

Difficulties in homogenizing a sludge sample



Difficulties in homogenizing a sludge sample in preparation for a new comparative test

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Planning to supply a new sample for OBT intercomparison by SCK CEN

- October 2020: Sampling of reed plants near the nuclear power plant in Doel (Antwerp)
 - Plants were cut, dried and grinded
 - Pyrolysis on the dried sample → < 20 Bq/kg dried sample
- June 2021: Basin of the cooling towers of BR2 were cleaned
 - Sludge of the basin was dried, grinded and mixed
 - Pyrolysis on the dried sludge
 - First results between : 600 Bq/kg DS and 16000 Bq/kg DS
 - Sample is not homogeneous
 - extra grinding and mixing necessary



Sampling

- September 2021 – sampling sludge Bassin BR2

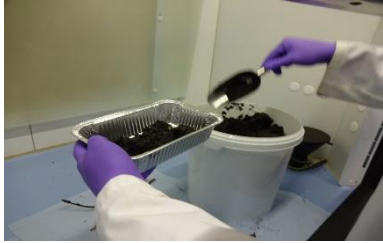


- Sub sampling sludge after cleaning the bassin of the cooling towers of BR2



Preparation

- Freezing the samples at -20°C



- Freeze-drying



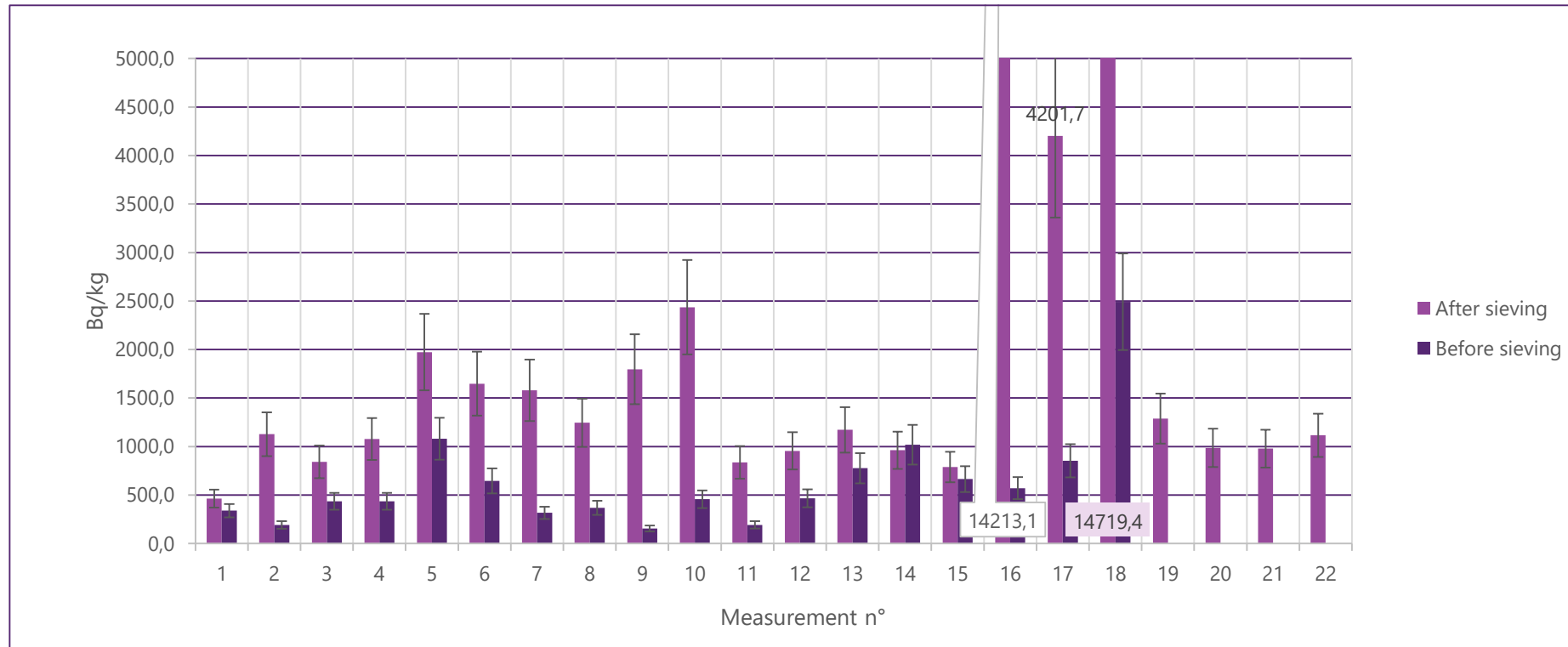
- Grinding and mixing



- Sieving over $200\ \mu\text{m}$



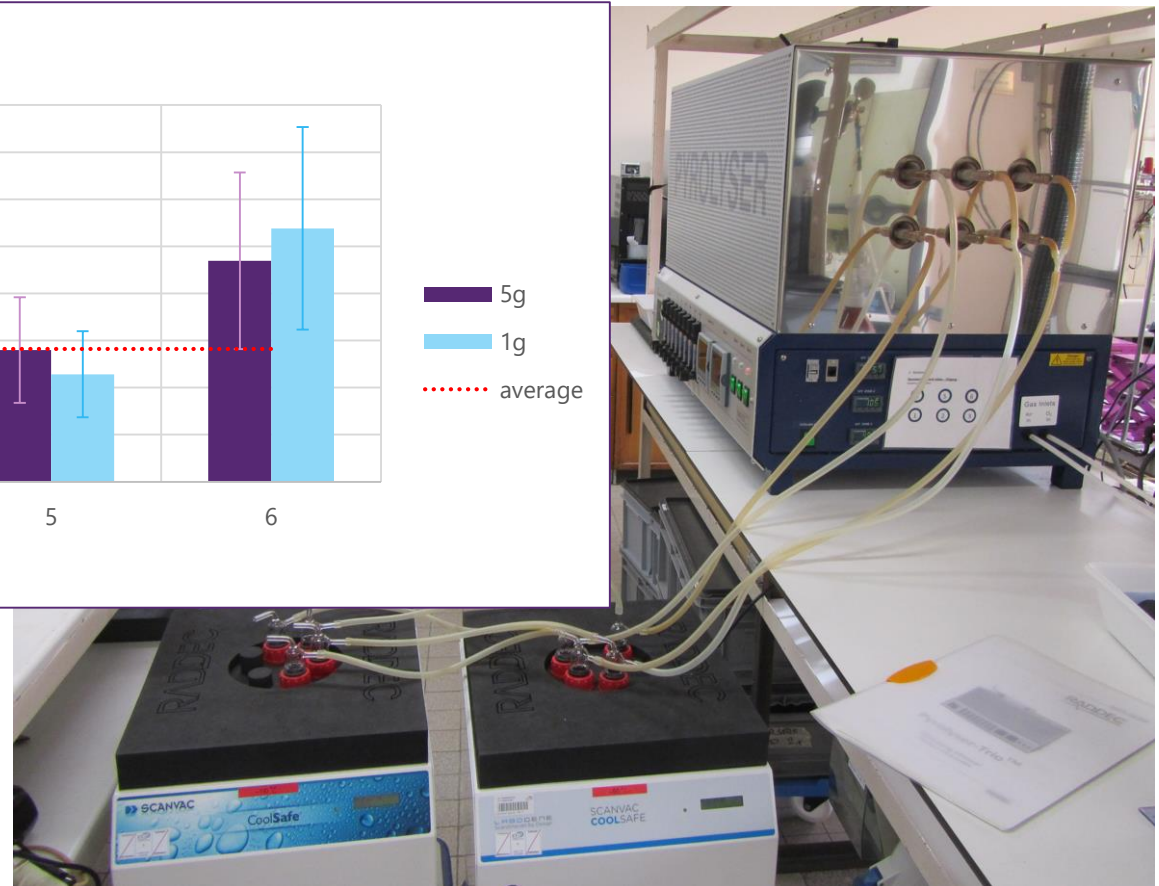
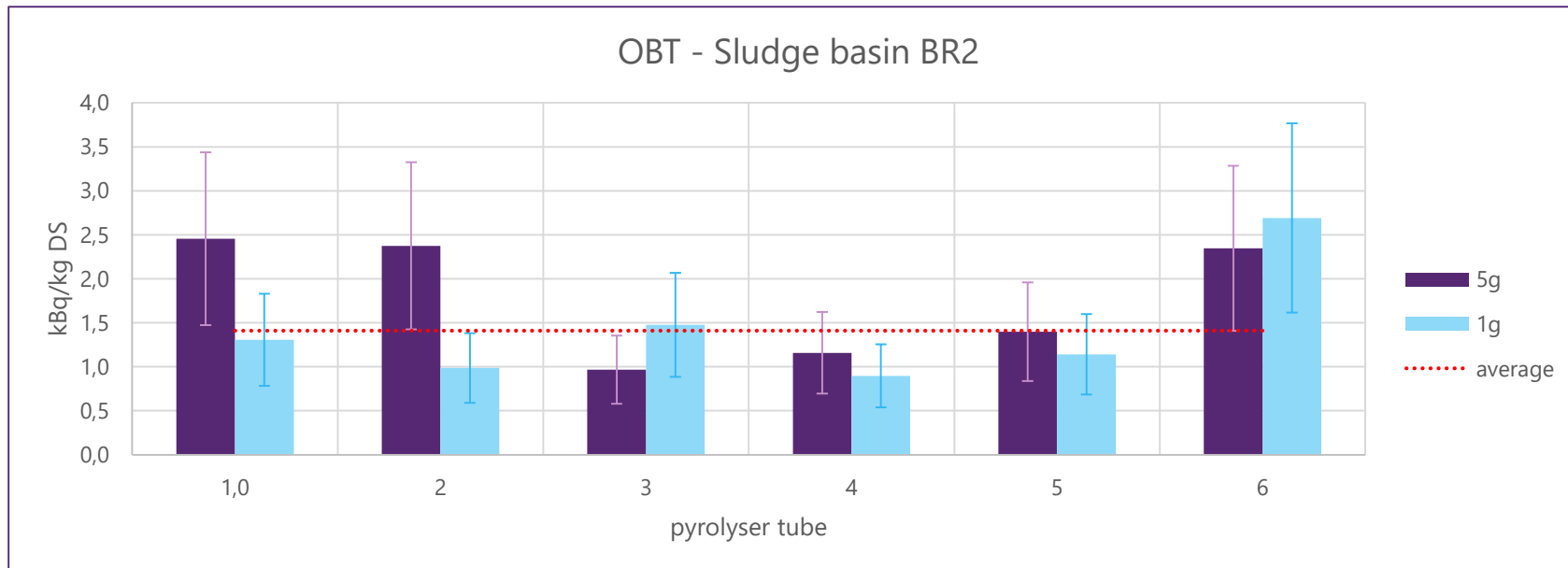
OBT Measurements



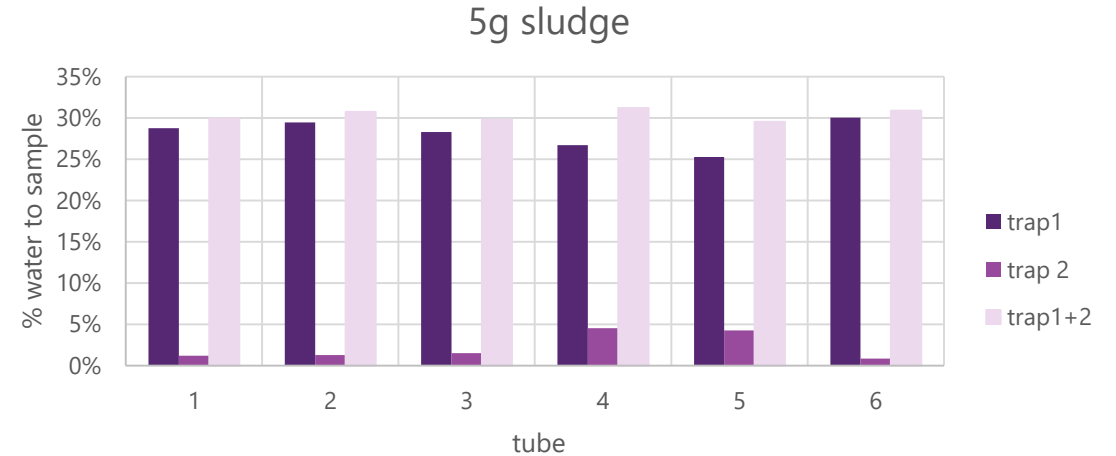
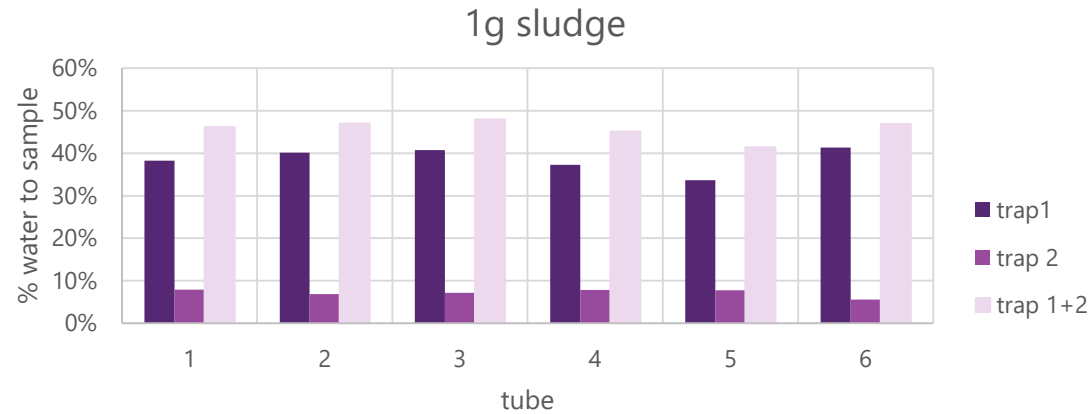
- The sample does not seem to be homogeneous
- Extra sieving does not give better results

Extra tests

Pyrolyse using cooling/freezing to trap the water (two cooling traps in serie)
OBT (Bq/kg dry sample)



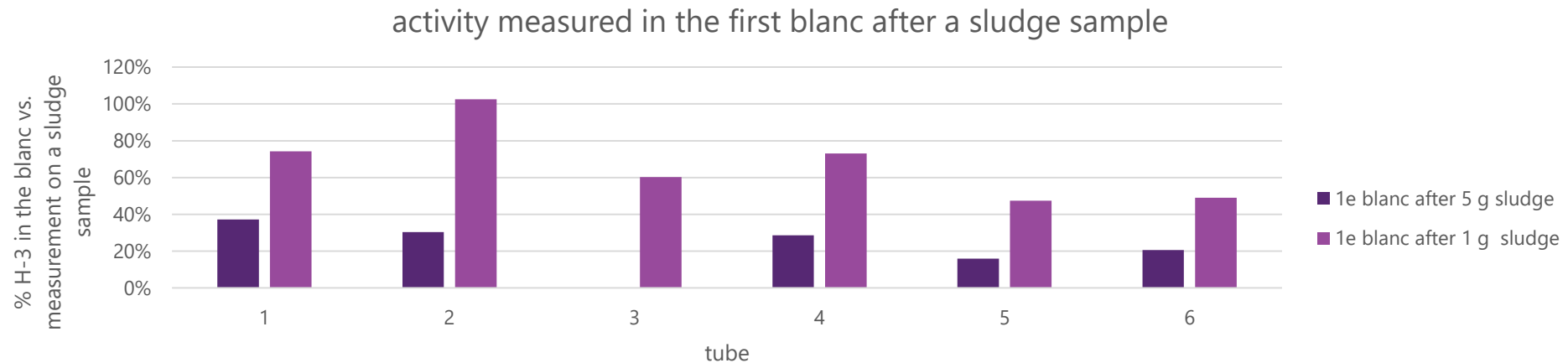
% water content after pyrolyse



- % water is \pm equal for the six samples combusted at the same time
- % water of 1 g sample seems to be higher than % water of 5 g sample
 - Average:
 - 1.3 g sample \sim 0.5 g water (38% water content from \pm 1g sample)
 - 5.0 g sample \sim 1.5 g water (30 % water content from \pm 5g sample)
 - Can be explained by
 - the water content in the compressed air (compressed air is used for pyrolysis)
 - the uncertainty on the mass (mass of one cooling trap = \pm 165 g)

Contamination?

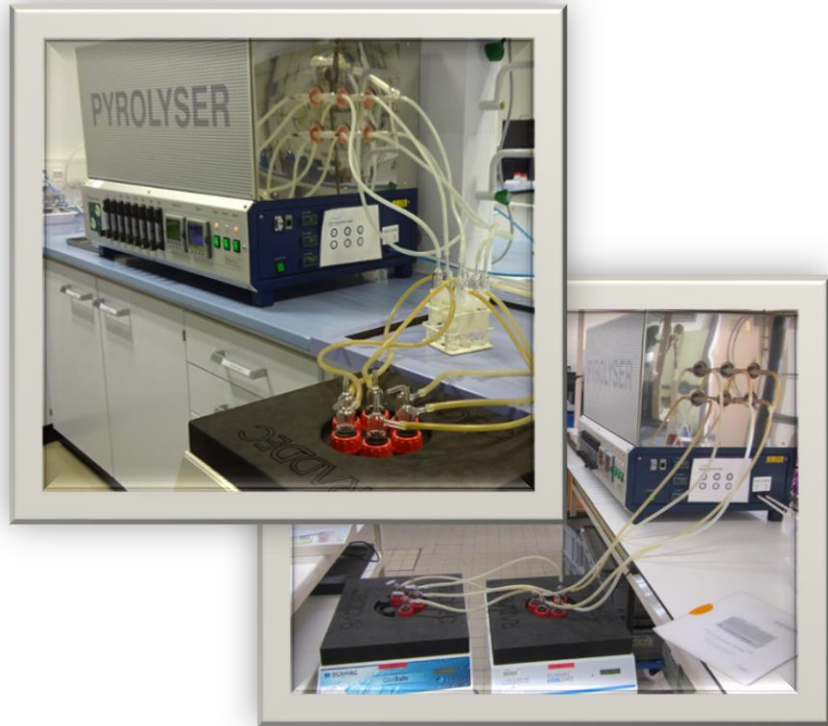
- After each run in the pyrolyser with sludge of the basin of BR2, two runs with blanc samples were done. (wood is used as blanc samples)
 - 1^e blanc is done with new sample boats (checking if contamination is in the tubes of the pyrolyser)
 - 2^e blanc is done with the same sample boat as used for the sludge (no activity measured)



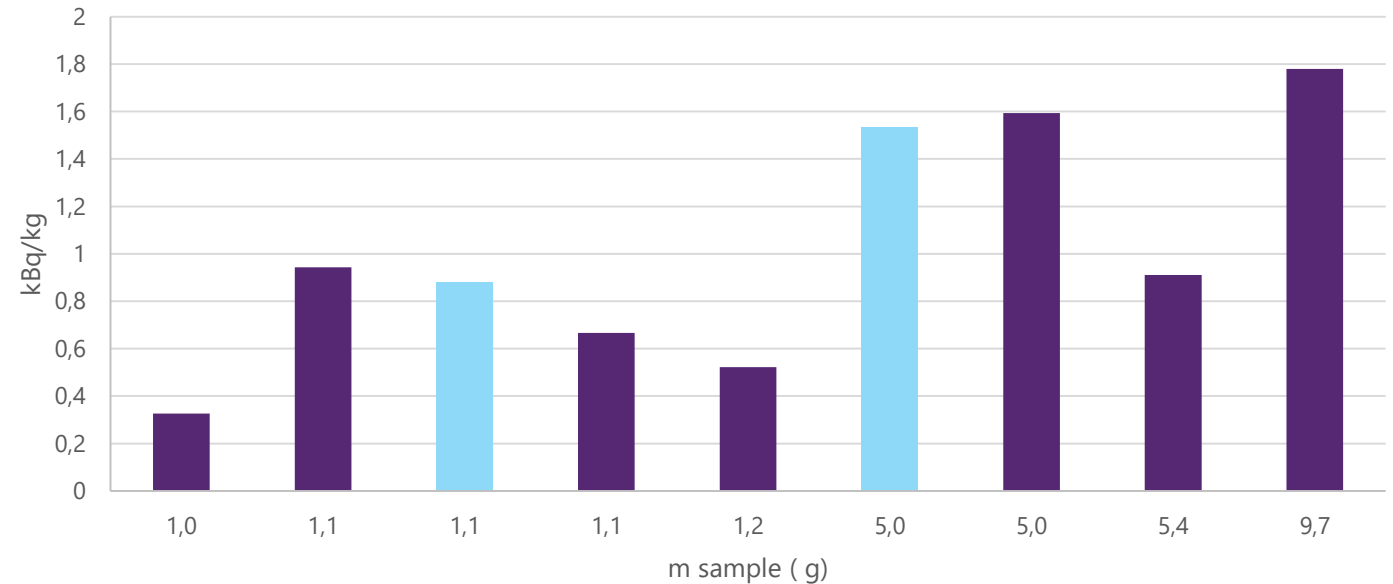
- Activity measured in the blanc samples compared to the activity measured in the sludge:
 - after 5 g sludge → average 27% (except tube 3: no result)
 - after 1g sludge → average of 68%

Pyrolyse using cooling/freezing to trap the water or bubblers?

Is the activity of OBT depending on the mass of the sample?



Ratio mass sample - activity



Remark: m = 1.0625 g en 4.995 g → bubblers

- 5 g sample → 900 - 1600 Bq/kg
- 1 g sample → 300 Bq/kg - 1000 Bq/kg

Asking external help !!!

- Extra grinding is done by Dr. Dave Reading (University Southampton)

- Sludge sample grinded and sieved by SCK CEN
- Grain size < 0.2 μm



- Sludge sample after extra grinding using a TEMA disk mill



- Pyrolyse after extra grinding

Mass combusted (g)	Result (Bq/kg) Uncertainty k=2
0.56	310 \pm 100
0.57	870 \pm 120
0.63	240 \pm 80
1.09	560 \pm 80
1.58	280 \pm 40
1.94	350 \pm 50

Conclusion?

- The sample does not seem to be homogeneous and can not be made homogeneous...

How to solve this?



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