## Comparison of Proposed Calibration Matrices for Gross Alpha and Beta Particle Radioactivity in High TDS Waters

<u>W.H. Johnson</u>, C.T. Bastain, T.W. Smith; Department of Health Physics, University of Nevada Las Vegas, Las Vegas, NV 89154-3037.

ASTM subcommittee D19.04 - Radioactivity in Water - is evaluating a proposed procedure for screening for alpha and beta particle radioactivity of waters containing elevated levels of dissolved solids. This procedure differs from the traditional dissolved solids method in that it treats the final residue with sulfuric acid. This results in a relatively stable matrix for residue thicknesses up to 50 mg cm<sup>-2</sup>. The final residue is hard enough that the planchets can be dropped onto a laboratory bench without losing material. Samples are then counted on a gas-flow proportional counter and efficiency corrected based on sample thickness.

The subcommittee is presently evaluating the influence of matrix composition on detection efficiency of alpha and beta emitters. It is focusing on four different matrices. One matrix is potable water from Lake Mead, NV (LM). This water was used to produce samples for the former EPA intercomparison program. Two of the matrices are formulations used in current procedures. The first is the recipe for reconstituted fresh water (very hard) used to prepared organisms for toxicity tests as per Standard Methods procedure 8010 (SMVH). The second formulation is recipe A from the USGS procedure for measuring gross alpha and beta radioactivity (method R-1120-76) (USGS). The fourth matrix being examined is a formulation consistent with the water used by Environmental Resource Associates for the gross alpha/beta intercomparison samples they currently distribute (ERA). Two beta emitting radionuclides ( $^{90}$ Sr/ $^{90}$ Y and  $^{137}$ Cs) and three alpha emitters ( $^{241}$ Am, natural uranium and  $^{230}$ Th) are to be examined in each matrix. All samples are being counted with a thin-window (80 µg cm<sup>-2</sup>) detector using time-resolved discrimination.

The proposed sulfation procedure produces a uniformly deposited and physically stable residue. The procedure though, is limited to sample volumes that result in a final thickness of between approximately 7 and 50 mg cm<sup>-2</sup>. For thinner residues, uneven deposition results in significantly different alpha efficiencies for equivalent samples. Thicker samples produce goo that is hard to work with during the final evaporation steps because of splattering and "popping" of the sample containers during heating. Samples above 50 mg cm<sup>-2</sup> separate into two layers upon final mounting on a planchet. A layer of material is bubbled up and another is on bottom. Sometimes the bubbles in the thicker samples exceed the height of the planchet (1 cm). This resulted in inconsistent alpha and beta efficiencies. Increased sample thickness did not result in increased alpha-to-beta cross talk. Increased thickness did, however, result in increased beta background, particularly for the LM and SMVH matrices which had significant potassium levels.

Initial results for <sup>241</sup>Am and <sup>90</sup>Sr/<sup>90</sup>Y show a significant difference in the mass attenuation curves for the various matrices. Waters prepared using the ERA formulation result in the highest detection efficiency; those prepared with the USGS formulation, the lowest. Samples prepared using the natural LM water had the most durable residues. Mounted residues of the ERA formulation were fragile and flaky, particularly at higher weights. Residues of samples of the USGS formulation contained significant moisture after a final heating to 105°C. To prevent this, the final drying temperature of all matrices on the planchets was increased to 350°C.





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