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Validation and Verification of the Correct Operation of the Spectro RS23 UV-VIS spectrometer used in ANC122 and NF-ANC122

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Introduction:

In September of 2011, the old qualified Bausch and Laum Spec 100 UV-Vis instrument used in the ANC122 and NF-ANC122 procedures failed its monthly calibration check. Attempts to get the system to pass by cleaning flow cells, replacing the light bulb and realigning the light path failed to get sustained reproducible results from the system. In October the decision to take it out of service was made as the system was 40+ years in age. To replace the system, a previously purchased Labomed Inc.'s spectrometer (model Spectro 23RS; Serial #: 23RS-004253) was retrieved from storage. This report shows that the Labomed Inc. system is performing as required and meeting all QC requirements listed in ANC122 and NF-ANC122. Upon submitting this report to records, the system will be considered qualified to report results to customers of C-AAC.

Electrical Testing:

During its initial set up, the system was found to be exhibiting intermittent changes in absorption values anytime anyone got too near or touched the case of the instrument. The ESO was brought in to investigate the possibility of a grounding loop failure. He found that the case itself had burrs where screw holes had been tapped that prevented proper seating of the case. It was also found during this time that the knob controlling the grating position was in contact with the case when it should not be touching. When the case was removed to fix these two issues, it was noticed that the drive belt that runs between the knob and the grating had come off one of two pulleys. The ESO removed the burrs and widened the hold that the knob passed through, and replaced the drive belt on the pulleys. Once these three fixes were in place, the system stabilized and no more random changes in the absorption values were observed.

Filter test

To prove that the system had not lost correlation between the grating positioning and the resulting wavelength a calibrated 510 nm filter was placed in the spectrometer and the absorbance was measured. Figure 1 shows both the certified absorption curve and the measured absorption curve on the Spectro RS23. There is reasonably good agreement between the two spectra. Thus the new system can be expected to produce the correct wavelength for the iron measurements with no required adjustments or offsets.

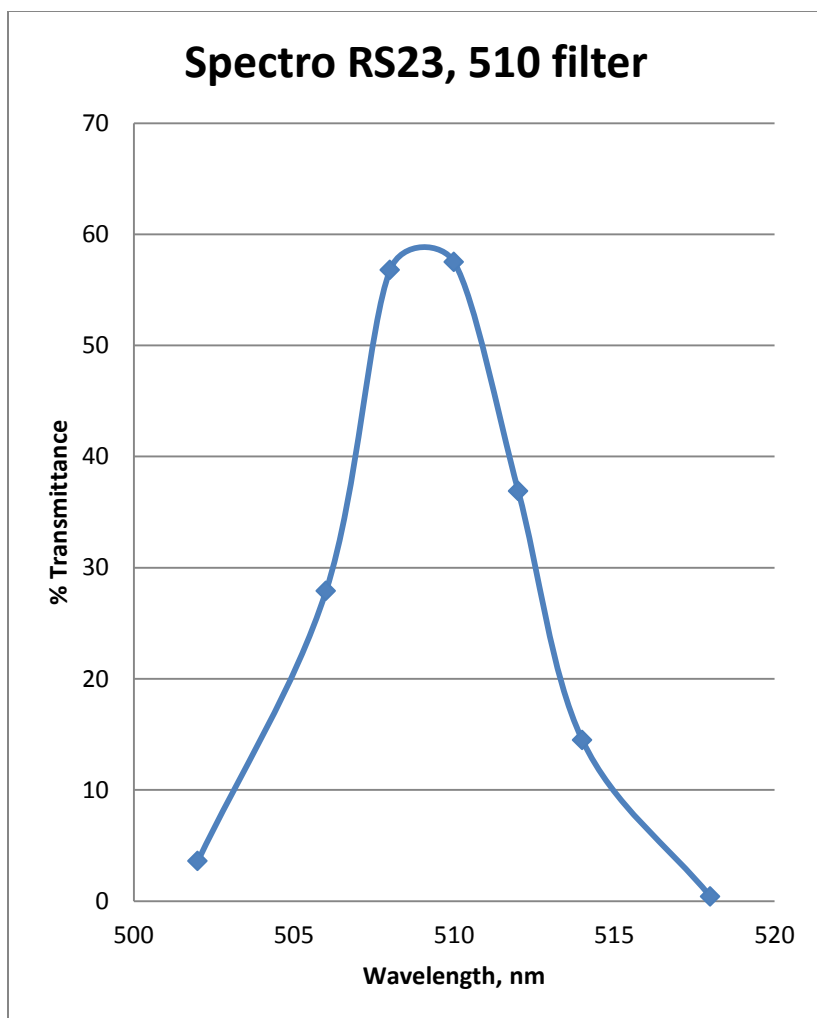
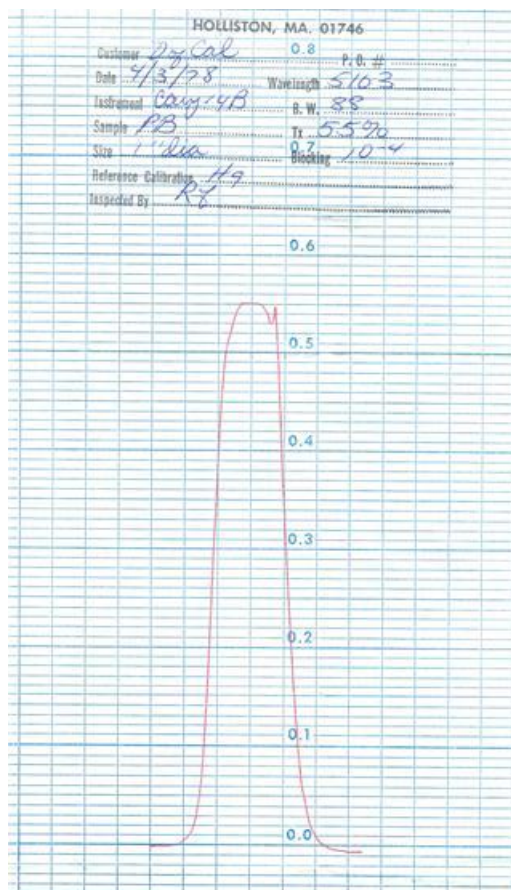
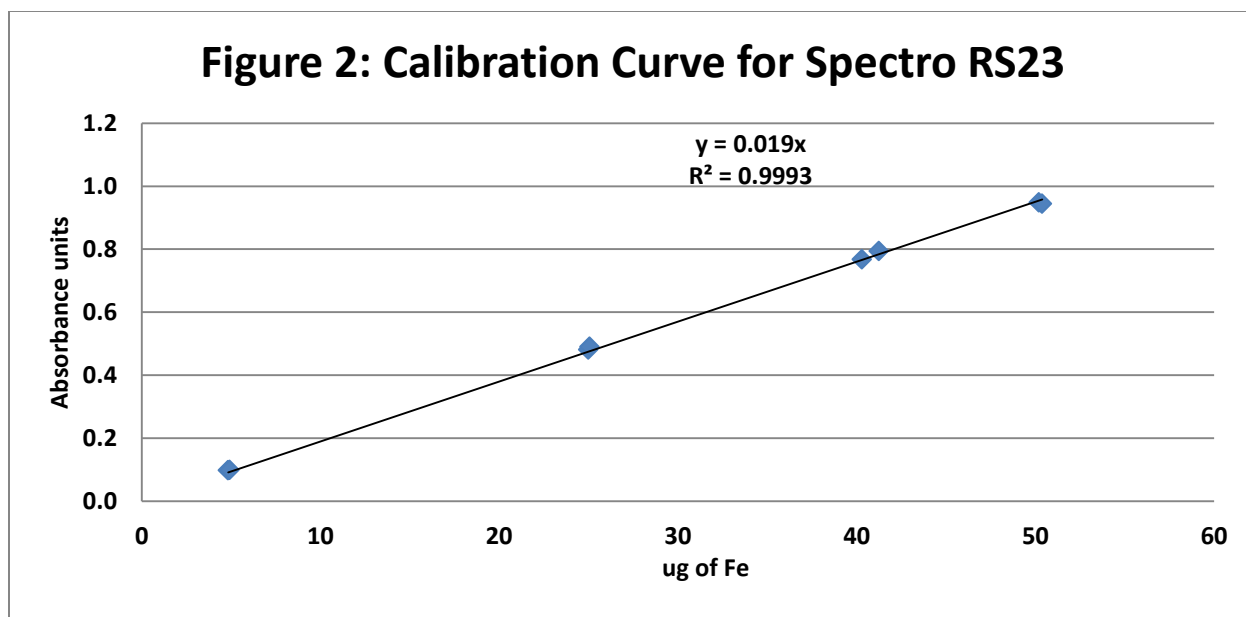


Figure 1: Spectra on the left is what the Filter is certified to be, spectra on the right is what the Spectro RS23 system measured.

ANC122 QC requirements:

The system must also prove that it can operate to the QC requirements of ANC122 and NF-ANC122, *Spectrophotometric Determination of Iron in Plutonium Materials*. The first requirement is that a calibration curve from 5 to 50 ug be linear and have an intercept on the Y-axis that is not significantly far from zero and a slope of 0.02 ± 0.001 absorbance unit per microgram of iron. Figure 2 shows that this system's calibration curve meets this requirement.



The second QC requirement is that the monthly calibration factor check must be between 48–52. This factor is just the inverse of the slope of the calibration curve and allows a quick check of the stability of the calibration curve without having to redo a full curve. Table 1 clearly shows that this system has been stable over 3 months of time in regards to this QC factor and thus meets this QC requirement.

Table 1:

Date run	CF
9/24/2012	51.55
9/24/2012	51.21
10/15/2012	50.13
10/15/2012	50.32
11/15/2012	50.66
11/15/2012	51.28

Final check, Comparison of results run on the currently WR qualified Prim System:

A final check of the system is to compare the results of actual samples to results produced by another qualified system. The Prim Spectrometer is a war reserve (WR) qualified system for ANC122. If the two systems produce results that are within the uncertainty of the method, then it can be concluded that the new system is functioning correctly and producing results that are acceptable for release to customers. Samples were chosen randomly and the two systems tested parallel in time to each other so that knowing the results from one system could not bias the results on the other system. Table 2 shows that the two systems compare well given the uncertainty associated with each sample's results. It's unfortunate that three of the samples randomly chosen were such low levels that they were at what we consider the limit of quantitation, <20 ppm, for the method and instruments. However, both are giving results that are

statistical identical. In both cases, the customers would receive results indicating that the sample has <20ppm on average. For the final test sample the averages are statistically identical and the standard deviation for both are well within the expected RSD of 7.5% for concentrations between 250 ppm and 2500 ppm as documented in methods ANC122 and NF-ANC122.

Spectrophotometric Determination of Iron in Plutonium Materials.

Instrument	Sample 1 (ppm)	Sample 2 (ppm)	Sample 3 (ppm)	Sample 4 (ppm)
Prim	17	9	9	362
	5	4	14	350
	19	18	18	357
	5	9	17	355
sample average (ppm)	11	10	14	356
sd (ppm)	7.6	5.7	4.0	5.0
RSD %	68.4	57.2	27.8	1.4
Spectro RS 23	25	13	4	352
	14	9	9	379
	19	23	13	341
	14	13	13	351
sample average (ppm)	18	14	10	356
sd (ppm)	5.4	5.8	4.0	16
RSD %	30.6	39.9	40.7	4.6