NERC-CEH Gamma calibration procedure – overview

- Analyse each 'blank' calibration material on a gamma detector to confirm no contamination is present in the material before it is 'spiked' with mixed gamma radionuclide calibrated reference solution.
- Perform a background subtraction count for 2 days on each detector.
- Create new 'Certificate' files for each matrix/geometry combination. These contain all the information supplied on the certificate that comes with the mixed gamma radionuclide calibrated reference solution and details of the volume/mass of reference solution added to each matrix/geometry combination

- Create new 'geometry' files (these define the size and density used in the efficiency calibration) for each new matrix/geometry combination for use on all detectors.
- Perform an energy calibration (once on each detector).
- Perform an efficiency calibration for each matrix-geometry combination on each detector.
- Compare efficiency calibrations with previous calibrations (if possible).
- 'Approve' each efficiency calibration.
- Count the 'new' calibration standard as a sample to check of the efficiency calibration (the activity concentrations obtained should be the same (within error) as the amount added; if not something has gone wrong (e.g. incorrect amount of isotope added to make the calibration standard, incorrect values entered into the certificate file, some material has been 'lost' during the transfer from the vessel used to make the calibration standard and the counting container etc.).
- If available count previous standards and QC material (if available) e.g. samples used in any proficiency testing schemes.

NERC-CEH Gamma calibration procedure – detail

1. Collate all data, validate, sign and date and get reviewed by another suitably qualified analyst and/or the technical manager
2. Gamma detectors are calibrated before analysis of environmental samples containing gamma emitting nuclides.
3. Environmental samples including waters, soils and vegetation which are analysed in a similar manner to calibration samples, which range in matrix density and masses.
4. All gamma detectors should be calibrated using a certified reference solution, which is mixed with uncontaminated reference material (e.g. deionised water, soil, compost, hay). The geometries for the material type

will range in size depending upon sample mass and geometry of the gamma container ‘pot’.

REAGENTS AND MATERIALS

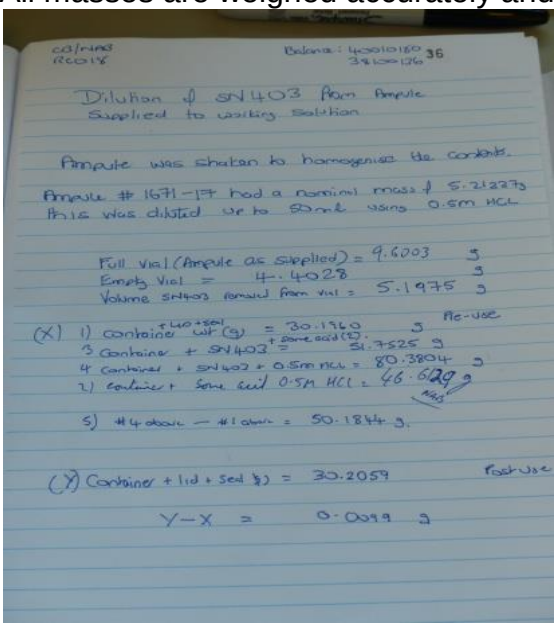
- Mixed gamma radionuclide calibrated reference solution (usually obtained from NPL) containing up to kBq levels for gamma emitting nuclides included.

- Dried and ground uncontaminated reference material (e.g. soil, compost and hay)

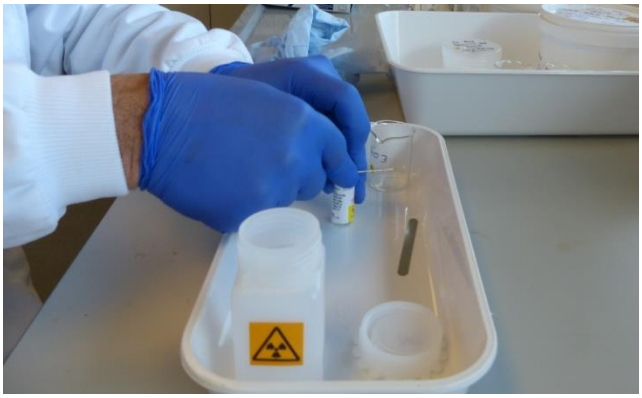
- Methanol and 0.5M HCl (carrier solution)

- Point sources of ^{137}Cs, ^{241}Am and ^{60}Co for energy re-calibration

5. All masses are weighed accurately and recorded in a lab book.



6. The gamma reference solution is diluted to required activity levels in a fumecupboard, which is monitored by appropriate contamination monitor of the work space.



7. Before diluting the reference solution, weigh the empty beaker and record the mass. The reference solution is added, re-weighed and diluted with carrier solution. The working solution is mixed thoroughly to ensure homogenisation.

8. Label the counting containers with geometry name, matrix and the date on the certificate supplied with the *calibrated reference solution.*

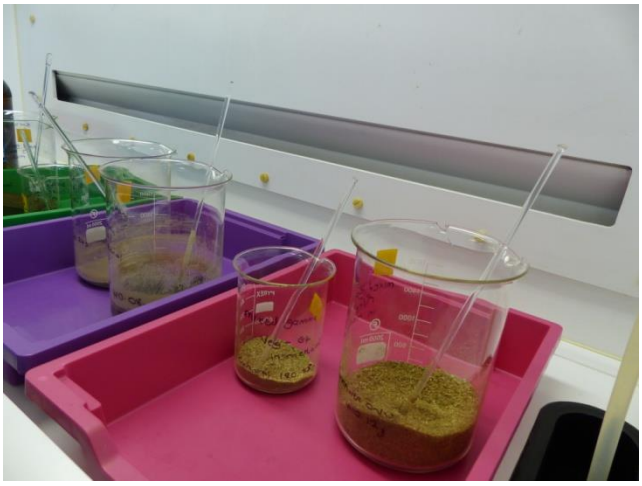
Solid materials – blank preparation

9. Taking (dried and ground) uncontaminated reference material of each matrix to fill all the required geometries twice (so it is possible to create a 'blank' and a 'standard' of the same matrix). It is good practice to add a bit extra (approximately 10g per kg) to allow for losses.
10. Oven dry the reference material to a constant weight.

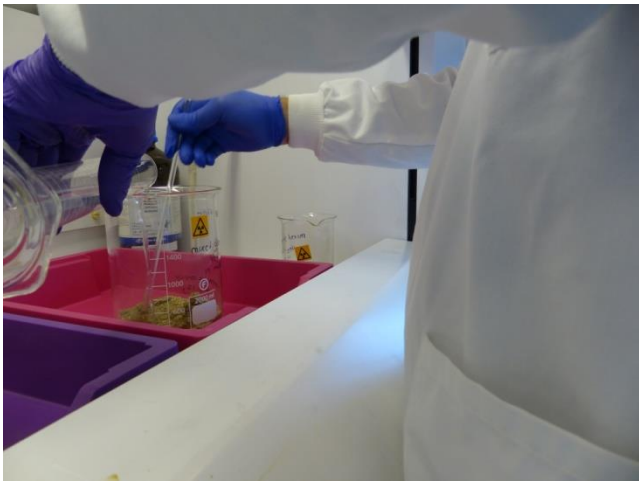
11. Fill the counting containers to the top with the dry uncontaminated reference material. Weigh the container before and after filling.
12. Seal and place in a plastic bag.

Solid materials – standard preparation

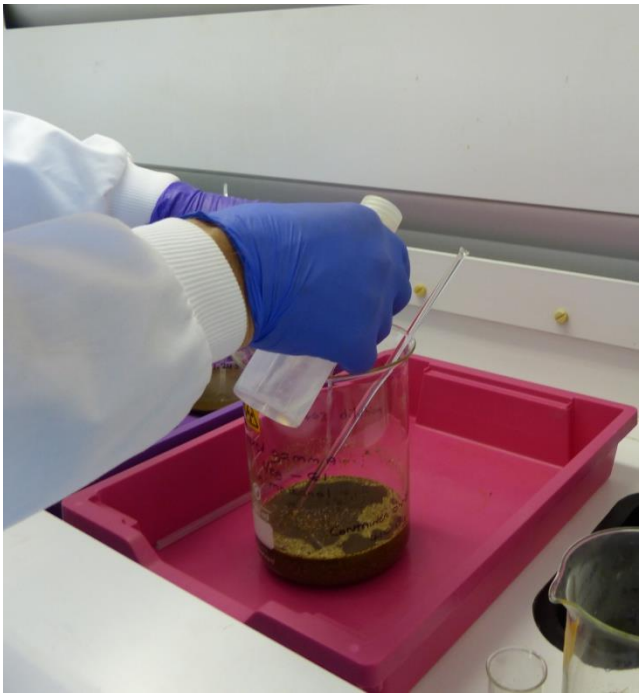
13. Weigh into the large glass beaker the same amount of uncontaminated calibration material required to fill the counting container to the top (i.e. the weight used in the 'blank' preparation) adding extra, to allow for losses.



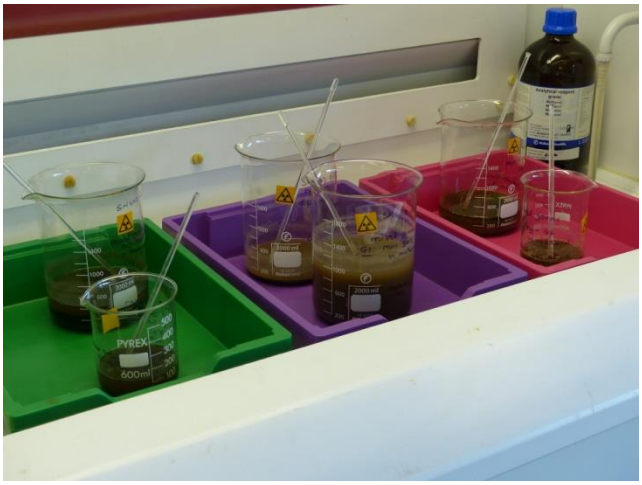
14. Depending upon the geometry of the container diluted reference solution is added to approximately 100 or 700 ml of methanol (volume dependent upon amount of calibration material).



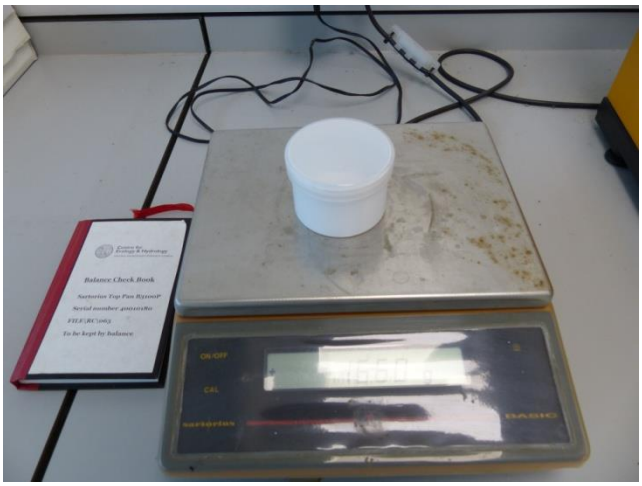
15. Methanol is added with gentle mixing to the reference material with gentle mixing, ensuring no losses. Add more methanol if required to the mixture and mix, repeat mixing every few hours to avoid the calibration material ‘caking’ and sticking to the sides of the beaker. Allow the standard to dry slowly to a constant weight.



16. The aim is to get as close as possible to the weight recorded previously. Note: this may take several days depending upon the geometry but must be done slowly to avoid ^{203}Hg volatility (if this isotope is included in the calibrated reference solution) – leaving the beaker in a switched on fume cupboard.



17. When a constant weight has been reached, tare a counting container, and transfer the standard into it, ensuring that the material reaches the top on the container. Take extreme care not to lose any material during the transfer (ensure work is performed in a foil tray).



18. Seal well and label with a radioactive warning sticker and place in a plastic bag.

19. The blank and solid materials are now ready for gamma counting.

Aqueous materials – blank preparation

20. Fill the counting containers to the top of container with deionised water and record the weight.
21. Seal and place in a plastic bag.

Aqueous materials – standard preparation

22. Taking c. 50 – 150ml of 0.5M HCL add predetermined aliquot of the diluted calibrated reference solution. Fill the container to the appropriate volume with 0.5 M HCl. gently mix with a glass stirring rod. Record weight of both the HCl and diluted calibrated reference solution added.
23. Ensure the counting container is identified as the Aq standard
24. Seal well and label with a radioactive warning sticker and place in a plastic bag.