**Posting type** Advisory

Subject Calibration bias in reported Vanadium concentration

**Module/Species** A/ V

**Sites** Entire network

**Period** Jan 2011 through Oct 2017

**Recommendation** Divide reported concentrations by 1.3

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## **Supporting information**

Elemental concentrations from XRF analysis are based on linear calibrations of the instruments. Since the 2011 sample year, two thin-film standards certified by a commercial manufacturer have underpinned the V calibration. 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 loadings reported in 2017 were lower than previously certified values by a factor of about 1/1.3.

The XRF systems have been 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 cover a range of lower mass loadings, as shown in Figure 1.

V concentrations in samples collected starting in November 2017 are being reported with the new XRF calibration. Retrospective application of the 2017 recalibration back to 2011 assumes that the standards themselves did not change between their 2011 and 2017 certifications, and annual calibration records provide evidence of this stability (Fig.2).

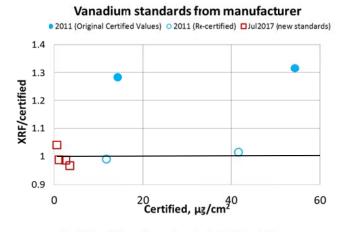


Fig. 1 Reported vs. quoted V mass loadings for commercial thin-film standards. The reported XRF values are based on a calibration to the loadings certified in 2017 for the two standards originally purchased in 2011.

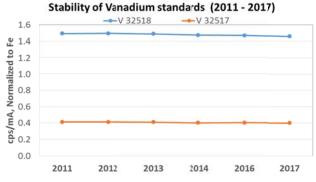


Fig. 2 Raw response of an XRF analyzer to two specific calibration standards used throughout 2011 – 2017, normalized to Fe to account for physical changes in the system. The results show no evidence of changes in the standards during this period.