

Development of Reference Materials

INSIDER Final Workshop

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Reference material production: Case studies

- Apply the methodologies developed to real sites under decommissioning
 - Work based around case studies
- 1. Decommissioning of a back/end fuel cycle and/or research facility (JRC Ispra)**
 - 2. Decommissioning of a nuclear reactor (SCK/CEN)**
 - Development of two simulated spiked materials and real materials from case study sites



Reference Materials Guidance

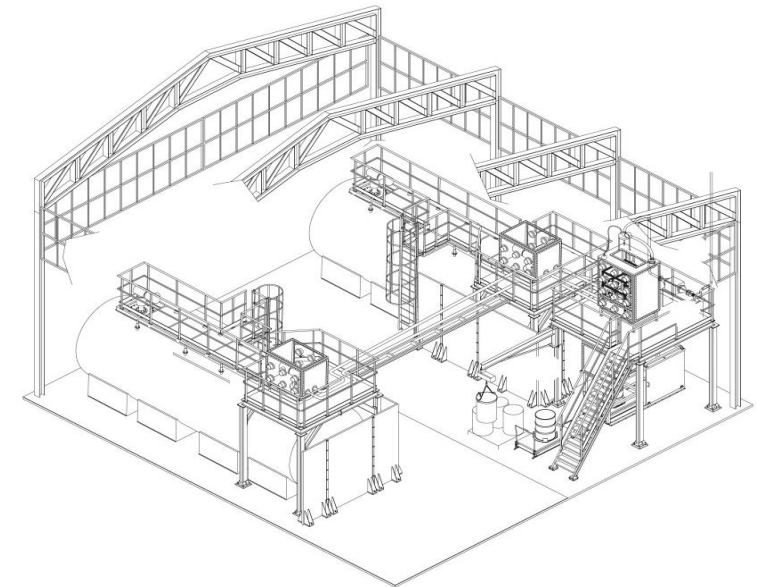
- **ISO Guide 30: 2015:** Selected terms and definitions
- **ISO Guide 31: 2015:** Contents of certificates and labels and accompanying documentation
- **ISO Guide 33: 2015:** Good practice in using reference materials
- **ISO Guide 34: 2009:** General requirements for the competence of reference materials producers
- **ISO Guide 35: 2017:** Guidance for characterization and assessment of homogeneity and stability



User Case 1 material (JRC ISPRA)

- Liquid effluent storage tank at JRC, Ispra
- Oil liquid effluent treatment station
- Two double walled side tanks
- In situ and lab-based measurements
- Aim to reproduce complete radiological characterisation of one tank

Activity concentrations range from 0.1 Bq/g to hundreds of Bq/g and are known for a long list of nuclides: ^{14}C , ^{41}Ca , ^{63}Ni , ^{79}Se , ^{90}Sr , ^{93}Zr , ^{99}Tc , ^{107}Pd , ^{147}Pm , ^{151}Sm e ^{241}Pu , ^{55}Fe , ^{59}Ni , ^{93}Mo , ^{129}I , ^{60}Co , ^{94}Nb , ^{134}Cs , ^{137}Cs , ^{152}Eu , ^{154}Eu , ^{241}Am , ^{235}U , ^{238}U , ^{237}Np , ^{238}Pu , $^{239+240}\text{Pu}$.



Development of aqueous reference material

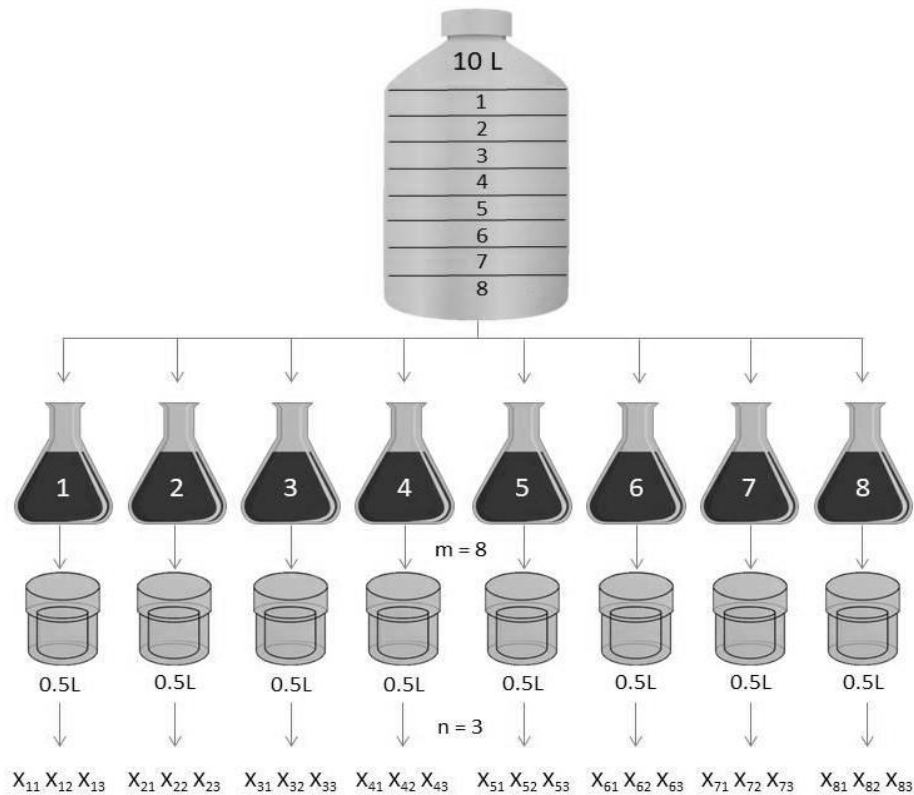
- UC1: Effluent tank storage characterised by JRC ISPRA, including pH, granulometry, density, elemental/isotopic composition by ICP-MS/ICP-OES
- Measurements:
 - Activity
 - Stability
 - Homogeneity
 - ^{60}Co , ^{137}Cs , ^{241}Am , $^{239,240}\text{Pu}$, ^{238}U

| Characteristics | Liquid CRM |
|---------------------------------|--|
| Matrix | Liquid (63 g HNO ₃ /L distilled water (pH<1)) |
| Volume | 10 l |
| Scale of homogeneity | 0,5 l |
| Suggested RNs for certification | |
| | ^{63}Ni (1-10 Bq/g) |
| | ^{90}Sr (1-100 Bq/g) |
| | ^{238}Pu (0.1-10 Bq/g) |
| | $^{239,240}\text{Pu}$ (0.1-10 Bq/g) |
| | ^{241}Am (1-10 Bq/g) |
| | ^{60}Co (0.1-10 Bq/g) |
| | ^{137}Cs (1-200 Bq/g) |
| | ^{55}Fe (0.1-5 Bq/g) |
| | ^{238}U (0.1-10 Bq/g) |

| Stable element content | Content range (mg/kg) |
|---|-----------------------|
| Al (Al(NO ₃) ₃ ·9H ₂ O) | 170-180 |
| B (H ₃ BO ₃) | 1-5 |
| Ca (CaCl ₂ ·6H ₂ O) | 35-45 |
| Co (Co(NO ₃) ₂ ·6H ₂ O) | 1-5 |
| Cr (Cr(NO ₃) ₃ ·9H ₂ O) | 250-310 |
| Cu (CuCl ₂ ·2H ₂ O) | 5-15 |
| Fe (FeCl ₃ ·6H ₂ O) | 1,000-1,500 |
| Hg (Hg(NO ₃) ₂ ·H ₂ O) | 30-35 |
| K (KNO ₃) | 60-80 |
| Mg (Mg(NO ₃) ₂ ·6H ₂ O) | 15-25 |
| Mn (MnCl ₂) | 35-70 |
| Na (NaNO ₃) | 6,000-6,200 |
| Ni (Ni(NO ₃) ₂ ·6H ₂ O) | 175-250 |
| Pb (Pb(NO ₃) ₂) | 15-25 |
| S ((NH ₄) ₂ SO ₄) | 3,100-3,400 |
| Zn (Zn(NO ₃) ₂ ·5H ₂ O) | 3-5 |



Homogeneity Testing



Random stratified sampling:

The whole liquid RM (10 L) was divided into 8 equal parts and from each part one Marinelli beaker of volume 0.5 L was created. Each sub-sample ($m = 8$) was measured 3 times ($n = 3$).



Short Term Stability Evaluation – ^{241}Am , ^{60}Co , ^{137}Cs

- Transport time - 1 week (transport by plane)
- Temperature
- 2 RM units for each combination of time and temperature



Additional Testing

Activity of ^{241}Am , ^{60}Co , ^{137}Cs

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**Short term stability of ^{241}Am ,
 ^{60}Co , ^{137}Cs**

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**Long-term stability
of ^{241}Am , ^{60}Co , ^{137}Cs**

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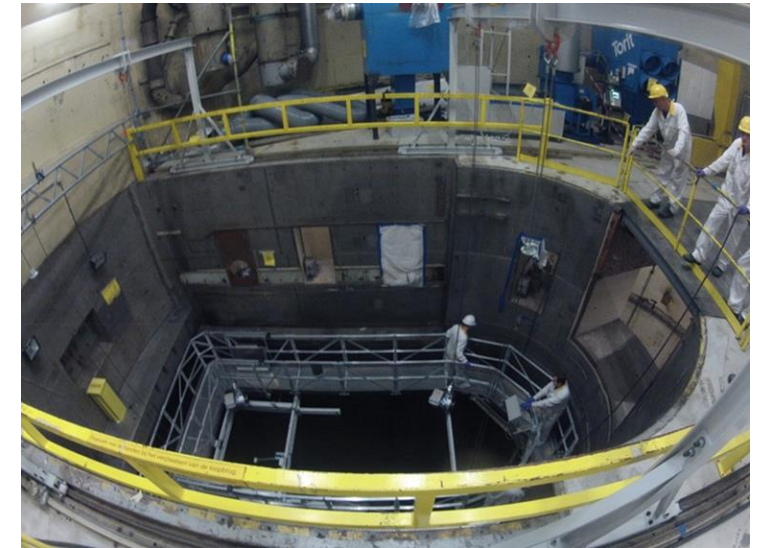
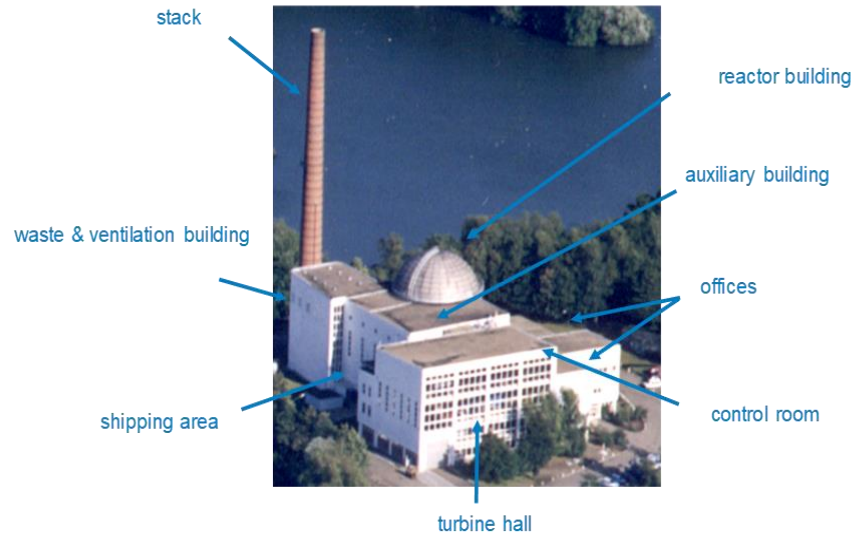
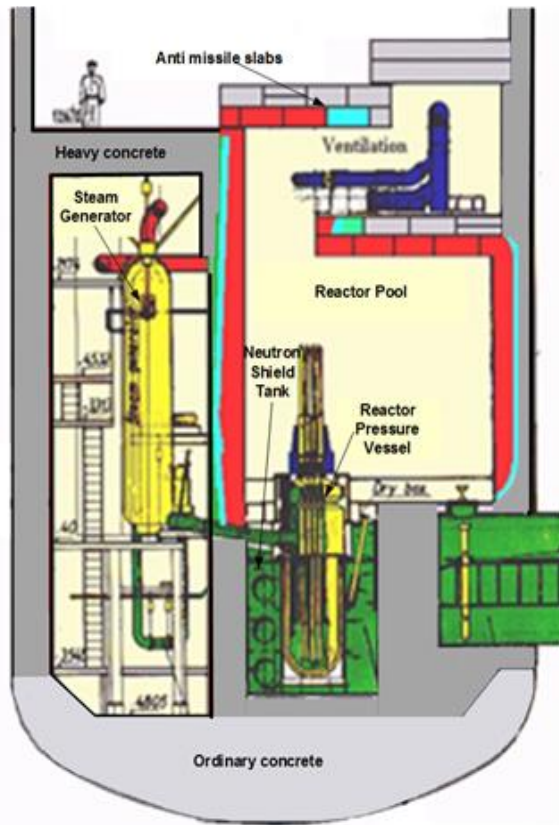
**Homogeneity (1 mL), stability
and activity of U^{238} and
 $\text{Pu}^{239,240}$**

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User case 2 material

Reactor Bio shield concrete (SCK/CEN)



User case 2 material

Reactor Bio shield concrete (SCK/CEN)



- Main radionuclides to measure: Ba-133, Eu-152, Co-60
- Reactor core material sent to NPL for homogenisation and distribution to Insider labs
- Additional non-irradiated concrete sent for making of spiked material
- In situ measures at two detector-to-source distances (contact and 10 cm)
 - Dose rate
 - Total gamma
 - Additional gamma spectrometry at the bottom of the pool in a fixed and defined position



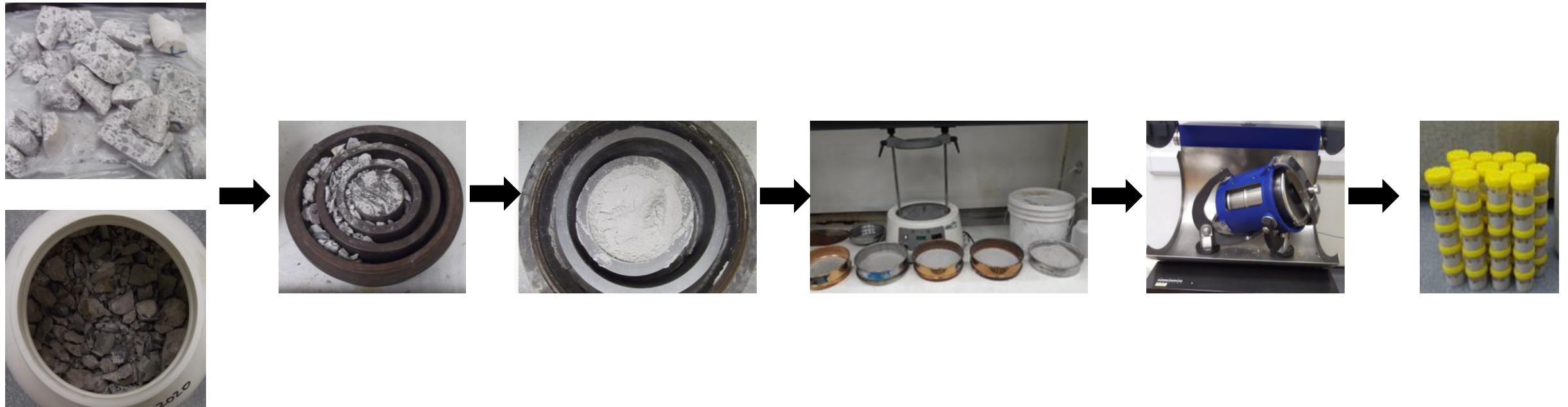
Development of solid reference material

- UC 2: Reinforced high density concrete, barite >40%, density $\sim 3.5 \text{ g/cm}^3$
- Higher activity parts 15-20 Bq/g ^{133}Ba , average activity 1-5 Bq/g
- Concrete core samples delivered to NPL
- Cores split and handled as 'front of bioshield' and 'back of bioshield'
- Non-irradiated material also delivered to enable production of matrix-matched spiked material

| Characteristics | Liquid CRM |
|---------------------------------|-------------------|
| Matrix | Concrete |
| Mass | 10 kg |
| Scale of homogeneity | 50 g |
| Suggested RNs for certification | |
| | ^{133}Ba |
| | ^{152}Eu |
| | ^{60}Co |



Preparation of solid reference material



Testing

Activity measurement of ^{133}Ba , ^{152}Eu , ^{60}Co

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**Homogeneity (50 g) and stability testing of
 ^{133}Ba , ^{152}Eu , ^{60}Co**

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Development of reference materials

- Materials distributed to participants in late 2019 and early 2020
- Measurement of samples in 2020 when lab restrictions and time allowed
- Inter-laboratory comparison led by Work Package 6
- Laboratories received:
 - Spiked aqueous material (0.5 L)
 - Spiked solid material (50 g)
 - 'Real' concrete samples (50 g)



THANK YOU

