Intercomparison exercise in analysis of DTM in decommissioning waste

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- Difficult to measure radionuclides (DTM)
- Intercomparison exercises for analysis validation
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Characterisation in decommissioning projects

Characterisation in decommissioning projects

Waste characterisation

Prior to dismantling

- Sampling
- Easy-to-measure (ETM) – gamma spectrometry
- Difficult-to-measure (DTM) – radiochemical analyses

Scaling factor

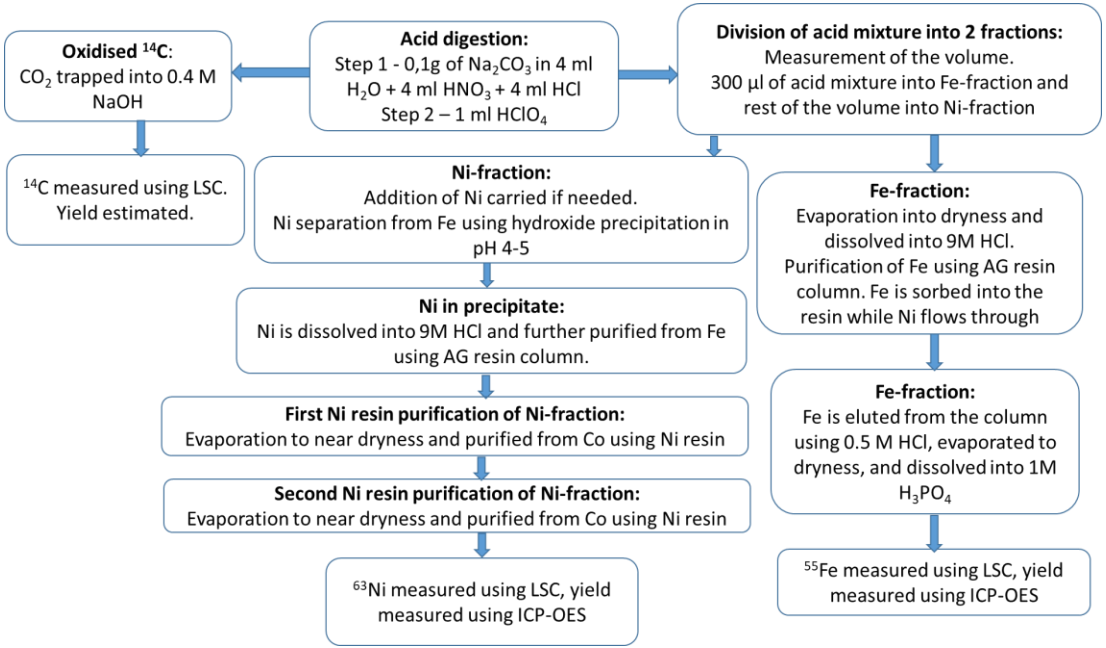
- Material specific
- DTM to key ETM radionuclide ratio

During dismantling

- ETM measurement of packed material
- Implementation of scaling factor for DTM

DTM – why are they difficult to measure?

- Sampling
  - Representative sample
  - Correct sampling technique
- Radiochemical analysis
  - Variety of materials and radionuclides
  - Low and high activities
  - Long radiochemical procedures
  - Solubility
  - Interferences
  - LSC quenching



Validation of the radiochemical analyses

Spiked samples vs real samples

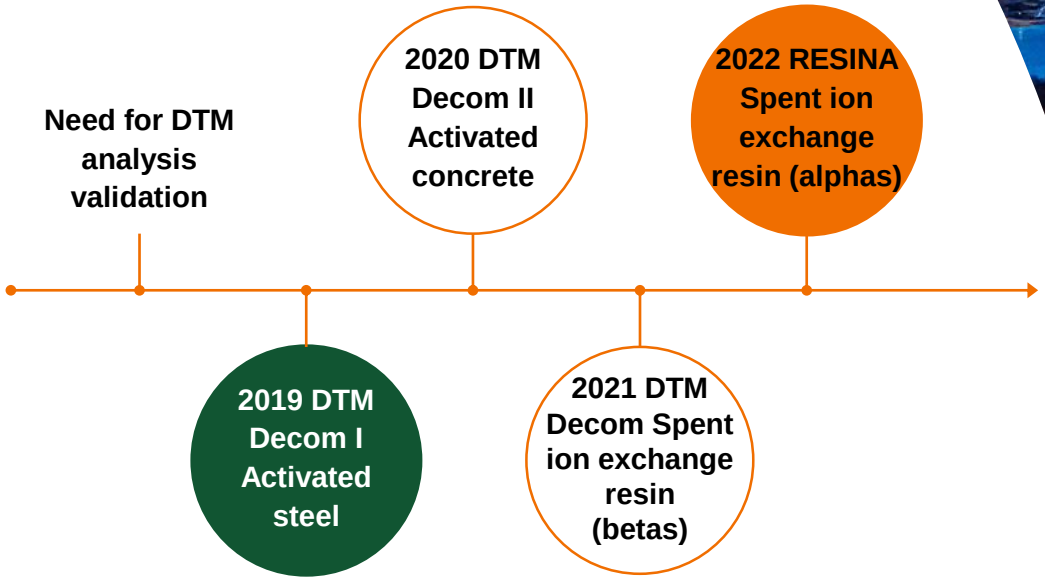
Intercomparison exercises for analysis validation

Intercomparison exercises

nks
Nordic nuclear safety research
http://www.nks.org/

Finland (3)
Sweden (1)
Denmark (1)
Norway (3)

France (1)
Taiwan (1)
UK (1)



Intercomparison exercises

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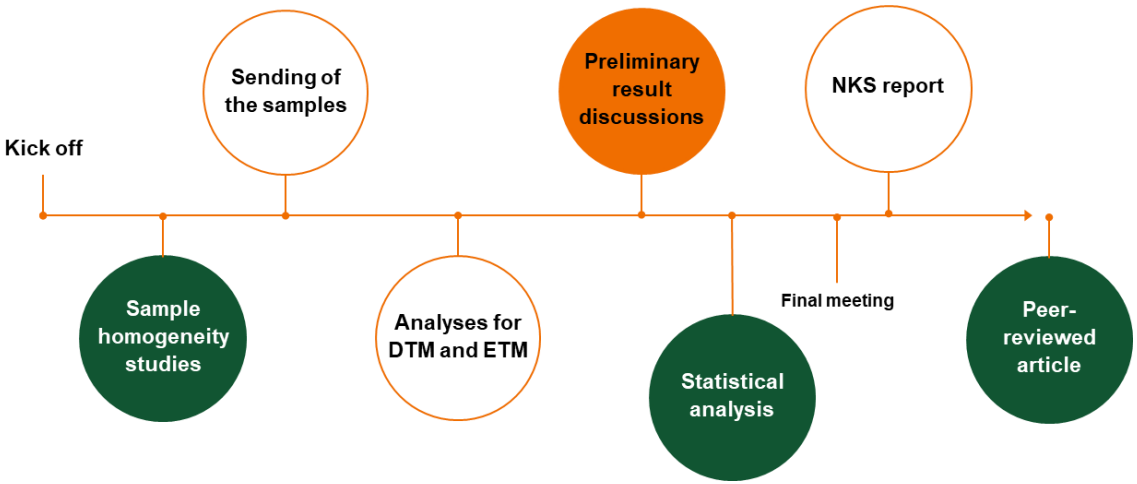


Analysis validation

- Statistical analysis according to ISO standard[1]
- Assigned values calculate from participants' results
- Comparison of performance using z score

$$z_i = (xi - xpt)/\sigma_{pt}$$

x_{pt}= the assigned value
σ_{pt}= standard deviation for the proficiency assessment

Z score	Analysis result
$\|z\| \leq 2.0$	Acceptable
$2.0 < \|z\| < 3.0$	Warning signal
$\|z\| \geq 3.0$	Unacceptable

[1] International Standard ISO 13528:2015(E) (2015) Statistical methods for use in proficiency testing by interlaboratory comparison

DTM Decom I – activated steel[2,3]

- Reactor Pressure Vessel (RPV) steel
  - 1 cm x 1 cm thin slices
  - 0.1-0.2 g
- Homogeneity check using gamma spectrometry
  - 1.8 RSD%
- 2-3 samples for each participant
- Radionuclides
  - Fe-55 and Ni-63
  - Optional C-14 and Co-60
- Additionally activation calculation
  - Neutron dosimetry samples

[2] Leskinen et al. (2020) NKS-B report NKS-429
[3] Leskinen et al. (2020) JRNC 324:1303-1316

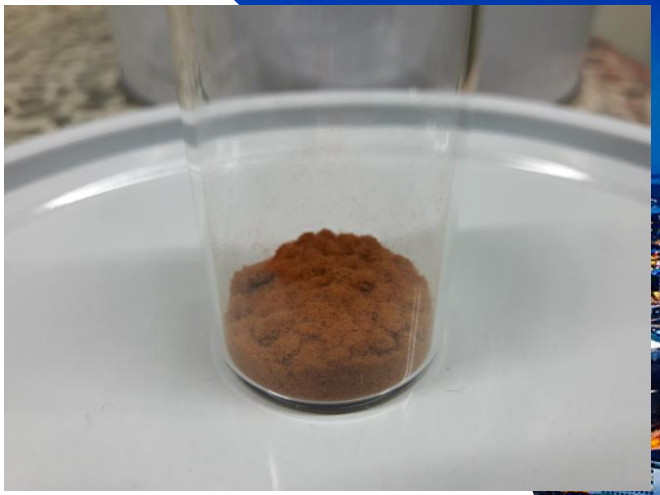
DTM Decom II – activated concrete[4,5]

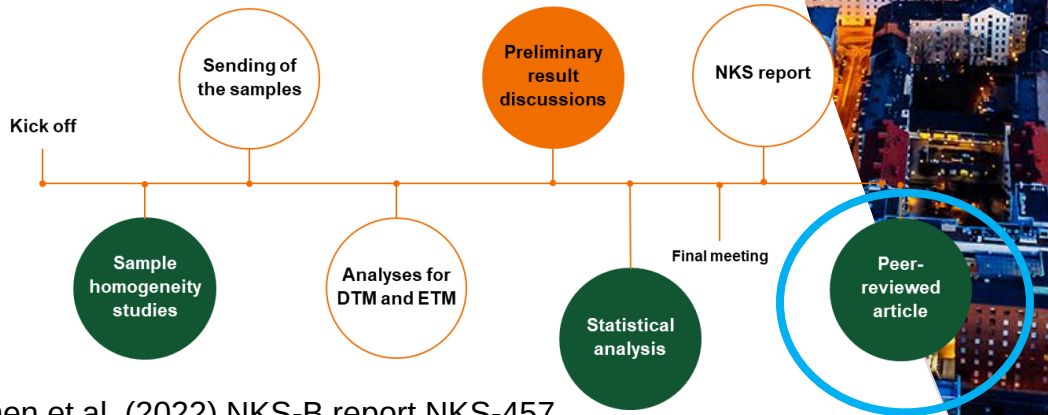
- Biological shield activated concrete
- Homogeneity
  - 1.7 RSD% Eu-152
- 20 g / participant
- Radionuclides
  - H-3, C-14, Fe-55 and Ni-63
  - Optional: Ca-41, Cl-36, Co-60, Eu-152
- Additionally activation calculation
  - Reactor history, neutron fluxes and chemical composition

[4] Leskinen et al. (2021) NKS-B report NKS-441
[5] Leskinen et al. (2020) JRNC 329:945-958

DTM Decom III – spent ion exchange resin [6]

- Spent ion exchange resin
- Homogeneity
  - 0.9 RSD% Co-60
- 0,8 g / participant
- Radionuclides
  - Fe-55, Ni-63, Sr-90, gammas
  - Optional H-3, C-14, Tc-99





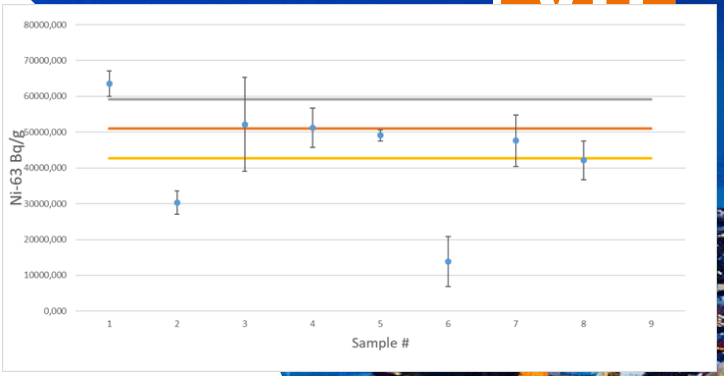
[6] Leskinen et al. (2022) NKS-B report NKS-457

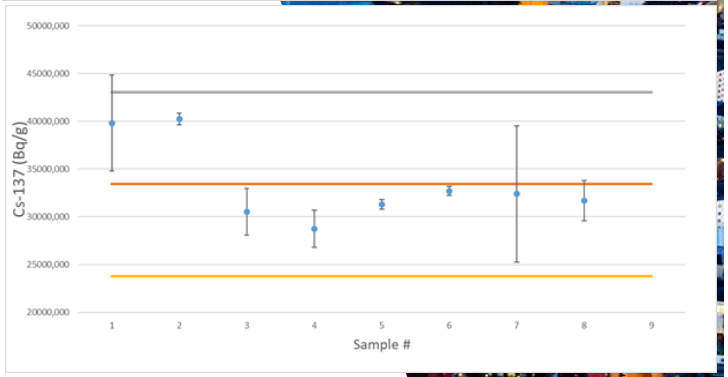
DTM Decom III – spent resin betas[6]

- Excerpt of results shown from final meeting
- Updated for the NKS report and upcoming publication
- Mainly good results for Fe-55, Ni-63, and Sr-90
- Excellent results for gammas

Sample #	Z score		
	Fe-55	Ni-63	Sr-90
1	0,6	1,6	-
2	0,9	5,1	5,3
3	0,4	0,2	0,5
4	1,4	0,0	1,2
5	0,3	0,2	0,9
6	0,3	4,6	-
7	0,2	0,4	0,1
8	1,3	1,1	0,8
9	-	-	-

Sample #	Z score		
	Mn-54	Co-60	Cs-137
1	1,3	1,2	1,3
2	1,4	1,5	1,4
3	0,7	0,6	0,6
4	1,1	0,9	1,0
5	0,2	0,1	0,4
6	0,4	0,7	0,1
7	0,1	0,1	0,2
8	0,2	0,4	0,4
9	-	-	-





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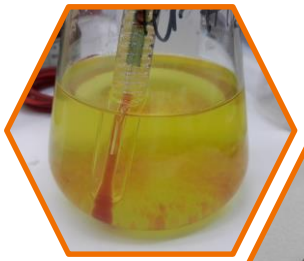
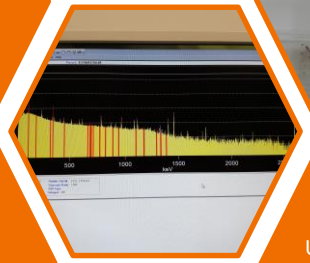
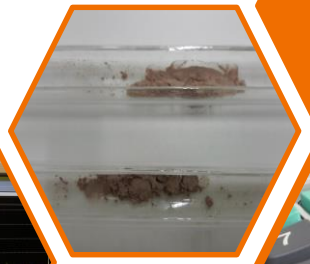
DTM Decom I – III & lessons learned



Oxidative acid digestion vs thermal combustion for volatile DTMs





Sr-90 measurement with Cerenkov or LSC. Can be time consuming.

High Fe content, EC decay & short half-life of Fe-55

H-3 and C-14 is challenging due to volatility and trapping

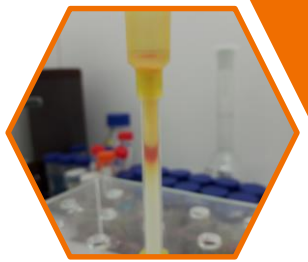
Uncertainty and limit of detection calculations

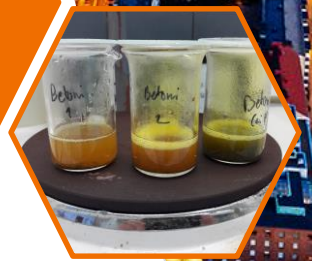
Collaboration, discussions and knowledge sharing

Ni-63 interference by Co-60

Easy to measure radionuclides are easier (danger of false positives, efficiency calibration, coincidence)

Input data in modelling





Conclusions

Conclusions

- Method validation is crucial
- High advantage in using real materials
- High advantage with collaborative project
- Interest of joining is increasing

- DTMs are still difficult to measure
- ETMs are easier than DTMs

beyond

the obvious

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