79 | 2.35 | 4.41 | 5.00 |
| Eu-152 | 1.62 | 0.59 | 1.51 | 4.62 | 4.86 |
| Eu-154 | 2.86 | 1.96 | 2.08 | 2.67 | 3.39 |

Treatment of data (I)

There is no assigned NPL value; instead the median value of the returned results was assigned as N

u_{med} was calculated according:

$$u_{med} = \frac{1.858 \text{ MAD}}{\sqrt{n-1}}$$

MAD (*median of the absolute deviation*)

Only zeta and z tests were performed

z test was modified for all nuclides except ^{60}Co and ^{133}Ba

Acceptance criteria were made nuclide specific (because there are a function of the degrees of freedom)

Treatment of data (II)

- (i) Zeta test
pass if $\text{Abs}(\text{zeta}) \leq t \text{ value}$ $P = 0.01$

$$\zeta = \frac{L - N}{\sqrt{u_L^2 + u_N^2}}$$

- (iii) z test
pass if $\text{Abs}(\text{z test}) \leq t \text{ value}$ $P = 0.01$

$$z = \frac{L - N}{u_N}$$

(⁶⁰Co and ¹³³Ba only)

$$z = \frac{L - N}{R_{\text{med}} N}$$

Treatment of data (III)

zeta test

z test

pass

pass

'In agreement'

fail

pass

'Questionable'

pass

fail

'Questionable'

fail

fail

'Discrepant'

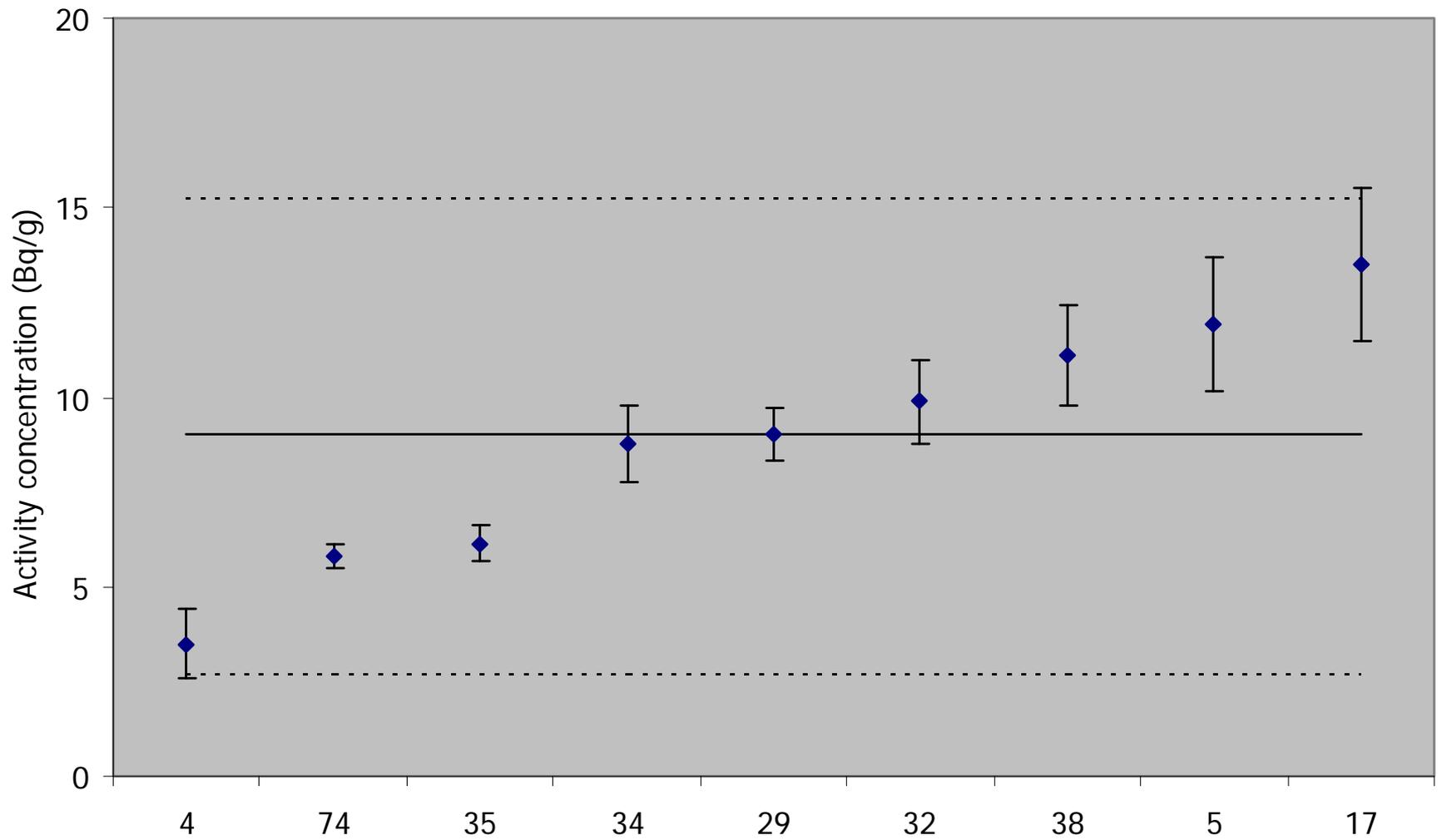
Results (I)

| | (Bq g ⁻¹) | Results | In agreement |
|--------------------------------|-----------------------|----------|--------------|
| ³H leachable | 0.9(7) | 5 | 60% |
| ³H total | 9.0(19) | 9 | 100% |
| ¹⁴C | 0.16(12) | 5 | 100% |
| ⁴⁰K | 0.18(4) | 6 | 67% |
| ⁴¹Ca | 0.39(19) | 3 | 100% |
| ⁵⁵Fe | 0.037(22) | 3 | 100% |
| ⁵⁸Co | - | 3 | - |
| ⁶⁰Co | 0.0750(19) | 26 | 73% |
| ⁶³Ni | 0.0170(24) | 3 | 67% |
| ¹³³Ba | 0.054(3) | 24 | 71% |
| ¹³⁷Cs | 0.00110(23) | 2 | 100% |

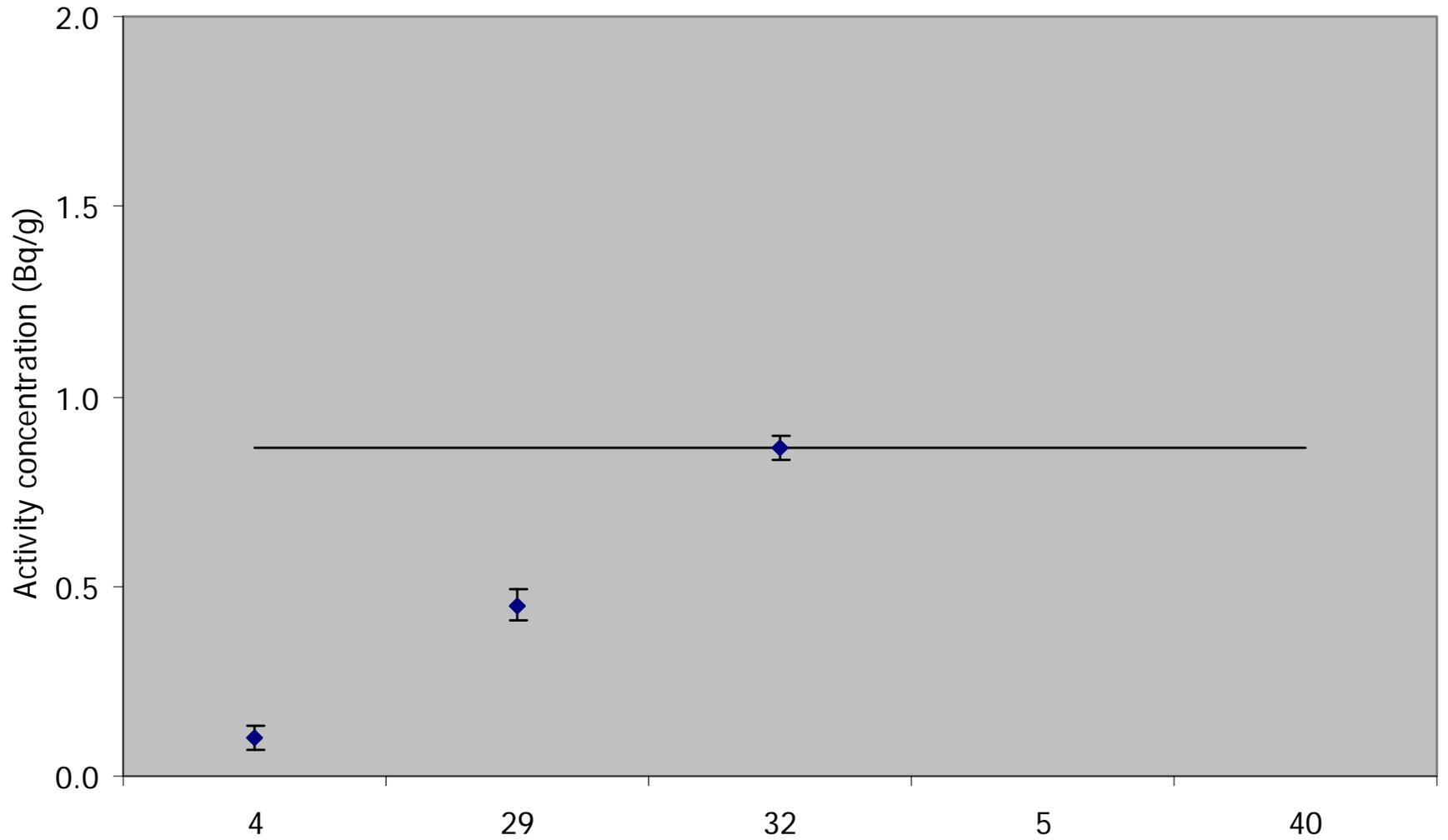
Results (II)

| | (Bq g ⁻¹) | Results | In agreement |
|-------------------|-----------------------|---------|--------------|
| ¹⁵² Eu | 1.16(13) | 25 | 88% |
| ¹⁵⁴ Eu | 0.048(5) | 21 | 86% |
| ²¹⁰ Pb | 0.60(3) | 1 | - |
| ²²⁶ Ra | 0.05020(19) | 2 | 100% |
| ²²⁸ Ra | 0.0102(23) | 2 | 100% |
| ²²⁸ Th | 0.0060(3) | 1 | - |
| ²³⁴ Th | 0.037(4) | 1 | - |

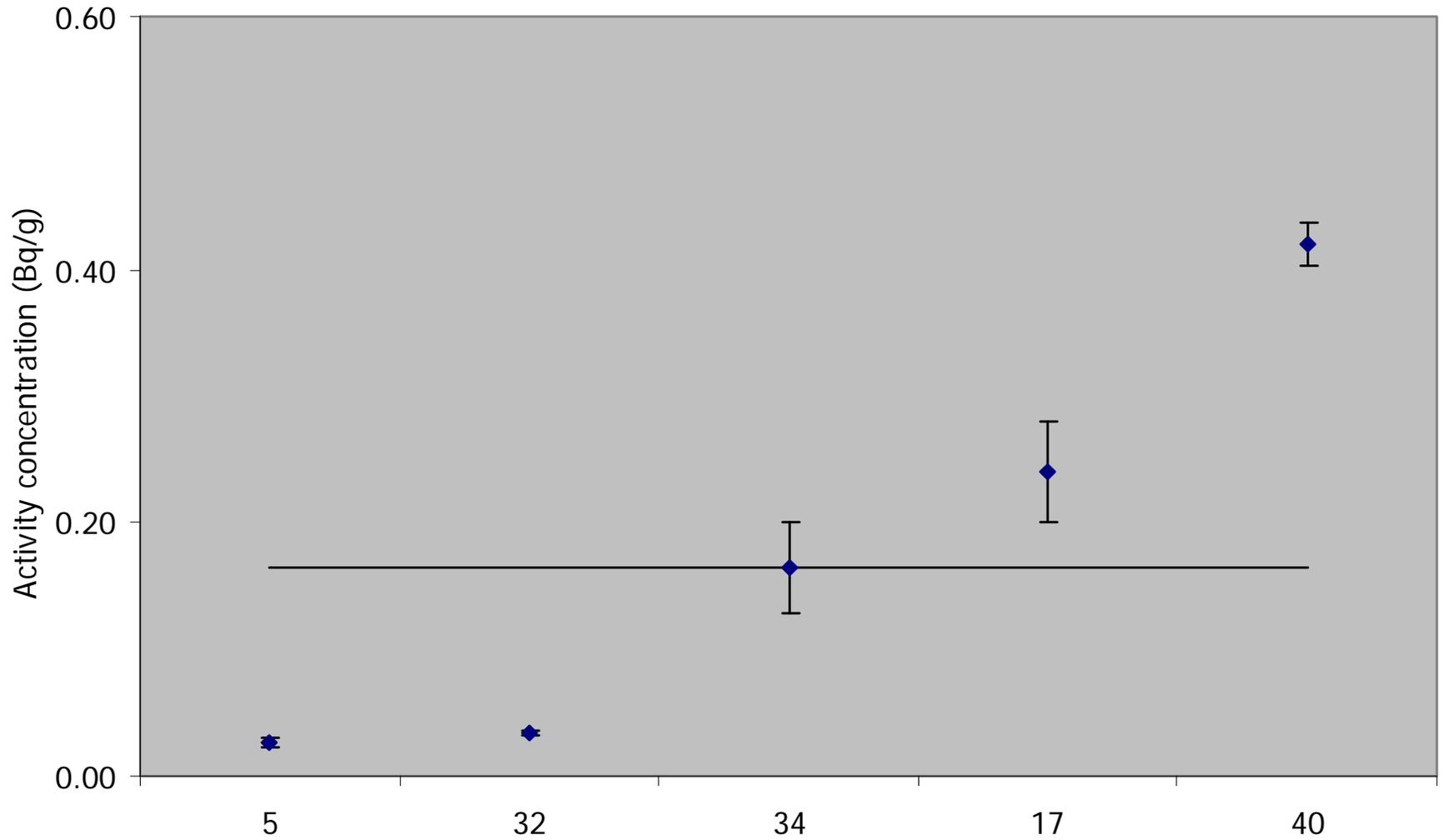
H-3 total C



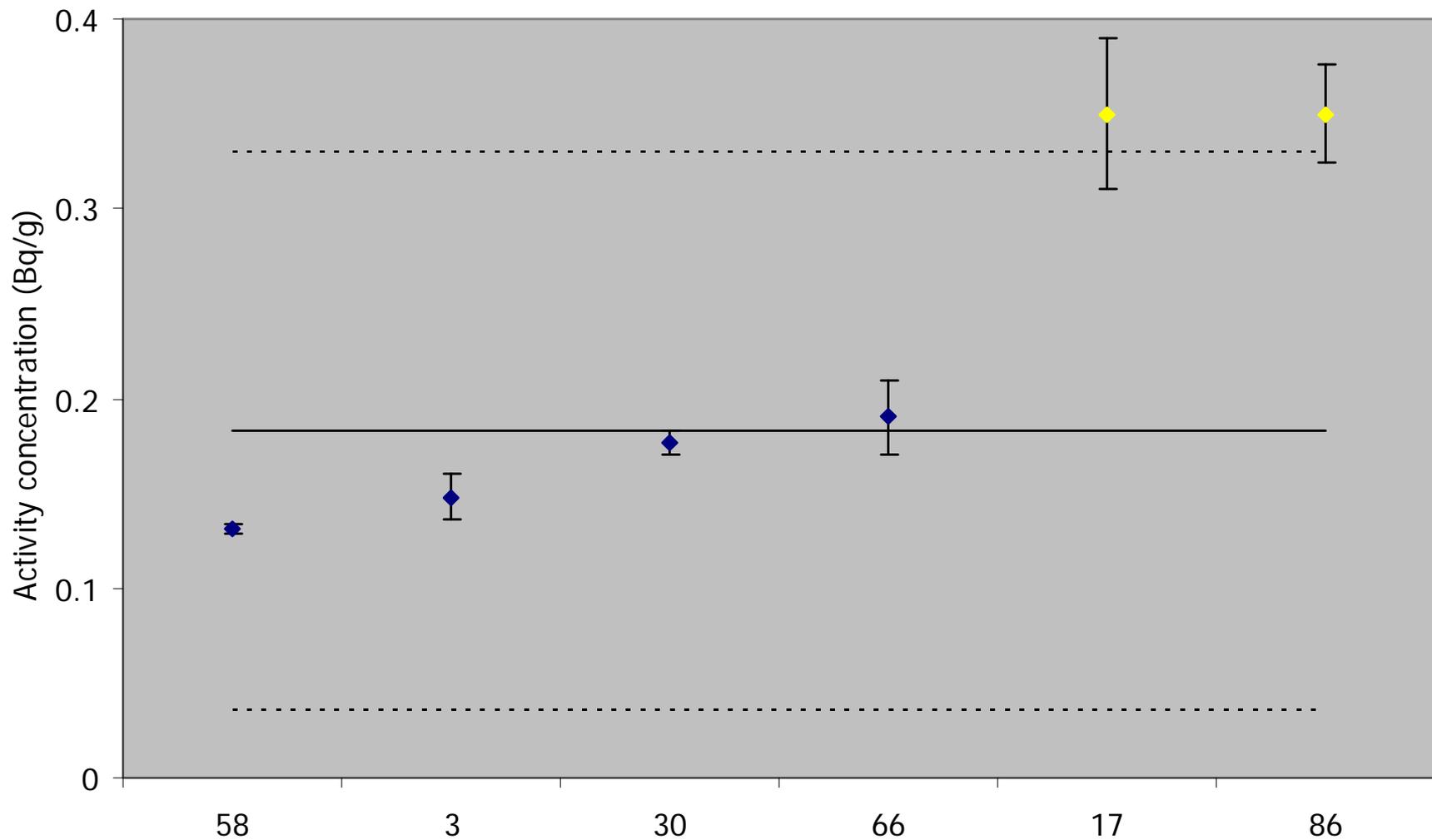
H-3 leach C



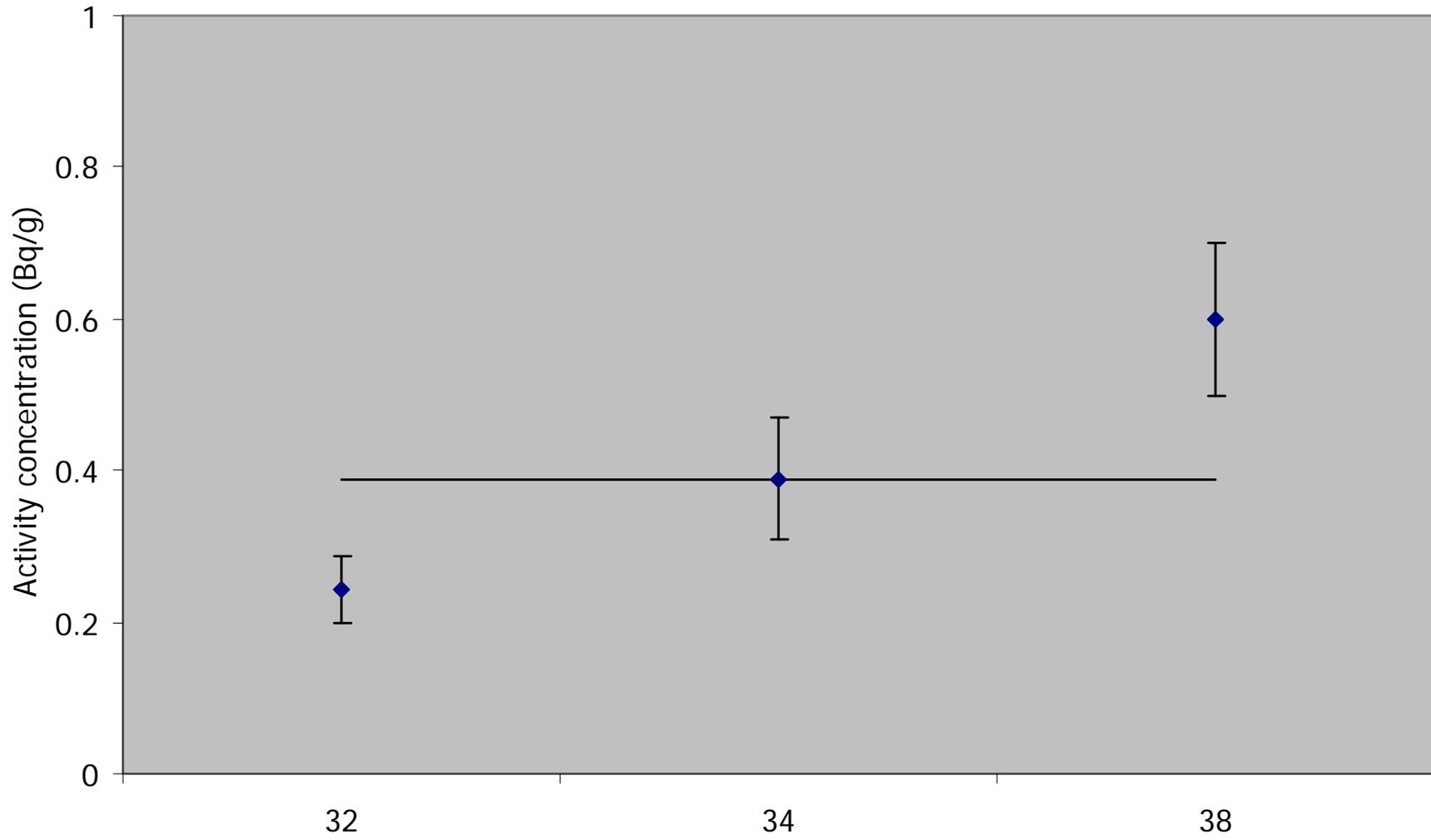
C-14 C



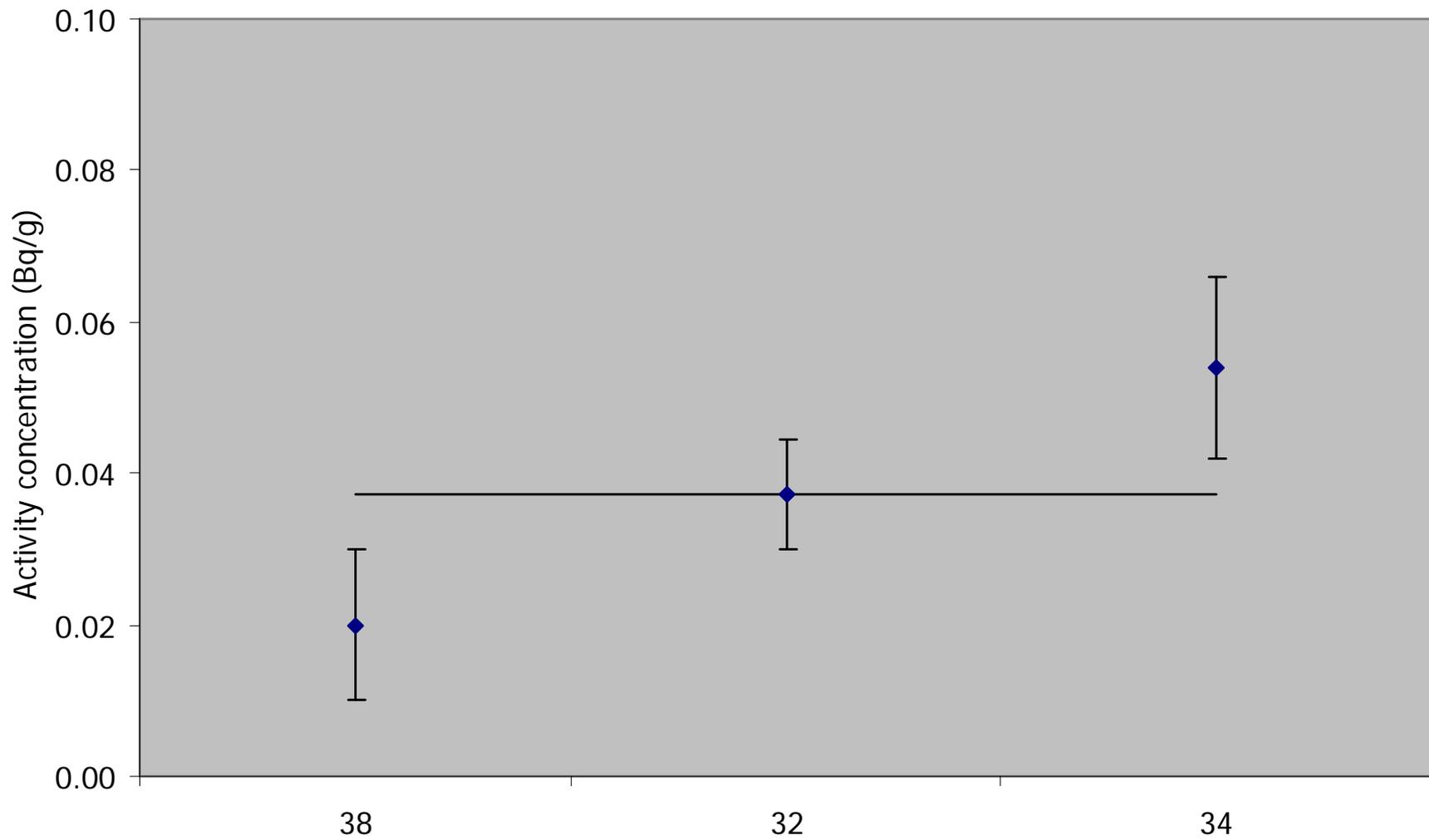
K-40 C



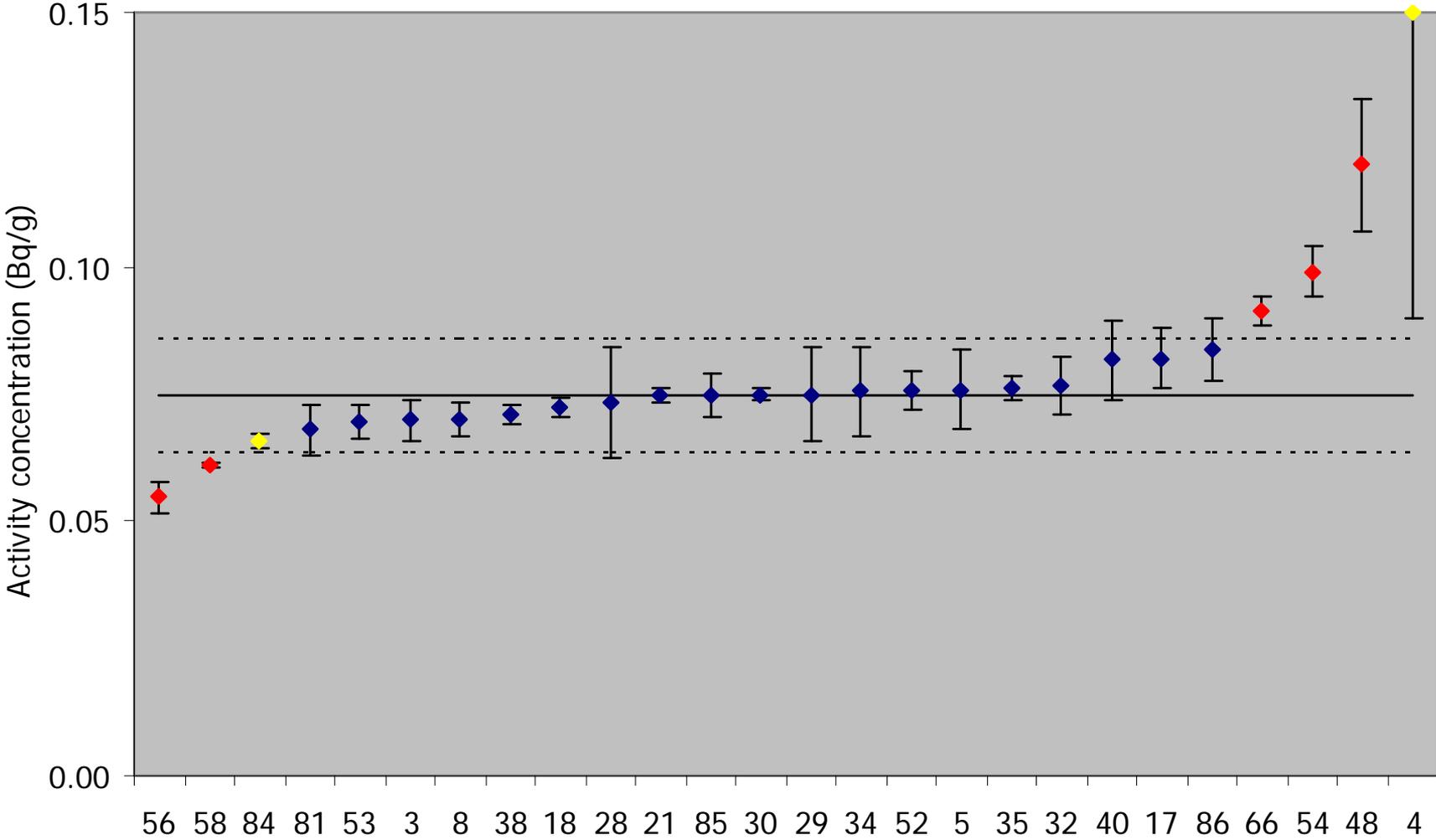
Ca-41 C



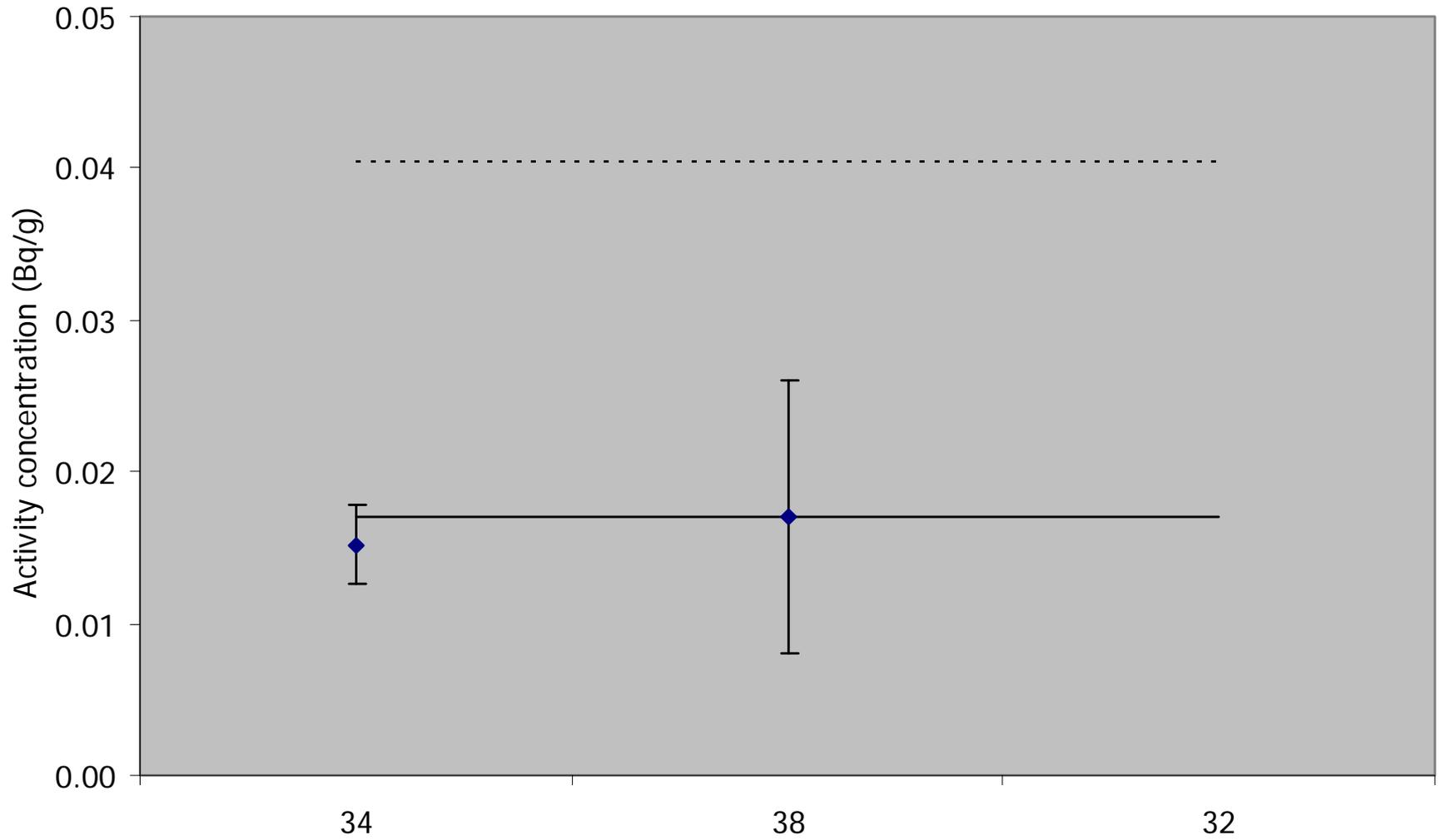
Fe-55 C



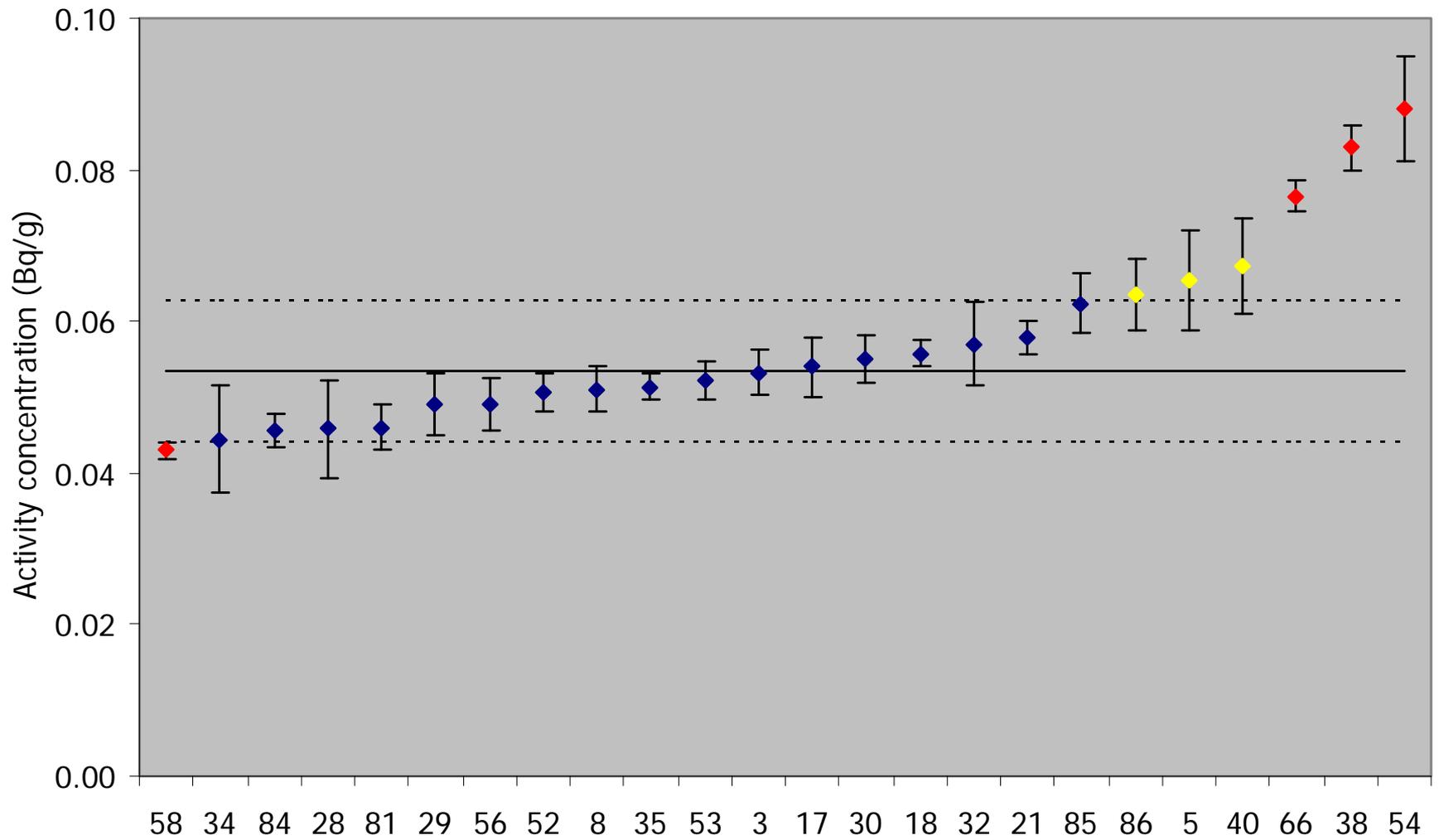
Co-60 C



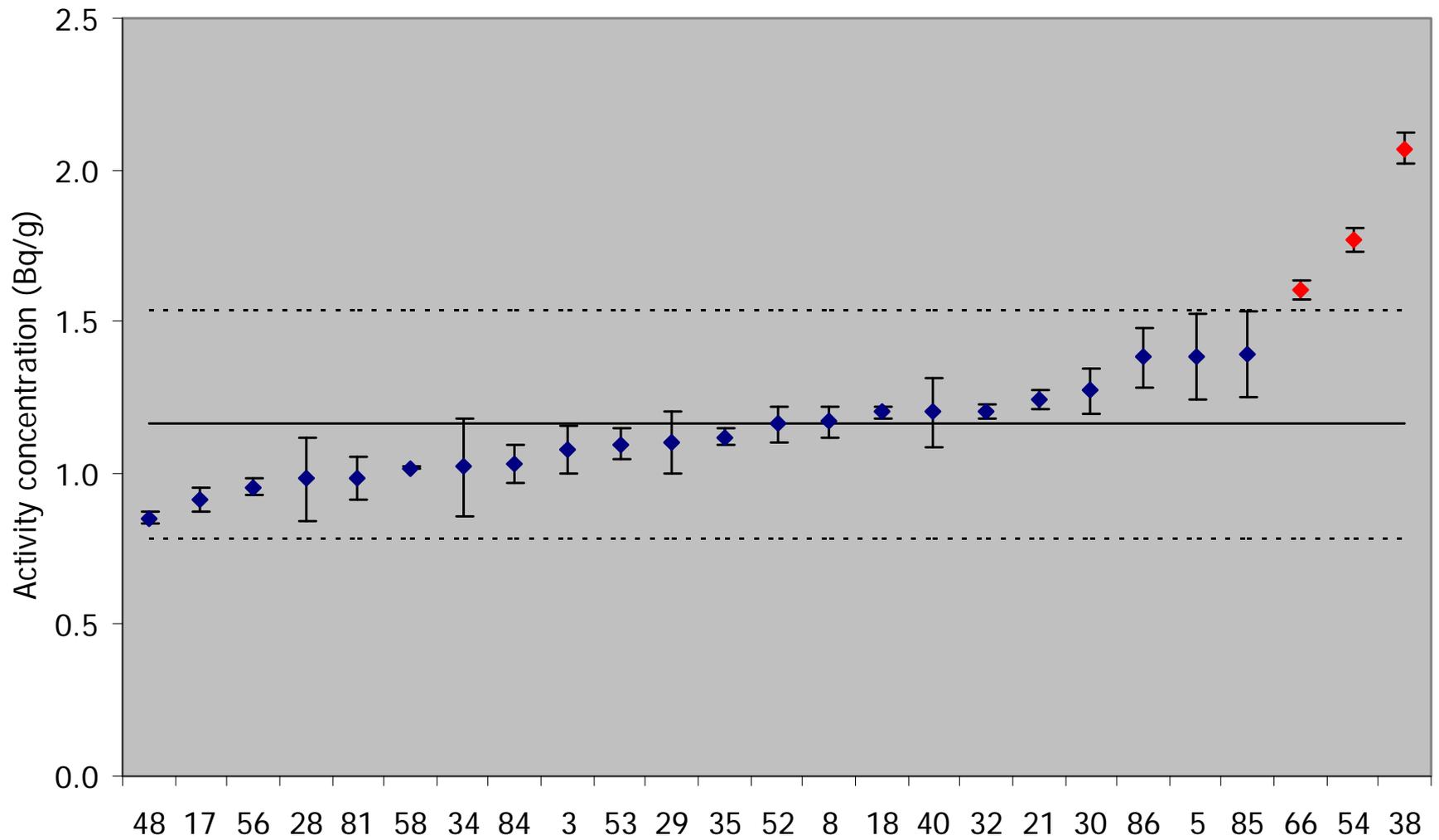
Ni-63 C



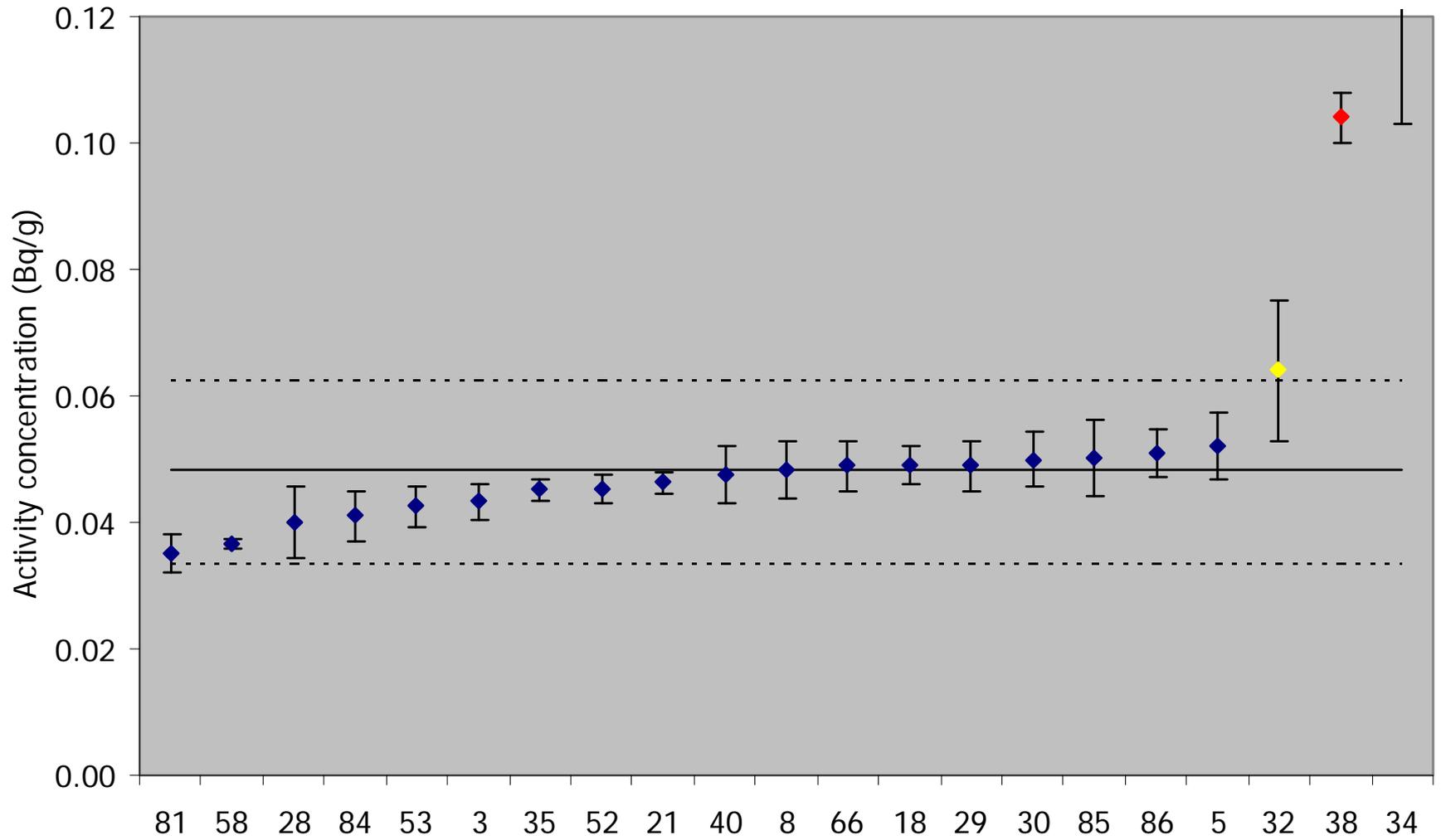
Ba-133 C



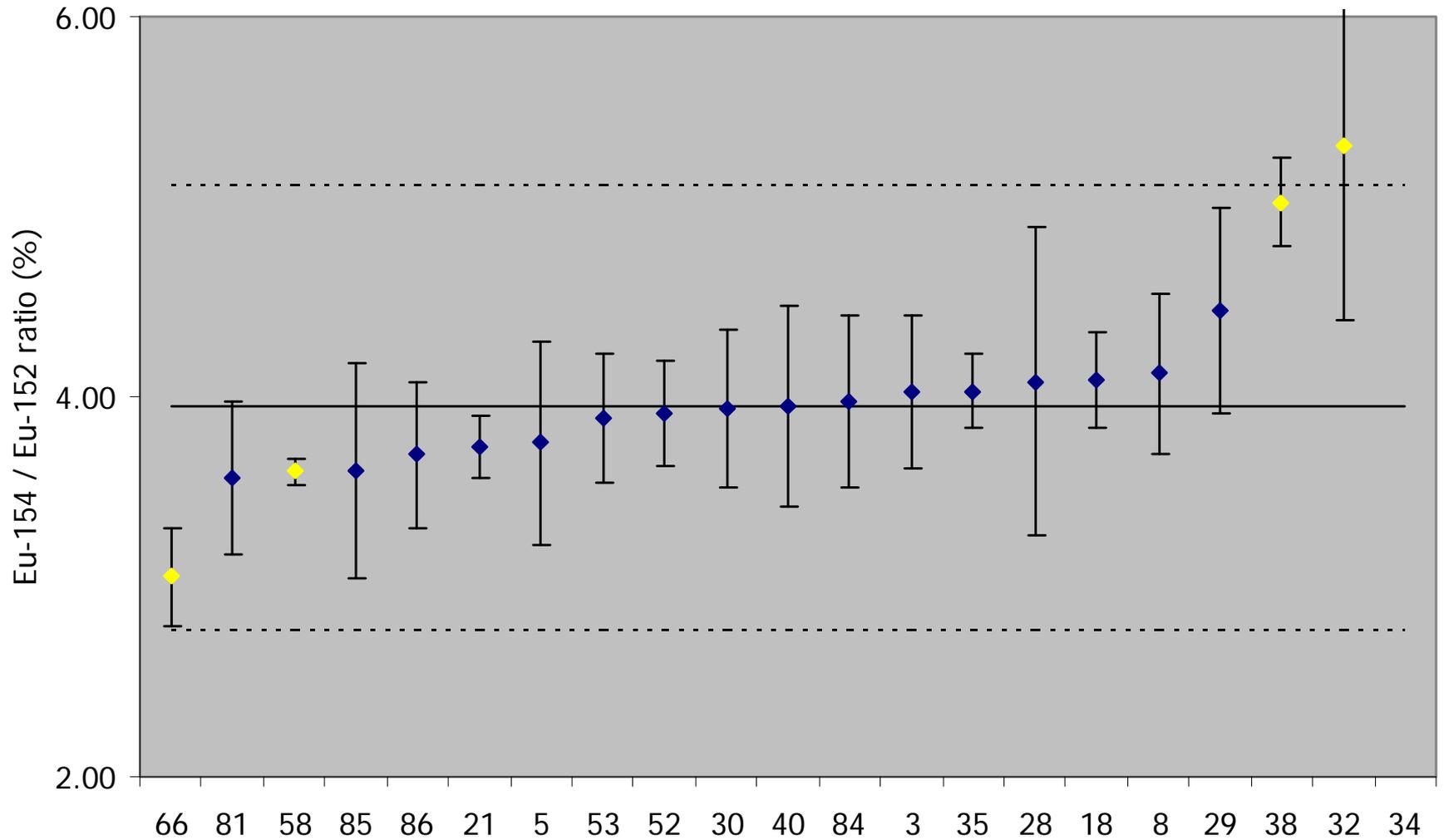
Eu-152 C



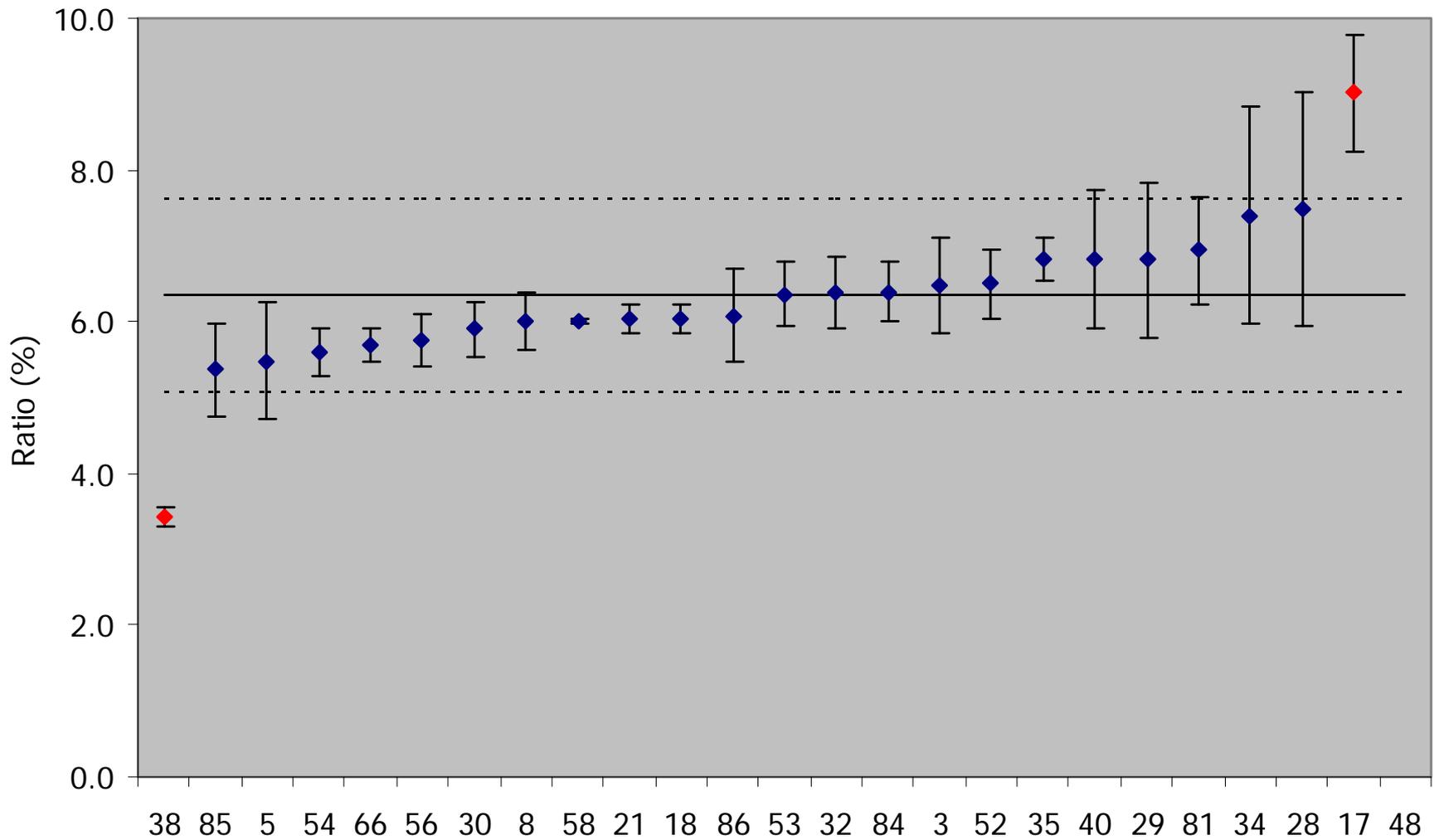
Eu-154 C



Eu-154 / Eu-152 ratio C



Co-60 / Eu-152 ratio C



Results

| | |
|-----|------------------|
| 80% | 'in agreement' |
| 7% | 'questionable' |
| 11% | 'discrepant' |
| 2% | 'false positive' |

Eu-152/154 inhomogeneity affecting the exercise?

One sample (X07050, which was kept at NPL) had a different composition than the other 14 samples tested (containing a “hot” Eu-152/154 particle?)

If similar samples were sent-out (~7% chance) then this may explain the “Q” and “D” Eu-152/154 results for Labs 32, 34, 38, 54 and 66

However, the Eu ratios for Labs 32, 34, 38 and 66 are all “Q”
Lab 54 did not report Eu-154 and had “D” results for Co-60 and Ba-133 as well

Sample X07051 (which was prepared directly after X07050) was normal

Labs 32, 34, 38, 54 and 66 analysed X07053, X07035, X07040, X07052 and X07032, respectively

The inhomogeneity component was incorporated in u_N

Thank you