

Posting type	Advisory
Subject	Calibration bias in Vanadium concentration reported to AQS
Module/Filter	Met One SASS / Teflon 47 mm
AQS Parameter Code	88164
Sites	Entire CSN network
Period	Nov 20 th , 2015 through Oct 31 st , 2017 sample dates
Recommendation	Divide reported Vanadium concentrations by 1.3
Submitter	K.Trzepla, ktrzepla@ucdavis.edu

Supporting information

Elemental concentrations from X-Ray Fluorescence (XRF) analysis are based on linear calibrations of the instruments. Two thin-film standards certified by a commercial manufacturer have underpinned the vanadium (V) calibration for XRF. After comparisons with other laboratories and other reference materials showed discrepancies in results for V, these standards were returned in 2017 to the original manufacturer for recertification. The recertified loadings reported in 2017 were lower than previously certified values by a factor of about $0.77 \left(\frac{1}{1.3} \right)$.

The XRF systems were recalibrated using the newly certified V loadings of the same two original standards. The updated calibration yields XRF values consistent with quoted loadings for four new V standards purchased from the same manufacturer in 2017. These new standards cover lower mass loadings, as shown in Figure 1.

V concentrations in samples collected starting November 1st, 2017 are being reported to AQS with the new XRF calibration. Retrospective application of the annual 2017 recalibration back to 2016 and 2015 calibrations assumes that the standards themselves did not change between their 2015, 2016 and 2017 certifications, and annual calibration records provide evidence of this stability (Figure 2). The University of California at Davis started performing the CSN analyses on sample dates of November 20th, 2015; therefore, the CSN vanadium concentrations reported to AQS are biased high from November 20th, 2015, through October 31st, 2017.

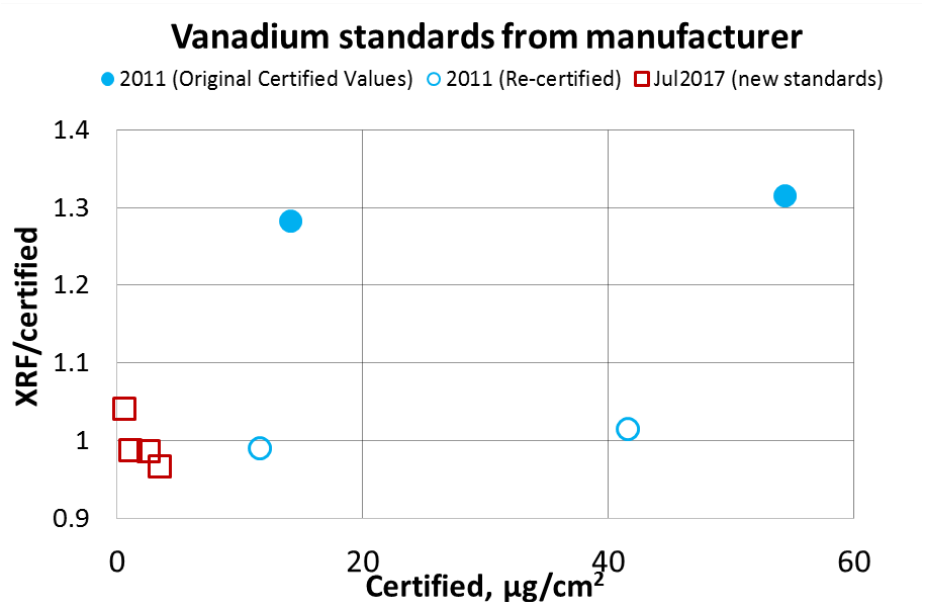


Figure 1. Ratio of the XRF measured to the certified value versus the certified V mass loadings for commercial thin-film standards. The reported XRF values are based on a calibration to the loadings certified in 2017 for the two standards originally purchased in 2011.

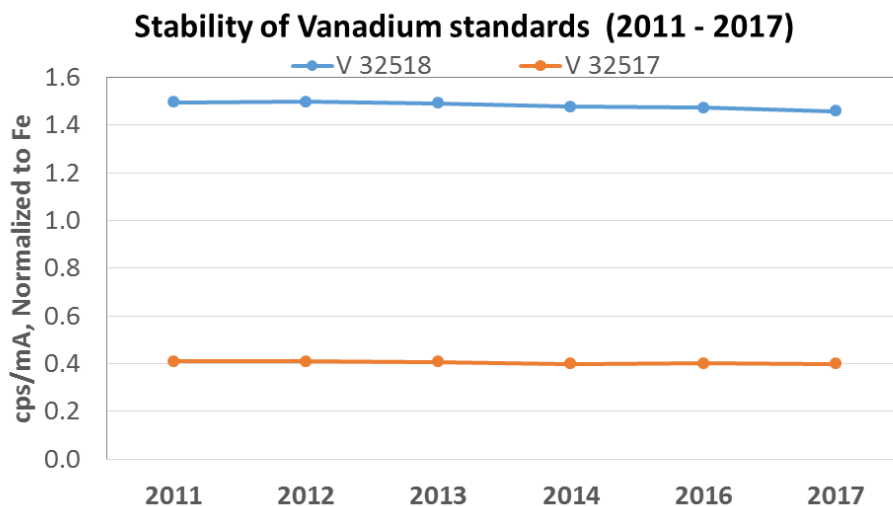


Figure 2. Raw response of the XRF analyzer to two specific calibration standards used throughout 2011 – 2017, normalized to Fe to account for physical changes in the system. The results show no evidence of changes in the standards during this period.