

Double separation of Ni-63



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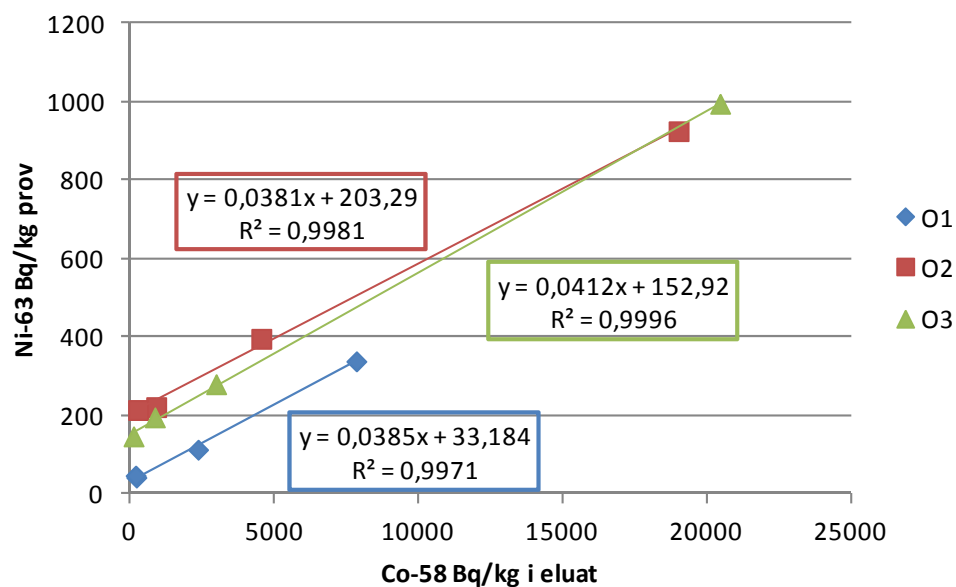
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Presentation Outline

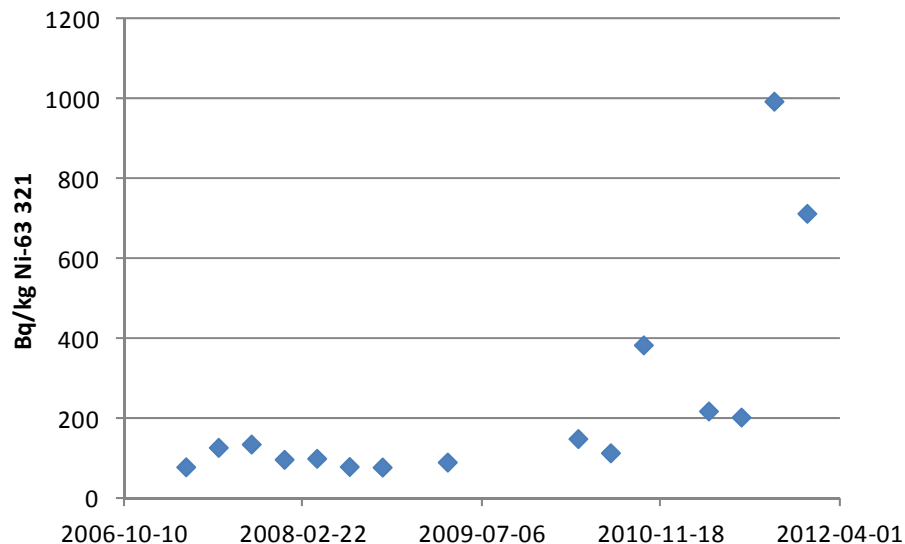
- Problem description
- Analyses
- Results
- Recommendations



First analyses of Ni-63

- Ni-63 is measured in reactor water (321), spent fuel pool water (324) and waste water (342) since 2011.
- Early the problem of interference from Co-58 in the liquid scintillation counting was identified (adjustments for Co-60 were already included). The last quarters in 2011 showed higher “Ni-63” activity levels than for the earlier quarters.

(samples below analysed in the end of 2011 / beginning of 2012)



- Quarter 3 2011
 - High level Co-58
 - Analysed 111017
- Quarter 4 2011
 - High level Co-58
 - Analysed 120213



Three alternatives to solve the problem with Co-58 interference

- Wait with the analyses until most of the Co-58 has decayed (half-life 71 days...)
Not possible because SSM wants their reports on time
- Adjust for Co-58 with a quench correction curve in the same way as for Co-60
Possible, but a bit complicated to use a relatively short-lived nuclide and adding a lot of extra uncertainty to the results (adjusting the total amount of counts for the background and for Co-60 already)
- Use “double” separation, i.e. to allow the sample to pass the Ni-specific column twice (approximately two hours extra work) in combination with a reasonable time between sampling and analysis.

Tested in this work and the best alternative!



Analytical method

- The analytical method is based on solid-phase-extraction (Eichrom columns) where Fe and actinides are separated using a TRU-resin followed by a separation step where Ni is selectively separated from the rest using a Ni-specific resin.
- Where Ag-110m is present an early precipitation step (AgCl) is included.
- The analytical yield is determined using ICP-OES measuring stable Ni (which is added in the beginning of the sample preparation).
- Amounts of interfering nuclides (presently only Co-60) are estimated using gamma spectrometry and a quench correction curve.
- Ni-63 is measured with liquid scintillation.



Analyses

Single and double separation

- **321 O3 quarter 1 2012
analyzed 121022**
- **321 O3 quarter 3 2012
analyzed 121107**

Long time test

- **321 O1+O2+O3
quarter 3 2011
analyzed in:**
 - **Oct 2011**
 - **Jan 2012**
 - **Apr 2012**
 - **Oct 2012**



321 O3 quarter 1 2012

- **Single separation**
 - In sample: 13 520 Bq/kg Co-60 (**high**) and 2 756 Bq/kg Co-58 (**low**)
In eluate: 2 912 Bq/kg Co-60 (1.6 %) and 662 Bq/kg Co-58 (1.3 %)
 - LSC: 672 of 3 475 cpm from Co-60 (20 %)
 - Ni-63: 1 070 Bq/kg
- **Double separation**
 - In sample: 13 520 Bq/kg Co-60 (**high**) and 2 756 Bq/kg Co-58 (**low**)
In eluate: 104 Bq/kg Co-60 (0.04 %) and 31 Bq/kg Co-58 (0.06 %)
 - LSC: 24 of 2 347 cpm from Co-60 (1 %)
 - Ni-63: 1 015 Bq/kg
- Approximately the same result for **Ni-63** but lower uncertainty because of lower activity for Co-60. No significant effect from Co-58.
- What happens when Co-58 is **high**?

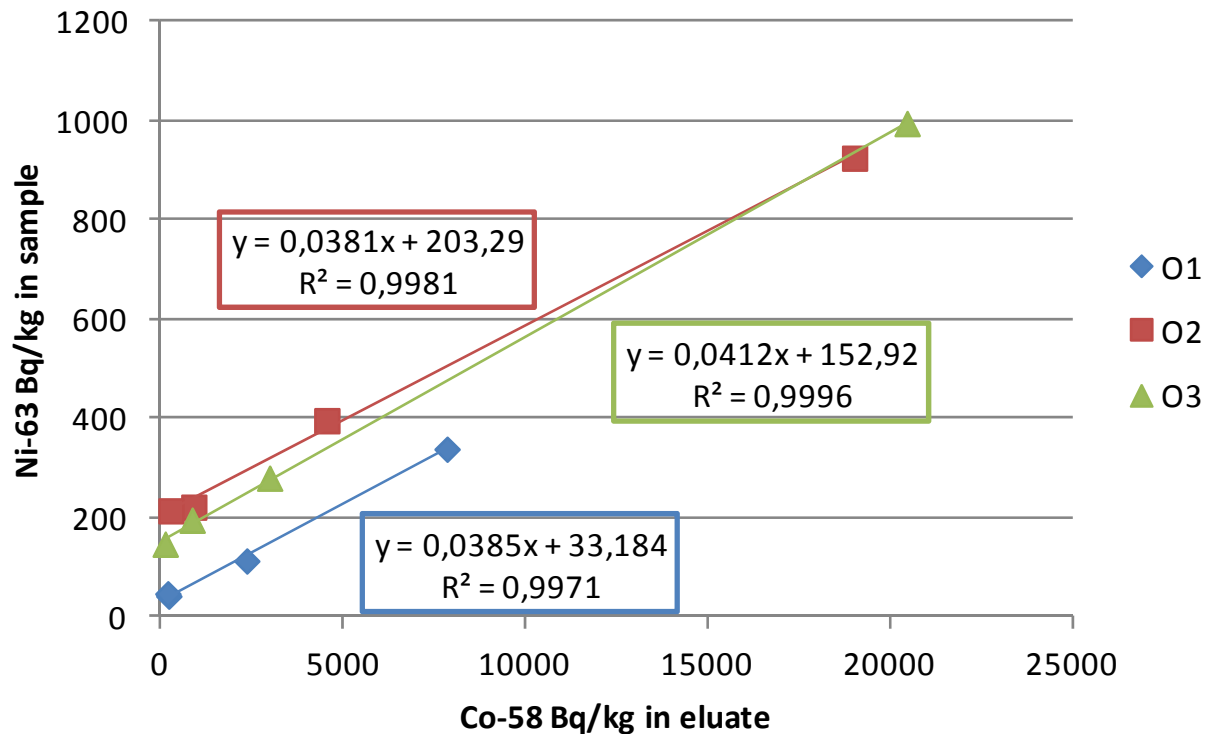


321 O3 quarter 3 2012

- **Single separation**
 - In sample: 3 349 Bq/kg Co-60 (**low**) and 9 729 Bq/kg Co-58 (**high**)
In eluate: 768 Bq/kg Co-60 (1.2 %) and 1 902 Bq/kg Co-58 (1.1 %)
 - LSC: 175 of 576 cpm from Co-60 (30 %)
 - Ni-63: **168 Bq/kg**
- **Double separation**
 - In sample: 3 349 Bq/kg Co-60 (**low**) and 9 729 Bq/kg Co-58 (**high**)
In eluate: 54 Bq/kg Co-60 (0.09 %) and 109 Bq/kg Co-58 (0.06 %)
 - LSC: 12 of 240 cpm from Co-60 (5 %)
 - Ni-63: **86 Bq/kg**
- The result for **Ni-63** is significantly changed by using double separation. The effect of Co-58 (not adjusted for) contributes.
- **How large is the effect of Co-58?**



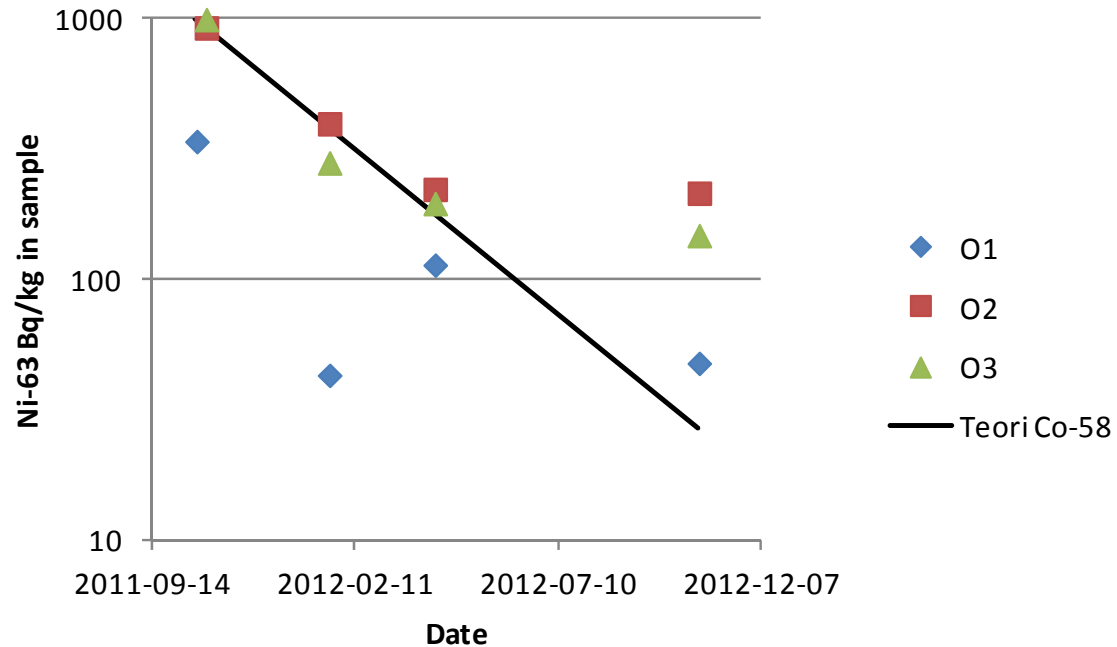
Long time test



The linear relationship indicates that the results for Ni-63 is directly proportional to the Co-58 activity in the eluate... Where Co-58 is zero the "true" Ni-63 activity can be estimated.



Long time test



**The “true” Ni-63 is reached when Co-58 has decayed.
The theoretical decay for Co-58 is indicated in the graph.
(No conclusions for the data from O1.)**



Results and recommendations

- **Co-58 directly affects the determination of Ni-63**
Through double separation a smaller amount of Co-58 will be reported as Ni-63.
- **Co-60 indirectly affects the determination of Ni-63**
Through double separation the uncertainty from the Co-60 adjustment will be decreased.
- **The combination of waiting with the analyses and using double separation will ensure that Ni-63 is not overestimated by more than 20 %.**
Especially in samples with a lot of Co-58. From our data the recommendation is to wait at least six weeks before starting the analysis.

