Molybdenum Isotopes

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Introduction

Molybdenum isotopes are produced by three nucleosynthetic processes: the p- (proton process), r- (rapid neutron capture) and s- (slow neutron capture) processes. The seven naturally occurring isotopes (^{92}Mo, ^{94}Mo, ^{95}Mo, ^{96}Mo, ^{97}Mo, ^{98}Mo and ^{100}Mo) are all stable or extremely long-lived: ^{100}Mo decays to ^{100}Ru with a half-life of ~10^{19} years (NNDC Chart of Nuclides). These isotopes extend over a mass range of ~8% and their natural abundances are rather uniform, (~9 to 25%). These characteristics, in conjunction with improvements in mass spectrometry since the mid-1990s, have resulted in the detection of small fractionations of Mo isotope ratios in meteoritic and terrestrial samples. It is now known that Mo isotopes are heterogeneously distributed in the solar system, resulting in mass-independent variations of Mo isotope ratios in meteoritic samples (Burkhardt et al. 2011). Compared to meteorites, Earth is enriched in s-process Mo isotopes implying, in conjuction with oxygen isotope data, that meteorites might not be representative of the Mo isotopic composition of the bulk Earth (Burkhardt et al. 2011). The improvements in mass spectrometry have also resulted in the recent revision of the absolute isotopic composition and atomic weight of terrestrial Mo (Berglund and Wieser 2011).

Isotope fractionations may vary with the masses of the isotopes: mass-dependent fractionation, or be mass-independent (MIF) e.g. as a result of nucleosynethic processes. The only known natural terrestrial MIF of Mo is in samples from the Oklo natural reactor in Gabon (Bros et al. 2003; Wieser et al. 2012). In contrast, investigations since the late 1990s have shown that mass-dependent fractionation of Mo isotopes in natural samples ranges over 2.4‰/amu and may result from a wide range of physical, chemical and biological processes (Figure 1 and references therein).

Molybdenum Isotopes, fig1 (Mo Isotopes_fig1.eps)

Molybdenum Isotopes, fig1_Caption: Mass dependent fractionation of Mo isotopes in natural and experimental samples. Bars represent the range of values for each category, the references are for the maximum and minimum values reported. Molybdenum isotopic compositions have been renormalised to NIST SRM 3134 where necessary. For additional data sources see Bibliography.

Analytical Methods

Molybdenum isotope ratios are measured by thermal ionisation mass spectrometers (TIMS) and multi-collector inductively-coupled-plasma mass spectrometers (MC-ICP-MS). Analysis of Mo isotopes by TIMS has a long history dating back to the 1960s (see: Wieser and De Laeter 2007) and continues to be used and improved. However, it was the advent of MC-ICP-MS in the 1990s with its improved analytical precision and smaller sample size requirements, compared to TIMS, (Lee and Halliday 1995; Anbar et al. 2001; Siebert et al. 2001) that provided the impetus for the development of Mo isotope geochemistry. Most Mo isotope ratios today are measured by MC-ICP-MS.

Molybdenum isotopic compositions are typically reported in delta-notation, as per mil (‰) deviations in the ^{98}Mo/^{95}Mo ratio relative to a reference standard:

$$\delta^{98}Mo/^{95}Mo_{sample}(‰) = (((^{98}Mo/^{95}Mo_{sample}/^{98}Mo/^{95}Mo_{standard}) - 1) \times 1000$$

In the absence of a certified standard reference material (SRM) for Mo isotope ratios, initial choices of reference standard were arbitrary. Recently, however, the NIST SRM 3134 has been proposed as the international reference standard for Mo isotopes and a number of inter-laboratory calibrations have been made (e.g. Goldberg et al. 2013).

Natural mass dependent fractionation

The important biogeochemistry of Mo, which involves varied redox chemistry and covalent bonding, both known drivers of isotope fractionation, made this element an early target for investigation of stable mass-dependent isotope fractionation in a heavy metal (Anbar 2004). Initial studies focused on samples from low temperature environments, and were quick to notice the similarity in the isotopic composition of Mo in seawater and in sediments deposited under euxinic conditions (i.e. anoxic and sulphidic) and the large isotope fractionation between Mo in seawater and in manganese crusts, which form in oxic seawater conditions (Figure 1). This led to the proposal that the $\delta^{98}Mo/^{95}Mo$ of seawater might co-vary with the relative proportions of anoxic (sic) and oxic sedimentation and thus be utilized as a paleoredox proxy (Barling et al., 2001; Siebert et al., 2003).

The hypothesis that Mo isotope fractionation between seawater and Mn crusts resulted from inefficient scavenging of Mo by Mn oxides under oxic conditions was confirmed experimentally (Barling and Anbar, 2004), while the lack of isotope fractionation between seawater and euxinic sediments was attributed to the quantitative removal of Mo from the water column under euxinic conditions (Helz et al., 1996). Theoretical and sample based studies determined that Mo isotopes are fractionated during the step-wise conversion of molybdate (MoO_4^{2-}) to tetrathiomolybdate (MoS_4^{2-}), but that this is only expressed under conditions where removal of Mo from the water column is non-quantitative (Tossel et al., 2005, Nägler et al., 2011).

Application of Mo isotopes as a paleoproxy requires a sound understanding of the modern marine Mo cycle and characterization of the sources and sinks of Mo (Anbar and Rouxel 2007). Recent data (Figure 1) have confirmed the isotopic homogeneity of seawater and characterized sources (rivers and hydrothermal fluids) and sinks (oxic, sub-oxic, anoxic and euxinic, marine sediments and carbonates) for Mo in the modern ocean. These have been used to revise the original simple box model on which the paleoproxy was based (e.g. Cheng et al. 2015). Since the first application of Mo isotopes as a paleoredox proxy by Arnold et al. (2004) it has become clear that Mo isotopes are not a stand-alone paleoredox proxy, but rather a useful tool in a multi-proxy approach to understanding paleoredox (Lyons et al. 2009).

Application of Mo isotopes as a paleoproxy has largely been focused in the Archean and Proterozoic where they have contributed, in conjunction with other paleoredox proxies, to understanding the rise of oxygen in Earth's early ocean (Lyons et al. 2014). The evolution of oxygenic photosynthesis is generally accepted to be the cause of the rise of oxygen in the atmosphere and oceans, but when this happened is still intensely debated (Farquhar et al., 2011). Planavsky et al. (2014) have used $\delta^{98}Mo/^{95}Mo$ values consistent with Mn oxide interaction to infer oxygenic photosynthesis in Archean (2.95Ga) shallow marine settings. However, it is the Mo isotope record in organic-rich sediments that has received the most attention, providing evidence for a reducing

atmosphere in the Archean prior to 2.7Ga and early "whiffs" of oxygen prior to the Great Oxidation Event (Wille et al. 2013 and references therein). This record exhibits a ~2‰ range in $\delta^{98}Mo/^{95}Mo$ through most of the Proterozoic, intermediate between the narrow range of Archean $\delta^{98}Mo/^{95}Mo$ (<1‰) and the Phanerozoic/present day range (>3‰), reflecting the increasing proportion of oxic ocean floor (Figure 1). The Phanerozoic is however punctuated by Ocean Anoxic Events and more local fluctuations in ocean redox that have been investigated using Mo isotopes (e.g. Dickson et al. 2016). Molybdenum isotopes have also been used to investigate the connection between ocean redox and radiations and extinctions of the fossil record since the Ediacaran (ca. 635-542 Ma; e.g. Chen et al. 2015).

Most paleoredox studies have been on the marine record but more recently attention has turned to continentally derived clastic sediments. These provide a record of contemporaneous atmospheric oxygen levels and like the marine record of organic-rich sediments, testify to the rise of oxygen through Earth's history (Figure 1). The average Mo isotopes of clastic sediments show that the continental crust has become lighter over time suggesting that supracrustal processes have resulted in the preferential loss of heavy Mo to the hydrosphere (Yang et al. 2015a). Samples from other continental settings such as estuaries, lakes and in soils have received less attention, as has Mo isotope fractionation related to biological and human activity (Figure 1).

Even though it has been clear from Mo isotope measurements of molybdenites (Breillat et al. 2016 and references therein) that hydrothermal processes (200 – 1200°C) are associated with significant Mo isotope fractionation (Figure 1), the potential for Mo isotope fractionation in other high temperature environments has been largely overlooked until recently. It is now known that magmatic systems, particularly those involving a fluid phase (arc volcanics and granites), exhibit Mo isotope fractionations spanning nearly 2.5‰. In contrast, Mo isotope fractionation is absent during crystal fractionation in anhydrous magmatic systems (Yang et al. 2015b). Mo isotope variations in the mantle, MORB and OIB are small and may reflect the presence of residual sulphide melts during small degrees of partial melting of the mantle (Liang et al. 2016). The Mo concentrations and isotopic compositions of basalts, komatiites and peridotites have also been used to constrain the Mo concentration and isotopic composition of the BSE (Figure 1). Average Mo isotope ratios for magmatic iron meteorites and enstatite-, ordinary- and carbonaceous chondrites typically overlap the BSE estimate, while Mo isotopes derived from the silicate portion of differentiated planetary bodies trend to values heavier than terrestrial magmatic samples (Burkhardt et al. 2014). Lunar basalts have Mo isotopes similar to MORB (Burkhardt et al. 2014).

Interpretation of Mo isotope fractionation in natural samples is aided by theoretical and experimental investigations of processes with the potential to fractionate isotopes. The relatively few such studies have investigated biological activity, adsorption, aqueous speciation (Malinovsky et al. 2007b), leaching of mine waste (Skierszkan et al., 2016) and partitioning between metal and silicate liquids (Figure 1).

Summary or Conclusions

The seven naturally occurring isotopes of Mo have rather uniform abundances and extend over a mass range of ~8%. These characteristics, in conjunction with the

interesting biogeochemistry of Mo, made Mo an early target for investigation of stable mass-dependent isotope fractionation in a heavy metal. To date, Mo isotope ratios in natural samples range over 2.4‰/amu. Investigations have focused predominantly on marine samples leading to a better understanding of the modern marine Mo cycle. Initial observations of the contrasting Mo isotopic compositions of modern oxic and euxinic sediments led to the proposal that the $\delta^{98}Mo/^{95}Mo$ could be developed as a paleoredox proxy. Since then Mo isotopes have become a useful tool in multi-proxy investigations of paleoredox, contributing in particular, to understanding of the rise of oxygen through Earth's history.

Mo isotope fractionation is less well documented in samples from continental environments, for biological and human activity and for samples from high temperature settings. There have also been comparatively few theoretical and experimental investigations of Mo isotope fractionation. Given the wide range in $\delta^{98}Mo/^{95}Mo$ in samples associated with high and low temperature fluids, analysis and theoretical and experimental investigations of samples and processes involving a fluid phase seem likely to be promising areas of future research.

Cross-references

Ab Initio Calculations
Analytical Techniques
Atmospheric Evolution
Atomic Number, Mass Number, and Isotopes
Black Shales and Sapropels
Fe-Mn Crusts and Nodules
Inductively Coupled Plasma Mass Spectrometry (ICP-MS)
Molybdenum
Nucleosynthesis
Stable isotope geochemistry
Thermal Ionization Mass Spectrometry

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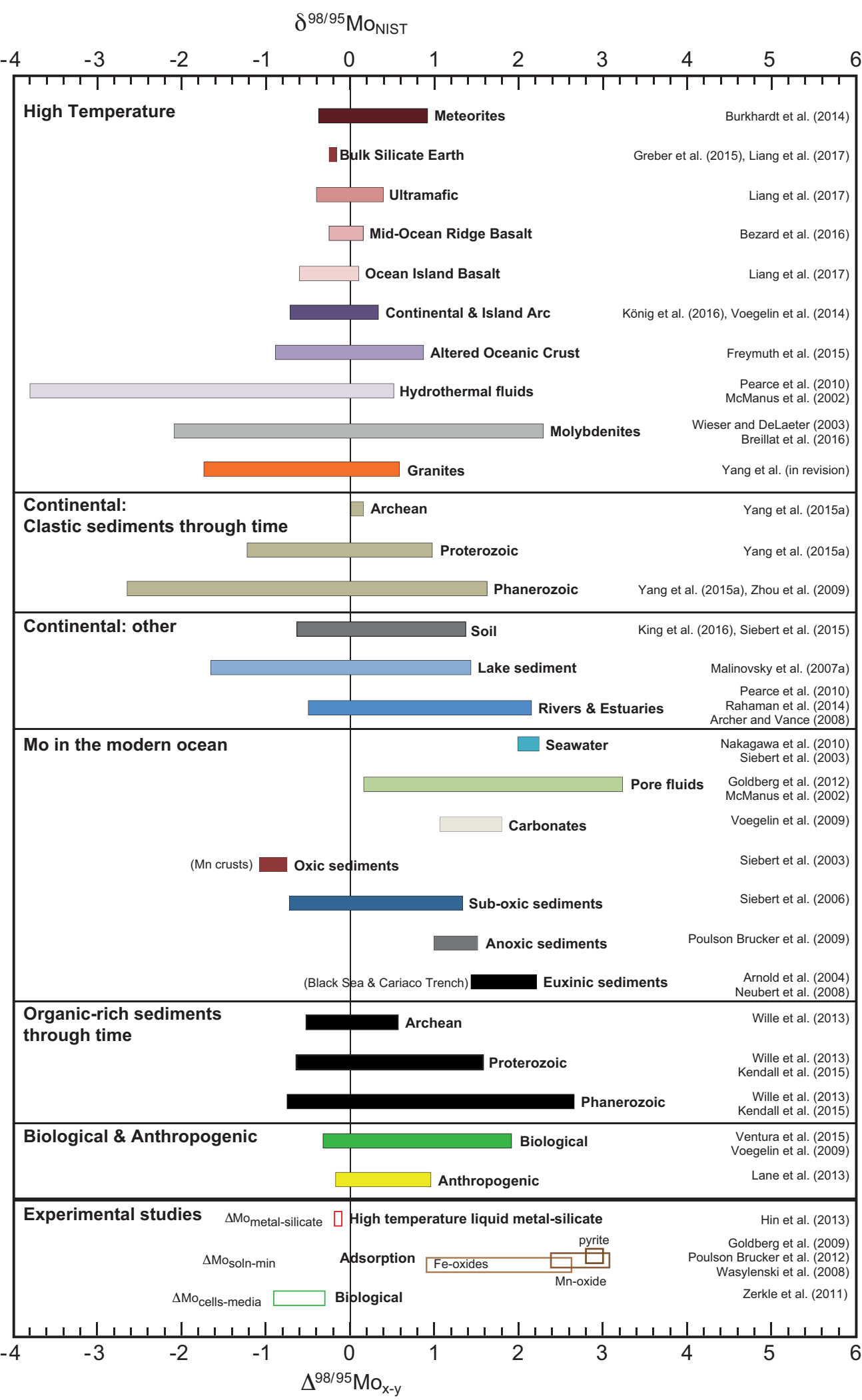
Figure 1. Mass-dependent fractionation of Mo isotopes in natural and experimental samples. Bars represent the range of values for each category; the references are for the maximum and minimum values reported. Molybdenum isotopic compositions have been renormalized to NIST SRM 3134 where necessary. For additional data sources see "References."


δ98/95MoNIST
-4
-3
-2
-1
0
1
2
3
4
5
6
High Temperature
Meteorites
Burkhardt et al. (2014)
Bulk Silicate Earth
Greber et al. (2015), Liang et al. (2017)
Ultramafic
Liang et al. (2017)
Mid-Ocean Ridge Basalt
Bezard et al. (2016)
Ocean Island Basalt
Liang et al. (2017)
Continental & Island Arc
König et al. (2016), Voegelin et al. (2014)
Altered Oceanic Crust
Freymuth et al. (2015)
Hydrothermal fluids
Pearce et al. (2010)
McManus et al. (2002)
Molybdenites
Wieser and DeLaeter (2003)
Breillat et al. (2016)
Granites
Yang et al. (in revision)
Continental:
Clastic sediments through time
Archean
Yang et al. (2015a)
Proterozoic
Yang et al. (2015a)
Phanerozoic
Yang et al. (2015a), Zhou et al. (2009)
Continental: other
Soil
King et al. (2016), Siebert et al. (2015)
Lake sediment
Malinovsky et al. (2007a)
Rivers & Estuaries
Pearce et al. (2010)
Rahaman et al. (2014)
Archer and Vance (2008)
Mo in the modern ocean
Seawater
Nakagawa et al. (2010)
Siebert et al. (2003)
Pore fluids
Goldberg et al. (2012)
McManus et al. (2002)
Carbonates
Voegelin et al. (2009)
(Mn crusts)
Oxic sediments
Siebert et al. (2003)
Sub-oxic sediments
Siebert et al. (2006)
Anoxic sediments
Poulson Brucker et al. (2009)
(Black Sea & Cariaco Trench)
Euxinic sediments
Arnold et al. (2004)
Neubert et al. (2008)
Organic-rich sediments
through time
Archean
Wille et al. (2013)
Proterozoic
Wille et al. (2013)
Kendall et al. (2015)
Phanerozoic
Wille et al. (2013)
Kendall et al. (2015)
Biological & Anthropogenic
Biological
Ventura et al. (2015)
Voegelin et al. (2009)
Anthropogenic
Lane et al. (2013)
Experimental studies
ΔMometal-silicate
High temperature liquid metal-silicate
Hin et al. (2013)
pyrite
ΔMosoln-min
Adsorption
Fe-oxides
Mn-oxide
Goldberg et al. (2009)
Poulson Brucker et al. (2012)
Wasylenski et al. (2008)
ΔMocells-media
Biological
Zerkle et al. (2011)
-4
-3
-2
-1
0
1
2
3
4
5
6
Δ98/95Mox-y