

# Analytical data for the analysis of a new instrumental method for vanadium isotopic analysis

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**Data Type:** experimental

**Version:** 1

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## Project

» [Fingerprinting and Calibrating Low Oxygen Conditions Using Vanadium Isotopes](#) (Vanadium Isotopes)

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## Abstract

This dataset includes the analytical data for the analysis of a new instrumental method for vanadium isotopic analysis.

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## Dataset Description

This dataset includes the analytical data for the analysis of a new instrumental method for vanadium isotopic analysis.

## Acquisition Description

### Methodology:

This work uses a new mass spectrometric method using medium resolution mode (~4000) of a Thermo Scientific Neptune MC-ICP-MS. The setup presented is able to resolve all interfering molecular species in the V mass spectrum, thus removing the uncertainty of minor isotope effects from combinations of polyatomic C, N, O, S, Ar and Cl. The method utilizes high-transmission interface cones that significantly lower the amount of V required per analysis to roughly 200–300 ng, or almost an order of magnitude less than previous methods.

To test this we use pure vanadium solutions and analyze various instrumental settings to reproduce the known isotopic values of these solutions. Additionally, we test sulfur interferences by spiking the pure vanadium solutions with pure an elemental sulfur solution. Using this new instrumental method we test any potential S interferences. This new method will allow for more accurate and precise seawater analysis.

## Sampling and analytical procedures:

Vanadium isotope compositions are measured using a Thermo Scientific Neptune MC-ICP-MS. The front-end is configured with a 'Jet' sampler cone and an 'X'-type skimmer cone in order to obtain the highest possible V transmission efficiency. Vanadium is introduced into the mass spectrometer as a dry aerosol by passing the sample through a CETAC Aridus II desolvator, which is fed by an ESI perfluoroalkoxy alkane (PFA) nebulizer that aspirates the sample solution at ~120 ml per minute. This configuration routinely produces a vanadium ion beam of ~2.5 nA for a solution containing 1 mg per g vanadium when the mass spectrometer is operated in medium resolution, equivalent to a total ion transmission of ~0.7%.

The masses 48 through 53 are collected simultaneously using the L2, L1, Center, H1, H2 and H3 collectors. The Center cup was equipped with a  $10^{12}$   $\Omega$  resistor, H1 is equipped with a  $10^{11}$   $\Omega$  resistor, while the rest of the collectors are connected to standard  $10^{11}$   $\Omega$  resistors. Each sample analysis consists of 30 s background that is measured by defocusing and deflecting the ion beam away from the detectors in the electrostatic analyzer, followed by 40 cycles of 4.194 s integrations. Correction for interferences on  $^{50}\text{V}$  from  $^{50}\text{Ti}$  and  $^{50}\text{Cr}$  that cannot be resolved in medium resolution (nor high resolution) mode are performed as outlined in Nielsen et al. 2011. Each sample is bracketed by four (two on each side) measurements of the Alfa Aesar (AA-V) standard that has been defined as  $\delta^{51}\text{V} = 0$  (where  $\delta^{51}\text{V} = 1,000 \times [({}^{51}\text{V}/{}^{50}\text{V})_{\text{sample}} - ({}^{51}\text{V}/{}^{50}\text{V})_{\text{AA-V}}] / ({}^{51}\text{V}/{}^{50}\text{V})_{\text{AA-V}}$ ). In addition, each sample measurement is bracketed by two measurements of the bracketing standard BDH vanadium solution that has an isotope composition of  $\delta^{51}\text{V} = \sim 1.19 \pm 0.12$  (2sd) (Nielsen et al., 2011).

Methods for V isotope analysis are published in Nielsen et al., 2016 (doi: [10.1039/C5JA00397K](https://doi.org/10.1039/C5JA00397K)).

## Processing Description

### Data Processing:

This was a methods paper using new instrumental settings and to resolve issues with sulfur interferences, thus all solutions were pure vanadium solutions and did not contain any issues. However, we checked for any potential titanium and chromium interferences and potential effects on the V isotope data using the previously published methods of Nielsen et al., 2011 (doi: [10.1111/j.1751-908X.2011.00106.x](https://doi.org/10.1111/j.1751-908X.2011.00106.x)).

### BCO-DMO Processing:

- modified parameter names.

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## Related Publications

Nielsen, S. G., Owens, J. D., & Horner, T. J. (2016). Analysis of high-precision vanadium isotope ratios by medium resolution MC-ICP-MS. *Journal of Analytical Atomic Spectrometry*, 31(2), 531–536.

doi:10.1039/c5ja00397k <https://doi.org/10.1039/C5JA00397K>

*Methods*

Nielsen, S. G., Prytulak, J., & Halliday, A. N. (2011). Determination of Precise and Accurate  $^{51}\text{V}/^{50}\text{V}$  Isotope Ratios by MC-ICP-MS, Part 1: Chemical Separation of Vanadium and Mass Spectrometric Protocols. *Geostandards and Geoanalytical Research*, 35(3), 293–306. doi:10.1111/j.1751-908x.2011.00106.x

<https://doi.org/10.1111/j.1751-908X.2011.00106.x>

*Methods*

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## Parameters

| Parameter           | Description                                  | Units       |
|---------------------|--|-------------|
| Sample              | Sample name                                  | unitless    |
| Current_51V_voltage | Signal intensity                             | volts       |
| Measured_d51V       | Measured V isotope                           | per mil (‰) |
| two_sd              | 2 standard deviation error from all analyses | per mil (‰) |
| n                   | Number of analyses                           | unitless    |
| Actual_d51V         | Reported value using previous methods        | per mil (‰) |

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## Instruments

|   |  |
|---|--|
| <b>Dataset-specific Instrument Name</b> | Thermo Neptune multi-collector ICP-MS  |
| <b>Generic Instrument Name</b>          | Inductively Coupled Plasma Mass Spectrometer   |
| <b>Generic Instrument Description</b>   | An ICP Mass Spec is an instrument that passes nebulized samples into an inductively-coupled gas plasma (8-10000 K) where they are atomized and ionized. Ions of specific mass-to-charge ratios are quantified in a quadrupole mass spectrometer. |

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## Project Information

### Fingerprinting and Calibrating Low Oxygen Conditions Using Vanadium Isotopes (Vanadium Isotopes)

NSF Award Abstract: Discovering, testing, and developing chemical proxies (relic materials) in marine sediments that reveal how strongly or weakly oxidizing near-surface environmental conditions were in the Earth's geological past are immensely important for understanding interactions between ocean chemistry, biological evolution and extinctions, and climate. To date scientists do not have a proxy for low but non-zero oxygen conditions -- the sort of conditions that are likely to have dominated in biologically important periods of Earth history. In this project, researchers will study the relationship between bottom water oxygen concentration and the isotopes of the trace metal vanadium (V) in a range of oxygen conditions in the modern ocean. Based on pilot data, theoretical calculations and dissolved seawater V concentrations they believe that stable V isotope ratios of core top sediments will correlate systematically over a range of bottom water oxygen conditions. By analyzing these materials, the research team expects to establish the relationship between V isotopes and bottom water oxygen concentrations. Given the importance of chemical proxies to quantify past climate change, the results of this study will be of great importance to the modern and paleoceanographic community, as well as for modelers to better understand a broad range of oxygen variability in Earth history. Although recent investigations have provided a wealth of information about the redox conditions of the ancient oceans, there is a significant gap in understanding low oxygen conditions throughout Earth history. Therefore, it is important to develop new paleoredox proxies that can provide additional and complementary knowledge about ocean redox conditions during these important periods of Earth history. In this study, scientists will analyze bulk sediments and their

organic and ferromanganese mineral fractions to investigate the V isotopic variability within the various sedimentary components. (These samples comprise organic rich to ferromanganese rich sediments due to a range in bottom water oxygen concentrations.) Reconstructing marine low oxygen conditions using vanadium isotopes would fill a void in the paleoredox proxy toolbox. Developing, calibrating, and fingerprinting the V isotopic variability in modern sediments is required to be able to apply vanadium isotopes as an accurate paleoredox proxy.

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## Funding

| Funding Source   | Award                       |
|--|-----------------------------|
| <a href="#">NSF Division of Ocean Sciences (NSF OCE)</a> | <a href="#">OCE-1434785</a> |
| <a href="#">NSF Division of Ocean Sciences (NSF OCE)</a> | <a href="#">OCE-1624895</a> |

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