

ROCKY FLATS ENVIRONMENTAL TECHNOLOGY SITE REGULATORY CONTACT RECORD

Date/Time: 8/26/2004 / 10:00 & 8/31/2004 / 11:00

Site Contact(s): Bob Shannon / ASD
Phone: 303-966-7091

Regulatory Contact: Tony Ranalli – USGS
Phone: 303-312-6671

Agency: USEPA Region 8

Purpose of Contact: Technical Assistance in the Area of Alpha Spectroscopy

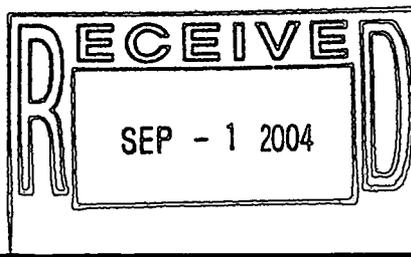
Discussion

Ed Brovsky asked me to respond to an EPA Region 8 request for alpha spectroscopy training for Tony Ranalli, a USGS chemist working on intragovernmental loan with them. The meeting took place on 8/26/2004 @ 10.00h at Region 8 headquarters, at Champa and 18th St., in downtown Denver and lasted 1½ -2 hours.

Early in the discussion, Tony Ranalli focused on a single sample from RIN 04D0380-004.001. He said that he had reviewed all of the associated QC and noted that the QC did not appear to indicate the likelihood of false positives due to contamination. I mentioned that I had glanced at the RIN in question the evening before and had asked myself about the possibility of contamination. I suggested that we continue with the alpha spectroscopy question and that we look at the data as we worked on the training.

I proceeded to provide a basic introduction to the physics of alpha spectroscopy including the physics of detection, general instrument configuration, spectrum characteristics and basic instrument quality control. We followed with a discussion of how using isotopic tracers, chemical separation of the element being sought and the alpha spectroscopic energy signature of alpha emitting isotopes makes the defensible isotopic determination of Pu-239 and Am-241 possible. We discussed weaknesses and strengths of the technique. We also discussed how use of blank populations functions both as a process control and as a mechanism to determine the method background and an empirical estimation of detection sensitivity of the method.

We reviewed the results for the sample in question. The batch prep blank showed no indication of contamination. The lab control sample met criteria. Chemical yield met criteria for all samples. In the case narrative, the lab indicated problems with the duplicate analysis. Consequently, the lab reanalyzed all samples in the RIN with the exception of sample 04D0380-004.001 which had been depleted in the first analysis. The lab reported the results of the reanalysis for all samples except 04D0380-004.001 where only the problematic results were available. *Note: Results for this sample were qualified (J) during validation since duplicate criteria were not satisfied.*



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Tony and I explored the question of why the duplicate results might have failed to meet acceptance criteria. The spectra for the sample and its duplicate were reviewed. It was noted specifically, that the Pu-238:Pu-239 ratio in the sample was ~170% while that in the duplicate was ~34%. Detecting Pu-238 at levels above Pu-239 would already be questionable in RFETS samples since Pu-238 is only present at very low levels relative to Pu-239 in RFETS samples. Observing significantly changed ratios of the Pu isotopes, however, is irreconcilable with a chemical process that can only preserve the ratios of isotopes of a single element. The data could be explained however, if the sample and its duplicate had been contaminated during processing at the laboratory. It is notable that since Pu-238 was present in both samples, it appears that both the sample and its duplicate were affected.

We discussed that Pu-238 is not a target analyte at RFETS and its presence / concentration is not evaluated by the data validators. Thus, routine reviews would not have likely revealed the contamination problem. All the validators saw was the problem with reproducibility of results.

Tony asked if it was safe to conclude that the samples had been contaminated and said that given our observations, he felt he might not be able to consider this a 'valid' result. We also explored the possibility of whether the data observed could have been attributable to detection system gain shifts or other instrumental anomalies. Given that the two spectra were acquired on separate instruments, I said that the chance of separate instruments malfunctioning in each of two spectra exactly such that three peaks present provide a unique energy fingerprint for Pu isotopes was diminishingly small. I reiterated that the problem clearly appeared to be one of contamination.

I pointed out that the blank population included in the data package (results of the twenty previous blanks showing no activity >MDA) provides empirical evidence that contamination problems are not a larger concern at this laboratory. As such, it would not be unreasonable to assume that this was a relatively isolated incident at the lab. Tony said he would speak with his management this week before he knew whether this would be an acceptable conclusion for them. We left the meeting without any plans for follow-up.

Note: I received a follow-up call from Tony yesterday (Monday) asking to reconfirm that instrumental anomalies could not have been responsible for the false positive. He asked about shared components of the instruments. Given the multiple results, and the clear isotopic signatures, I stated that I felt possibility this was extremely unlikely.

Contact Record Prepared By: Robert Shannon

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