Data Article

X-ray absorption spectroscopy and X-ray diffraction data for molybdenum minerals and compounds

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Abstract

We report Mo K- and L_{III}-edge X-ray absorption spectroscopy (XAS) and X-ray diffraction (XRD) data collected for 15 molybdenum minerals and compounds sourced from museum collections, mineral dealers, and chemical suppliers. The samples were finely ground and analyzed at the Canadian Light Source synchrotron (Saskatoon, Canada). The L_{III}-edge XAS data were collected in fluorescence and total electron yield mode, while the K-edge XAS data were collected in transmission and fluorescence modes. Molybdenum L_{III}-edge spectra cover the X-ray absorption near edge structure (XANES) region and Mo K-edge spectra cover the extended X-ray absorption fine structure (EXAFS) region. Tabulated XAS data are provided to support analysis of XAS data obtained for geological or environmental research. Furthermore, Mo K-edge EXAFS and L_{III}-edge XANES spectra, the k^3 weighted oscillatory χ(k) functions, and the Fourier-transforms in χ(R) of these K-edge data are presented graphically. Corresponding XRD data were collected as two-dimensional images against an area detector and integrated to form line scans. The data were collected at a wavelength of 0.68866 Å (18 keV) and is tabulated and presented graphically over a

Abbreviations: EXAFS, extended X-ray absorption fine structure; XANES, X-ray absorption near edge structure.

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## Specifications Table

| Subject | Environmental Chemistry |
|---------|-------------------------|
| Specific subject area | X-ray absorption spectroscopy and X-ray diffraction of common and rare molybdenum minerals and compounds |
| Type of data | Table Figure |
| How the data were acquired | All data was collected at the Canadian Light Source synchrotron (Saskatoon, Canada). Molybdenum LII-edge XANES data were collected at the SXRMB bending magnet beamline under high vacuum, Mo K-edge EXAFS were collected at the BioXAS-Main wiggler beamline under liquid nitrogen, and X-ray diffraction data were collected at the CMCF beamline. The XAS data were processed using the Demeter XAS data processing package [1], while XRD data were processed using GSAS-II crystallography data analysis software [2]. |
| Data format | Raw Processed |
| Description of data collection | Common and rare Mo compounds and minerals were ground and thinly spread on carbon coated or Kapton tape. Mo LII-edge XANES were collected under high vacuum and Mo K-edge EXAFS spectra were collected under liquid nitrogen at the Canadian Light Source synchrotron in total electron yield, transmission, or fluorescence mode. Synchrotron-based XRD spectra were collected on capillary-packed material. |
| Data source location | Molybdenite – University of Saskatchewan Mineral Collection, Saskatoon, Saskatchewan, Canada. Sample 2961 K1 collected from Easton, PA, USA (40.69, -75.22) Wulfenite – Collected from La Morita Mine, Chihuahua, Mexico (30.90, -107.60) Powellite – Collected from Pune District, Maharashtra, India (18.52, 73.86) Bamfordite – Museums Victoria, Melbourne, Victoria, Australia. Sample M44484 collected from Bamford Hill W-Mo-Bi deposit, Grandon’s Shoot, Norton’s lease, Queensland, Australia (-17.31, 144.92). Kamiokite – The University of Arizona Mineral Museum, Tucson, Arizona. Sample 19002 collected from 5th May Adit, Vrchoslav Mine, Krupka, Krušné Hory Mountains, Ústechý, Czech Republic (50.69, 13.83). Ferrimolybdate – Canadian Museum of Nature, Ottawa, Ontario, Canada. Sample CMNMC 40358 collected from Mt. Quyon, Pontian Co., Quebec, Canada (45.51, -76.23). Lindgrenite – Canadian Museum of Nature, Ottawa, Ontario, Canada. Sample CMNMC 57163 collected from Magma Mine, #3 Pit, Arizona, USA 33.3, -111.09). Molybdate – Royal Scottish Museum Department of Geology, Edinburgh, Scotland. Sample G.1884.20.82 collected from Ross, Ontario, Canada (45.67, -76.82). Sidwillite – Royal Scottish Museum Department of Geology, Edinburgh, Scotland. Sample G.1985.3.7 (Dana No. 4.5.6.1) collected from Lake Como, Colorado, USA (37.92, -107.62). Ilsemannite – Royal Ontario Museum, Toronto, Ontario, Canada. Sample ROMESM 3255.1 collected from Himmlersfurst Mine, Mittelsachsen, Saxony, Germany (50.76, 13.30). Mo foil – Standard Mo(0) XAS reference foil purchased from EXAFS Materials (exafsmaterials.com) Mo coprecipitated ferrihydrite – Co-precipitated Na2MoO4 and ferrihydrite synthesized via rapid titration [3]. |

(continued on next page)
Sodium molybdate – CAS: 7631-95-0, Sigma-Aldrich, >98%
Mo(VI) oxide – CAS: 1313-27-5, Alfa-Aesar, 99.9995%
Mo(IV) oxide – CAS: 18868-43-4, Sigma-Aldrich, 99%

Data accessibility
Repository name: Federated Research Data Repository
Direct URL to data: doi.org/10.20383/103.0600

Value of the Data

- Extensive molybdenum sequestration is thought to occur in mining locations and in the deep oceans, however, Mo identification and speciation in these situations is limited due to the lack of reference materials.
- Environmental geochemists, paleoclimatologists, and geologists interested in Mo speciation will benefit from these data.
- This dataset is intended to be used as reference material in the identification and characterization of Mo minerals from future studies.

1. Data Description

Table 1. A list of details for the reference materials and minerals prepared in this dataset. The table includes the molecular formula, the Mo oxidation state, the space group, crystal system, class, Nickel-Strunz group, IMA-CNMNC symbol [4], and some notes. A reference to crystallographic information files are also listed.

Fig. 1. a) The Mo K-edge XAS spectra collected for the XANES region of the 15 reference materials, obtained over the energy range –20 eV to +100 eV around the Mo K-edge (20,000 eV) and collected at the BioXAS beamline at the CLS. All data is reported as edge-step normalized absorbance and was collected in fluorescence mode with the exception of the Mo foil and wulfenite which were collected in transmission mode. b) The $k^3$ weighted k-space equivalent of the EXAFS region of each of the references, reported from 2–15 Å$^{-1}$. c) The corresponding R-space for each reference, where data is shown from 0–10 Å. The Fourier Transform was conducted over the k-space of 3–11 Å$^{-1}$.
| Reference name                  | Formula                        | Mo oxidation state | Space group | Crystal system | Class              | Nickel-Strunz group | IMA-CNMNC symbol | Notes                                                                 |
|--------------------------------|--------------------------------|--------------------|-------------|----------------|--------------------|---------------------|--------------------|-----------------------------------------------------------------------|
| bamfordite<sup>1</sup>          | FeMo<sub>2</sub>O<sub>4</sub>(OH)<sub>3</sub> · H<sub>2</sub>O | VI                 | P1          | Triclinic       | Pinacoidal oxalate | oxide               | Bfd                |                                                        |
| Ferrimolybdite ilsemannite      | Fe<sub>2</sub>(MoO<sub>4</sub>)·nH<sub>2</sub>O | VI                 | Pmmn        | Orthorhombic    | Dipyramidal sulfite | oxide               | Fmyb               |                                                        |
|                                | Mo<sub>2</sub>O<sub>8</sub>    | VI                 |             | Amorphous       |                    |                     |                    | amorphous, questionable mineral status                                  |
| kamiokite<sup>2</sup>           | Fe<sub>2</sub>Mo<sub>3</sub>O<sub>8</sub> | IV                 | P 6<sub>3</sub>mc | Hexagonal       | Dihexagonal pyramidal | sulfite element     | Lgr                | commercial molybdate coprecipitated into ferricydrite                  |
| lindgrenite<sup>3</sup>         | Cu<sub>3</sub>(MoO<sub>4</sub>)<sub>2</sub>(OH)<sub>2</sub> | VI                 | P<sub>2</sub>1/m | Monoclinic      | Prismatic sulfide |                     |                    | semicrystalline, poor fluorescence                                     |
| Mo foil                        | Mo                            | 0                  |             |                |                    |                     |                    |                                                        |
| Mo coprecip ferricydrite molybdenite<sup>4</sup> | MoO<sub>4</sub> - Fe(OH)3 | VI                 |             |                |                    |                     |                    |                                                        |
| molybdenum dioxide             | MoO<sub>2</sub>                | IV                 |             |                |                    |                     |                    | commercial                                                       |
| molybdenum trioxide            | MoO<sub>3</sub>                | VI                 |             |                |                    |                     |                    | commercial                                                       |
| molybdate<sup>5</sup>           | MoO<sub>3</sub>                | VI                 | Pbnm        | Orthorhombic    | Dipyramidal oxalate | oxide               | Myb                | limited sample                                                      |
| powellite<sup>6</sup>           | CaMoO<sub>4</sub>             | VI                 | I 4<sub>1</sub>/a | Tetragonal      | Dipyramidal sulfite | oxide               | Pwl                | limited sample                                                      |
| sidwillite<sup>7</sup>          | MoO<sub>3</sub> · 2H<sub>2</sub>O | VI                 | P 2<sub>1</sub>/n | Monoclinic      | Dipyramidal oxalate | sulfite             | Sdw                | limited sample                                                      |
| sodium molybdate                | Na<sub>2</sub>MoO<sub>4</sub>  | VI                 | I 4<sub>1</sub>/a | Tetragonal      | Dipyramidal sulfite | oxide               | Wul                | commercial                                                      |
| wulfenite<sup>8</sup>           | PbMoO<sub>4</sub>              | VI                 |             |                |                    |                     |                    |                                                        |

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Fig. 2. The Mo L_{III}-edge XANES spectra for reference materials.

Fig. 2. The Mo L_{III}-edge XAS spectra was collected for the XANES region, where data was collected from -30 eV to +55 eV around the Mo L_{III}-edge (2525 eV) at the SXRMB beamline at the CLS. All files were collected in fluorescence mode with the exception of molybdenite, which was collected in TEY. All data is reported as edge-step normalized absorbance.

Fig. 3. X-ray diffraction data for each reference material with the exception of the Mo X-ray absorption foil, molybdate and sidwillite, which were not collected because of limited material. Data was collected at a wavelength of 0.6888 Å on an 18 keV beam at the CMCF beamline at the CLS.

Dataset 1. Mo K-edge energy calibrated Fluorescence XAS data collected from -200 eV to 15 k around the Mo K-edge (20,000 eV). This folder contains an Athena project file containing all references collected in fluorescence mode when possible. A subfolder lists each individual spectra as an .xmu file. These individual files are energy calibrated but not normalized to preserve the edge jump. Incident energy, measured in the first ionization chamber (I_0) and often noted as ‘Energy Feedback’, is listed in the first column, the absorption data of the desired reference in the second, and a Mo(0) reference foil is presented in the third column and was collected between chambers I_1 and I_2 for each scan to allow for alignment between scans.

Dataset 2. Mo K-edge energy calibrated Transmission XAS data collected from -200 eV to 15 k around the Mo K-edge (20,000 eV). This folder contains an Athena project file containing all references collected in transmission mode when possible. A subfolder lists each individual spectra as an .xmu file. These individual files are energy calibrated but not normalized to preserve the edge jump. Incident energy, measured in the first ionization chamber (I_0) and often noted as ‘Energy Feedback’, is listed in the first column, the transmission data of the desired reference in the second, and a Mo(0) reference foil is presented in the third column and was collected between chambers I_1 and I_2 for each scan to allow for alignment between scans.
Dataset 3. Mo L_{III}-edge energy calibrated Fluorescence XANES data was collected from -30 eV to +55 eV around the Mo L_{III}-edge (2525 eV). This folder contains an Athena project file containing all references collected in fluorescence mode when possible. A subfolder lists each individual spectra as an .xmu file. These individual files are energy calibrated but not normalized to preserve the edge jump. Incident energy, measured in the first ionization chamber (I₀) and often noted as ‘Energy Feedback’, is listed in the first column and the absorption data of the desired reference in the second. Subsequent columns record the pre- and post-edge and the derivative of the spectra. Mo(0) foil was not collected.

Dataset 4. Mo L_{III}-edge energy calibrated Total Electron Yield XANES data was collected from -30 eV to +55 eV around the Mo L_{III}-edge (2525 eV). This folder contains an Athena project file containing all references collected in Total Electron Yield mode when possible. A subfolder lists each individual spectra as an .xmu file. These individual files are energy calibrated but not normalized to preserve the edge jump. Incident energy, measured in the first ionization chamber (I₀) and often noted as ‘Energy Feedback’, is listed in the first column and the TEY data of the desired reference in the second. Subsequent columns record the pre- and post-edge and the derivative of the spectra. Mo(0) foil was not collected.

Dataset 5. Mo compound XRD was collected at 18 keV (\(\lambda = 0.6888 \text{ Å}\)). This folder contains .asc files of the XRD patterns of each available reference from 2-40 °2\(\Theta\). Sidwillite, molybdite, and the Mo foil were not collected due to limited material. Select files are not confirmed with a published crystallographic information file (CIF), and are presented as is. The first column represents the °2\(\Theta\) and the second represents the intensity, while the third column is the error term.

2. Experimental Design, Materials and Methods

**Reference material preparation** – Following a visual assessment for uniformity, samples were finely ground using an agate mortar and pestle.

**L_{III}-edge XANES analysis** – About 20 \(\mu\)g of finely ground reference materials were pressed onto a conductive Cu plate using double sided carbon tape as an adhesive. The total area of
each sample was approximately 1-2 cm² and several materials were affixed to one copper plate, taking care to avoid contamination during preparation and transport.

X-ray absorption spectroscopy (XAS) data was collected at the Canadian Light Source (CLS) in Saskatoon, Saskatchewan, Canada. The storage ring was operated in continuous top-up mode to a current of 220 mA. We collected XAS data over the X-ray absorption near-edge structure (XANES) region of the Mo L_{III} edge. Total electron yield (TEY) as well as fluorescence yield data for the Mo L_{III} edge were obtained at the SXRMB bending magnet beamline (06B1-1) under a vacuum of ~5 × 10^{-8} Torr. The sample was mounted at 45 degrees to the incoming beam and another 45 degrees to the 7-element fluorescence detector. This beamline has an energy resolution of 1 × 10^{-4} eV using a dual Si(111) crystal monochromator, a flux > 1 × 10^{11} photons/sec, and a spot size approximately 1 × 4 mm, focused using a toroidal mirror. Scans ranged from -30 eV to -5 eV at 2 eV steps in the pre-edge region, from -5 eV to +25 eV at 0.15 eV steps in the near-edge region, and from +25 eV to +55 eV at 0.75 eV steps in the post-edge region, relative to the Mo L_{III}-edge (2525 eV). Two scans were collected for each reference material to ensure replicability.

K-edge XANES and EXAFS analysis – Approximately 20 µg of finely ground reference material was adhered as a thin layer to polyimide (Kapton) tape, folded onto itself, and enclosed in another layer of polyimide tape, to create two layers of each reference material. This method was designed to reduce pinhole effects and self-absorption, while allowing sufficient signal transmission. Several reference materials were not abundant enough to achieve two layers, and therefore one thin layer was used.

Molybdenum K-edge X-ray absorption spectroscopy, including XANES and extended X-ray absorption fine structure (EXAFS) was performed at the BioXAS-Main wiggler beamline (07ID-2) at the CLS. This beamline has an energy resolution of < 1 × 10^{-4} eV with a flux > 1 × 10^{12} photons/second and a spot size approximately 3 × 0.5 mm, focused using Rh-coated toroidal mirrors while a Si(220) double crystal monochromator selects the energy. The setup includes three ionization chambers and two Ge-32 element fluorescence detectors, along with a liquid nitrogen cryostat, Soller slits, and a Zr6 fluorescence filter to enhance the signal to noise ratio. Spectra were collected in fluorescence mode, and additional transmission spectra were collected when enough sample was present. Fluorescence and transmission scans ranged from -200 eV below the theoretical Mo K edge (20,000 eV) to k=15 Å⁻¹ at 5 eV steps in the pre-edge region (i.e., 19800–19900 eV), 0.5 eV steps in the XANES region (i.e., 19900–21000 eV), and 0.05 k in the EXAFS region (i.e., 21000 eV to 15 Å⁻¹). At least two scans per samples ensured replicability, while a Mo reference foil was collected downstream from the sample at each scan for energy calibration and alignment.

XAS Data preparation and treatment – Data analysis was conducted using the Athena program in the Demeter software package [1]. Raw scans were corrected for the total flux in I₀ and imported into Athena as μ(E) as either fluorescence or transmission data for the K-edge or imported as fluorescence or total electron yield data for the L_{III}-edge. Each scan was quality checked and individually deglitched when necessary. Replicate scans were aligned to each other and merged. When one file remained for each reference material, the centroid of the pre-edge peak in the sodium molybdate scan was calibrated to 20010.5 eV for the K-edge and 2523.5 eV for the L_{III}-edge [5], and all other files from the same beamtime were aligned to this file using an energy shift or the associated Mo(0) reference foils. This results in the K-edge Mo(0) foil having an approximate E₀ of 20016.5 eV. An E₀ value for each individual spectra was selected as the highest point in the main peak of the first derivative. Due to a strong pre-edge peak associated with Mo(VI), this selection was not always on the first peak in the first derivative, but the peak associated with the main rising edge in μ(E). The pre-edge range was determined at the L_{III} and K-edges visually, by selecting as large a range as possible while maintaining a relatively flat line. The post-edge, edge-step normalization range used the automatically selected lower bound, as this bound is typically accurate, while manually extending the upper bound of this range as far as the data allowed, and these selections were modified if necessary after viewing the normalized spectra. The normalization step was one arbitrary absorbance unit. Data are reported as the
l₀ corrected, energy calibrated, non-normalized μ(E) data, with this data retaining the edge-step associated with each reference material, as concentrations and signal intensity varied per reference material and by analysis mode. However, data normalized to the edge step are presented visually.

When preparing to plot the χ(k) and χ(R) space for the K-edge spectra, the R background was selected to be between 0.7 and 1.0 with a k-weight of 2 and these values remained constant throughout scans of the same beamtime. This R background value was chosen to minimize the peak in the background function that appeared at approximately k = 1, while also not allowing the background function to invert at k < 1. However, as the forward Fourier transform k-range was set from 3.0-11.0 and data/background below the k-range of 3.0 is ignored, the R background function at these low k values was not highly important. The spline range for the R background function extended throughout the entirety of the dataset from 0-15 k, and corresponded to a spline range in E from 0-857 eV. There was no spline clamp in the low energy range, while a strong spline clamp was used in the high energy range to constrain the background function. The forward Fourier transform parameters used a k-range from 3.0-11.0 with a dk of 3 in a Hanning window, while the backwards Fourier transform was from 1-3 with a dR of 0.0 in a Hanning window.

X-ray diffraction – Synchrotron based powder x-ray diffraction (XRD) for all materials was conducted on the CMCF-BM beamline (08-B1-1) in both manual mode and remote access mode using the Stanford Automated Mounting system. Finely ground powder samples were loaded into 0.5 mm inner diameter polyimide (Kapton) capillaries around 2.2 cm in length. These capillaries were sealed on both ends with ethyl cyanoacrylate adhesive, loaded into magnetic Unipuck stubs, placed on a SSRL cassette, and transported to the CLS where it was mounted for access manually or by a robotic arm onto the goniometer. Capillaries were rotated during the 10-60 second exposure to the 18 keV (λ = 0.6888 Å) beam at room temperature, resulting in a 2θ range of 2-40°. The detector was at a distance of 350 mm and used an aperture of 200 μm. Three to six two-dimensional scans were collected using a high resolution Pilatus3 S 6M X-ray detector. The flux at this beamline is approximately 1.5 × 10¹¹ photons/second with an energy resolution of ~1.5 × 10⁻⁴ eV.

The two-dimensional, concentric images were processed using the program GSAS-II [2], where instrument parameters, including the exact incident wavelength, were calibrated using a LaB₆ calibrant. Following calibration, sample files were imported into GSAS-II and background subtracted using an empty capillary of the same diameter exposed for the same duration as the associated sample. Scans were integrated to convert the two-dimensional image to a line scan, and the line scans binned using equal weighting for each. The data was saved as a powder Topas XYE file, which was manually converted to an .asc file. Data is plotted as normalized from 0-1.

Ethics Statement

The authors declare that the manuscript adheres to ethics in publishing standards.

CRediT Author Statement

Valerie A. Schoepfer: Formal analysis, Validation, Investigation, Data curation, Writing – original draft, Visualization; Matthew B.J. Lindsay: Resources, Validation, Writing – review & editing, Supervision, Funding acquisition.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.
Data Availability

X-ray absorption spectroscopy and X-ray diffraction data for molybdenum minerals and compounds: Datasets and Supplementary Materials (Original data) (Federated Research Data Repository).

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