

Triple oxygen isotope variations in magnetite from iron-oxide deposits, central Iran, record magmatic fluid interaction with evaporite and carbonate host rocks

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ABSTRACT

Oxygen isotope ratios in magnetite can be used to study the origin of iron-oxide ore deposits. In previous studies, only $^{18}\text{O}/^{16}\text{O}$ ratios of magnetite were determined. Here, we report triple O isotope data ($^{17}\text{O}/^{16}\text{O}$ and $^{18}\text{O}/^{16}\text{O}$ ratios) of magnetite from the iron-oxide–apatite (IOA) deposits of the Yazd and Sirjan areas in central Iran. In contrast to previous interpretations of magnetite from similar deposits, the triple O isotope data show that only a few of the magnetite samples potentially record isotopic equilibrium with magma or with pristine magmatic water (H_2O). Instead, the data can be explained if magnetite had exchanged O isotopes with fluids that had a mass-independently fractionated O isotope composition (i.e., MIF-O), and with fluids that had exchanged O isotopes with marine sedimentary carbonate rocks. The MIF-O signature of the fluids was likely obtained by isotope exchange with evaporite rocks of early Cambrian age that are associated with the IOA deposits in central Iran. In order to explain the triple O isotope composition of the magnetite samples in conjunction with available iron isotope data for magnetite from the deposits, we propose that magnetite formed from magmatic fluids that had interacted with evaporite and carbonate rocks at high temperatures and at variable water/rock ratios; e.g., magmatic fluids that had been released into the country rocks of a magma reservoir. Additionally, the magnetite could have formed from magmatic fluids that had exchanged O isotopes with SO_2 and CO_2 that, in turn, had been derived by the magmatic assimilation and/or metamorphic breakdown of evaporite and carbonate rocks.

INTRODUCTION

The formation of iron-oxide–apatite (IOA) deposits is much debated. Studies on the origin of IOA deposits have often focused on the $\delta^{18}\text{O}$ values of magnetite, which were suggested to reflect isotopic equilibrium with magmas or with magmatic water; i.e., H_2O in equilibrium with magma (e.g., Jami et al., 2007; Nyström et al., 2008; Knipping et al., 2015; Bilenker et al., 2016; Johnson et al., 2016). In some cases, the $\delta^{18}\text{O}$ of the magnetite was interpreted to also reflect isotopic equilibrium with water of a nonmagmatic origin (e.g., Rhodes and Oreskes, 1999). Based on $\delta^{18}\text{O}$ data, magnetite from IOA deposits was therefore suggested to have predominantly formed by magmatic and magmatic-hydrothermal processes, whereas a minor

portion of the magnetite would have formed by late-stage hydrothermal processes that involved fluids of a nonmagmatic origin (Jonsson et al., 2013; Troll et al., 2019).

Additional constraints on the origin of IOA deposits may be obtained from determining not only $\delta^{18}\text{O}$, but also $\delta^{17}\text{O}$ of magnetite. Mass-dependent isotope fractionation processes can result in variable $\delta^{17}\text{O}/\delta^{18}\text{O}$ between different rocks and minerals, due to variations in the triple O isotope exponent theta (θ ; e.g., Pack and Herwartz, 2014; Sharp et al., 2018). Studying the triple O isotope composition (i.e., $\delta^{17}\text{O}$, $\delta^{18}\text{O}$) of magnetite from IOA deposits therefore potentially provides better constraints on the phases with which magnetite isotopically exchanged when it formed. A second motivation for studying the triple O isotope com-

positions of magnetite is the fact that evaporite rocks were conjectured to play a role in the formation of iron-oxide deposits (e.g., Barton and Johnson, 1996; Hitzman, 2000; Torab and Lehmann, 2007; Li et al., 2015; Ghazi et al., 2019). Oxygen in evaporitic sulfate can in part be derived from atmospheric O_2 and, consequently, carry a mass-independent deficit in ^{17}O relative to ^{16}O and ^{18}O (e.g., Crockford et al., 2019). Determining the triple O isotope composition of magnetite from IOA deposits therefore potentially allows us to test whether evaporite rocks were involved in the formation processes of these deposits.

We determined the triple O isotope compositions of 23 magnetite samples from IOA deposits in the Yazd and Sirjan areas in central Iran (Fig. 1). The mineral assemblages of these deposits are characteristic of “Kiruna-type” ore deposits. They are hosted within and close to early Cambrian felsic igneous rocks (Ramezani and Tucker, 2003). For comparison, a sample from a Cenozoic granite-related magnetite skarn deposit in the Sangan area in northeastern Iran was also analyzed. We studied the triple O isotope compositions of magnetite from the IOA deposits of the Yazd and Sirjan areas, in particular, because the country rocks of the felsic intrusions and their IOA deposits consist of limestone and evaporite rocks of late Ediacaran to early Cambrian age (Faramarzi et al., 2015). Evaporites from the Ediacaran and early Cambrian carry large ^{17}O deficits compared to evaporites that formed later in the Phanerozoic (Bao et al., 2008; Crockford et al., 2019). The deposits from these areas are therefore well suited to investigating the putative role of evaporite rocks in the formation of iron-oxide deposits.

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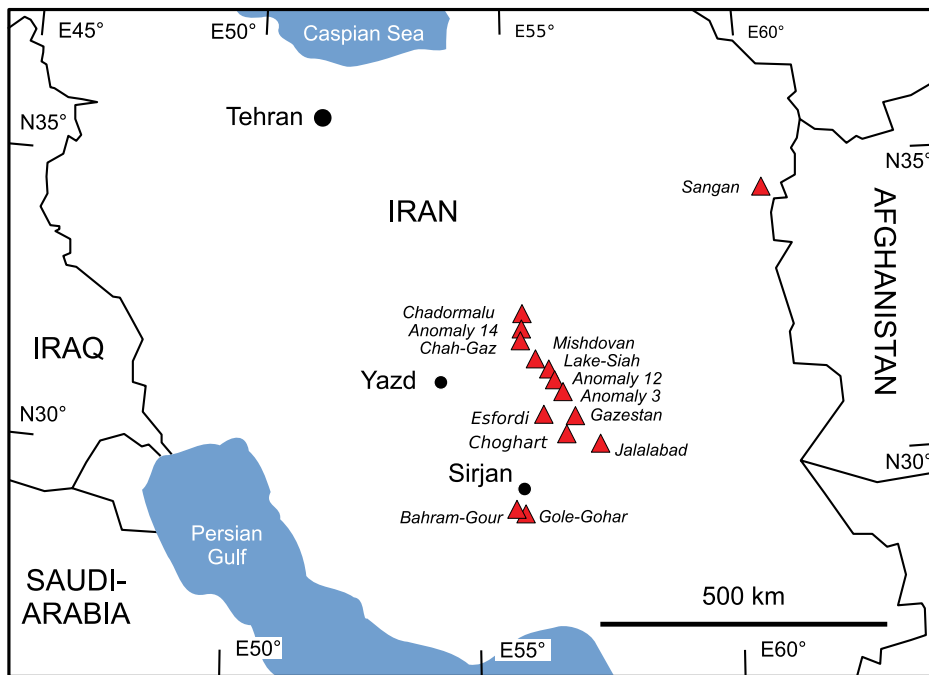


Figure 1. Location of magnetite deposits in central Iran that were sampled for this study.

METHODS AND DEFINITIONS

Magnetite samples were taken from high-grade magnetite ore from run-of-the-mine material in open pits and drill cores. Individual, millimeter-sized magnetite crystals were hand-picked for triple O isotope analysis. Oxygen was extracted from the samples as O_2 by laser fluorination, using BrF_3 as the fluorinating agent, at the University of Göttingen, Germany (Pack et al., 2016). The extracted O_2 was cleaned from contaminant gases using freeze-thaw techniques in combination with liquid nitrogen, a 5 Å mesh molecular sieve, and a gas chromatograph. The cleaned sample gas was released at 65 °C from a molecular sieve into the sample bellows of a dual-inlet system of a MAT253 gas-source mass spectrometer. The O isotope composition of the sample gas was then measured as $m/z = 32, 33,$ and 34 relative to a reference gas that had been calibrated against the Vienna standard mean ocean water 2 (VSMOW2) standard. The samples were analyzed together with UWG2 garnet standard as an external reference material (Valley et al., 1995). In order to test the reproducibility of the laser fluorination method for iron oxides, we also performed replicate extractions of a hematite pseudomorph after magnetite from a chlorite schist (NZ-IO) of the Pounamu ultramafic belt (New Zealand) as an internal reference material (Cooper and Ireland, 2013, 2015).

Measured oxygen isotope ratios ($^{17}O/^{16}O, ^{18}O/^{16}O$) of samples are henceforth expressed as $\delta^{17}O$ and $\delta^{18}O$; i.e., as per mil deviation from the VSMOW2 standard (McKinney et al., 1950). Variations in $\delta^{17}O/\delta^{18}O$ ratios between samples are conventionally expressed as the pa-

rameter $\Delta^{17}O$, which denotes the deviation in linearized triple O isotope space of a sample's $\delta^{17}O$ from its $\delta^{18}O$ relative to a reference line with given slope and intercept, in ppm. Based on the considerations by Sharp et al. (2018), we used a reference line with a slope of 0.528 to express the composition of our samples, i.e., $\Delta^{17}O_{0.528} = 1000 \times (\delta^{17}O - 0.528 \times \delta^{18}O)$. The primes in the notation indicate that the measured δ values were linearized (i.e., $\delta^{17,18}O = 1000 \times \ln[\delta^{17,18}O/1000 + 1]$; Miller, 2002) for calculation of $\Delta^{17}O_{0.528}$, whereas the subscript 0.528 indicates the choice of slope. The $\Delta^{17}O_{0.528}$ values for the samples are reported relative to VSMOW2, using the calibration of San Carlos olivine on the VSMOW2 scale by Pack et al. (2016). The mean values for the internal iron oxide reference material NZ-IO were $\delta^{18}O = -1.43\text{‰} \pm 0.06\text{‰}$ and $\Delta^{17}O_{0.528} = -41 \pm 10$ ppm (\pm standard deviation [SD], $n = 7$; Table DR1 in the GSA Data Repository¹).

RESULTS

The $\delta^{18}O$ values of magnetite samples from the Yazd and Sirjan areas vary between 1.9‰ and 6.9‰ (Table DR2); i.e., in good agreement with previously published $\delta^{18}O$ data for mag-

¹GSA Data Repository item 2020057, Table DR1 (analytical reproducibility of internal reference material NZ-IO for triple oxygen isotope analysis of iron oxides), Table DR2 (triple oxygen isotope compositions of magnetite from iron oxide deposits in central Iran), and a supplementary discussion and specification of model parameters, is available online at <http://www.geosociety.org/datarepository/2020/>, or on request from editing@geosociety.org.

netite from deposits in these areas (e.g., Moore and Modabberi, 2003; Jami et al., 2007; Taghipour et al., 2015; Ghazi et al., 2019). The corresponding $\Delta^{17}O_{0.528}$ of magnetite ranges from -35 to -174 ppm (Table DR2). The lowest $\Delta^{17}O_{0.528}$ values in the data set are among the lowest $\Delta^{17}O_{0.528}$ values that have been reported for terrestrial rocks and minerals so far, at the given range of $\delta^{18}O$ values, together with some samples of carbonate-associated sulfate and evaporites (Bao et al., 2008; Crockford et al., 2018, 2019). Magnetite from the skarn ore of the Sangan area has a lower $\delta^{18}O$ and a higher $\Delta^{17}O_{0.528}$ ($\delta^{18}O = -0.68\text{‰}$; $\Delta^{17}O_{0.528} = -19$ ppm) than magnetite from the IOA deposits of the Yazd and Sirjan areas.

DISCUSSION

The $\Delta^{17}O_{0.528}$ values of some of the magnetite samples from the Yazd and Sirjan areas are close to -40 ppm; i.e., the $\Delta^{17}O_{0.528}$ of these samples is similar to most igneous rocks and minerals (Fig. 2). The $\delta^{18}O$ values of these samples are typical for magnetite that is in equilibrium with magma and with magmatic water; i.e., typical for “orthomagmatic magnetite” ($\delta^{18}O = 1\text{‰}–4\text{‰}$; Taylor, 1968). These particular samples were therefore potentially in equilibrium with magma or with magmatic water at high temperatures when they formed. The majority of the samples, however, have $\Delta^{17}O_{0.528}$ values $\ll -40$ ppm. Those samples cannot have been in equilibrium with magma or with pristine magmatic water when they formed. The samples were not in equilibrium with fluids derived from seawater or from meteoric water either, because magnetite that is in equilibrium with such fluids has $\Delta^{17}O_{0.528} > -40$ ppm (Fig. 2).

Three End-Member Mixing Model for Magnetite Triple O Isotope Variations

In triple O isotope space, the magnetite samples from central Iran define trends with higher $\Delta^{17}O_{0.528}/\delta^{18}O$ slopes than predicted for mass-dependent O isotope variations (Fig. 2). The variations in magnetite triple O isotope compositions may therefore reflect mixing of isotopically distinct magnetite end-member components in different proportions. At least three magnetite end-member components are apparently reflected in the triple O isotope compositions of the samples (Fig. 3). One magnetite end-member component (Mgt A) has $\delta^{18}O$ and $\Delta^{17}O_{0.528}$ similar to orthomagmatic magnetite; i.e., $\delta^{18}O = 1\text{‰}–4\text{‰}$ and $\Delta^{17}O_{0.528} \sim -47$ ppm. The second magnetite end-member component (Mgt B) has $\delta^{18}O$ similar to orthomagmatic magnetite, but it has a significantly lower $\Delta^{17}O_{0.528}$ than orthomagmatic magnetite; i.e., $\Delta^{17}O_{0.528}$ is approximately -200 ppm. The third magnetite end-member component (Mgt C) has a higher $\delta^{18}O$ than orthomagmatic magnetite ($\delta^{18}O > 4\text{‰}$), and it has $\Delta^{17}O_{0.528}$ in between the two other end-member components.

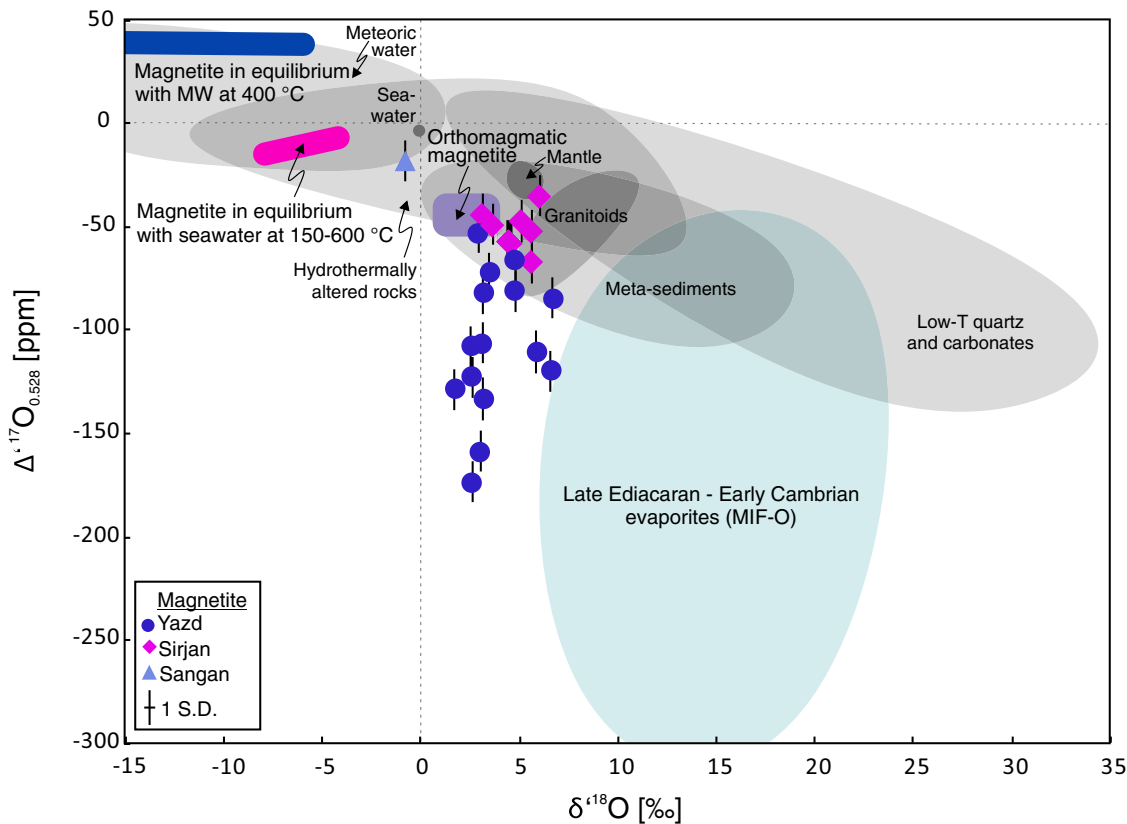


Figure 2. Triple O isotope compositions of magnetite from iron-oxide deposits in central Iran, compared to mass-dependently fractionated oxygen in common rocks, minerals, and water, and mass-independently fractionated oxygen (MIF-O) in late Ediacaran to early Cambrian evaporites (data sources: Sharp et al., 2018, and references therein; Crockford et al., 2019, and references therein). Plotted are the linearized $\delta^{18}\text{O}$ values (i.e., $\delta^{18}\text{O}$) of the samples versus their $\Delta^{17}\text{O}$, expressed relative to a value for λ of 0.528 (indicated by subscript), at the 1 standard deviation (S.D.) level. Also shown are modeled compositions of magnetite that is in equilibrium with magma and magmatic water (i.e., “orthomagmatic magnetite”), magnetite that is in equilibrium with seawater at a temperature (T) range of 150–600 °C, and magnetite that is in equilibrium with meteoric

water (MW) at 400 °C, respectively. Field for orthomagmatic magnetite shows the commonly cited range in $\delta^{18}\text{O}$ of 1‰–4‰ (Taylor, 1968), in conjunction with modeled $\Delta^{17}\text{O}_{0.528}$ of -47 ± 10 ppm for magnetite that is in equilibrium with igneous rocks, excluding S-type granitoids (see the Data Repository [see footnote 1]; Pack and Herwartz, 2014; Pack et al., 2016; Sharp et al., 2018). Fields for magnetite that is in equilibrium with seawater, and magnetite that is in equilibrium with meteoric water, were calculated from fractionation factors and theta values for magnetite-water equilibrium from Hayles et al. (2018), using the composition of modern seawater ($\delta^{18}\text{O} = 0$; $\Delta^{17}\text{O}_{0.528} = -5$ ppm) and modern meteoric waters (Luz and Barkan, 2010).

Mgt B: Influence of MIF-O from Evaporitic Sulfate

The lowest $\Delta^{17}\text{O}_{0.528}$ values in the data set, at the corresponding $\delta^{18}\text{O}$ of the samples, plot significantly below the array of mass-dependent O isotope variations (Fig. 2). It is therefore conceivable that the magnetite end-member component with $\Delta^{17}\text{O}_{0.528} \sim -200$ ppm (Mgt B) contains oxygen with a relative deficit in ^{17}O , i.e., oxygen with a mass-independently fractionated isotope composition (MIF-O). Terrestrial rocky materials that carry MIF-O have gained oxygen from atmospheric O_2 . Rocky materials that are known to have incorporated atmospheric O_2 are biopapatite (Gehler et al., 2011), cosmic spherules (Pack et al., 2017), some tektites (Magna et al., 2017), possibly ocean-floor manganese nodules (Sharp et al., 2018), carbonate-associated sulfate (CAS), and sulfate in marine barite and in evaporite rocks (Bao et al., 2008; Crockford et al., 2018, 2019). It is therefore probable that the magnetite end-member component that is recorded in the data set with $\Delta^{17}\text{O}_{0.528} \sim -200$ ppm (Mgt B) formed from fluids that had exchanged O isotopes with the early Cambrian evaporite rocks that are associated with the IOA deposits in central Iran. The $\Delta^{17}\text{O}_{0.528}$ of these evaporite rocks was likely similar to that of the evaporite rocks from the

Hormuz Formation in south Iran, which is stratigraphically equivalent to the evaporite horizons in the Yazd and Sirjan areas. Gypsum samples from the Hormuz Formation have an average $\Delta^{17}\text{O}_{0.528}$ of ~ -200 ppm (Crockford et al., 2019).

Mgt C: Influence of Oxygen from Marine Sedimentary Carbonates

The magnetite end-member component with $\delta^{18}\text{O} > 4\text{‰}$ that is recorded in the data set (Mgt C) exchanged O isotopes with a phase that had higher $\delta^{18}\text{O}$ than most magmas and magmatic H_2O ($\delta^{18}\text{O} = 5.5\text{‰}$ – 10‰ , excluding S-type granitoids; Bindeman, 2008; Eiler, 2001), and that had a higher $\Delta^{17}\text{O}_{0.528}$ than evaporite rocks. Such a triple O isotope composition is predicted for fluids that have interacted with marine sedimentary carbonate rocks ($\delta^{18}\text{O} = \sim 20\text{‰}$ – 30‰ ; Hoefs, 2009). The third magnetite end-member component (Mgt C) therefore possibly records magnetite that formed from fluids that had exchanged O isotopes with the marine sedimentary carbonate rocks that are associated with the iron-oxide deposits in central Iran, and/or with CO_2 that was derived from those carbonate rocks. The possible relevance of fluid interaction with carbonate rocks in the formation of the IOA deposits is also demonstrated by the presence of

abundant secondary carbonate minerals in most of the deposits in central Iran (Jami et al., 2007; Daliran et al., 2010; Heidarian et al., 2017; Deymar et al., 2018).

Geological Mechanisms of Isotope Exchange

In order to explain the triple O isotope compositions of the magnetite samples, we propose that magnetite from the IOA deposits in the Yazd and Sirjan areas formed from magmatic fluids that had exchanged O isotopes with evaporite and carbonate rocks at variable water/rock ratios (Fig. 3). The available iron isotope data for magnetite from the deposits in the Yazd and Sirjan areas ($\delta^{56}\text{Fe} = 0.2\text{‰}$ – 0.5‰) indicate a high-temperature magmatic (e.g., ≥ 800 °C) rather than a low-temperature hydrothermal origin for the magnetite (Troll et al., 2019; Alibabae et al., 2019, personal commun.). We therefore propose two geological mechanisms by which O isotopes could have been exchanged at high temperatures between magmatic fluids and evaporite and carbonate rocks. First, magmatic fluids could have reacted with evaporite and carbonate rocks, e.g., as would be the case for magmatic fluids that were injected into the country rocks of a magma reservoir. A second

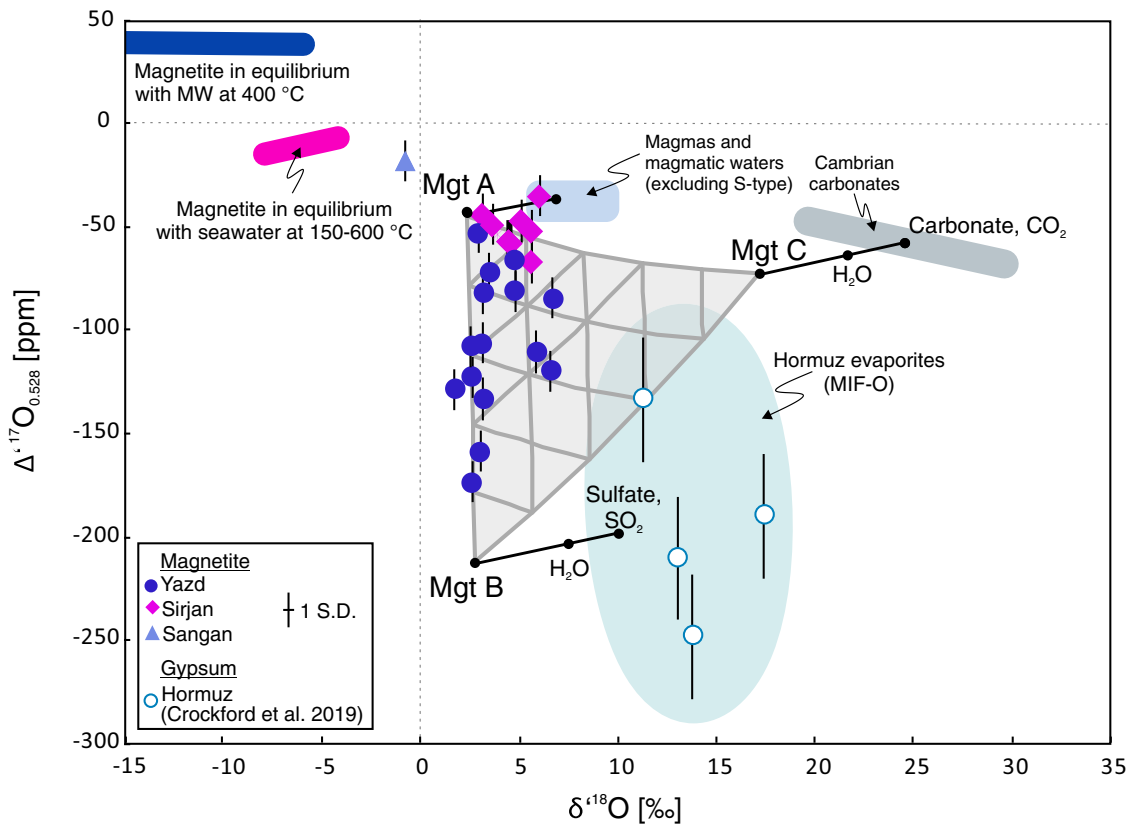


Figure 3. Triple O isotope compositions of magnetite from Yazd and Sirjan iron-oxide-apatite (IOA) deposits, compared to suggested end-member magnetite compositions (Mgt A–C) that are in O isotope equilibrium at high temperatures (~800 °C) with: (A) magmas and magmatic waters (excluding S-type granitoids); (B) aqueous fluids that had exchanged O isotopes with sulfate or with SO₂ from early Cambrian evaporites; or (C) aqueous fluids that had exchanged O isotopes with carbonate or with CO₂ from Cambrian carbonate rocks. Plotted are the linearized δ¹⁸O values (i.e., δ¹⁸O) of the samples versus their Δ¹⁷O, expressed relative to a value for λ of 0.528 (indicated by subscript). Triangular field depicts putative mixing between suggested magnetite end-member compositions (increments correspond to 20% mixing). Black lines depict isotope fractionation

between indicated phases and were constructed from fractionation factors and theta values, in conjunction with literature triple O isotope data, which are detailed in the Data Repository (see footnote 1). For simplicity, common fractionation lines are shown for the sulfate-water system and SO₂-water system, as well as for the carbonate-water system and CO₂-water system, despite minor differences in Δ¹⁸O between those systems at high temperatures. Fields for magmas and magmatic waters correspond to δ¹⁸O values of 5.7‰–10‰, in conjunction with a Δ¹⁷O_{0.528} of -40 ± 10 ppm (see the Data Repository). The field for evaporites from the Hormuz Formation is plotted together with data for individual samples (Crockford et al., 2019). The array for Cambrian carbonates depicts rocks with δ¹⁸O = 20‰–30‰ that formed in equilibrium with modern seawater (δ¹⁸O = 0, Δ¹⁷O_{0.528} = -5 ppm; Luz and Barkan, 2010) at an average theta value of 0.5258 (Hayles et al., 2018). MW—meteoric water; S.D.—standard deviation.

possible scenario is that magmatic fluids exchanged O isotopes with SO₂ and CO₂ that, in turn, had been derived by the magmatic assimilation and/or metamorphic breakdown of evaporite and carbonate rocks, respectively (cf. Rye et al., 1984; Sharp et al., 2003; Troll et al., 2012; see the Data Repository). The latter suggestion is supported by, e.g., the occurrence of dolomite xenoliths in the volcanic sequences that host some of the deposits, which attest to the magmatic assimilation of carbonate rocks (Moore and Modabber, 2003).

The conclusion that magnetite from the iron-oxide deposits in central Iran formed from magmatic fluids that had interacted with evaporite and carbonate rocks is in good agreement with the continuum that is found between, respectively, magmatic and crustal δ³⁴S in sulfide minerals from the deposits, and also with the continuum between magmatic and crustal δ¹³C and δ¹⁸O in carbonate minerals from the deposits (Jami et al., 2007; Heidarian et al., 2017). Whereas magnetite likely formed from fluids at magmatic temperatures, sulfide and carbonate minerals in the deposits, and also apatite, likely formed when temperatures of the magmatic fluids had

dropped below about <600 °C (Heidarian et al., 2017). Whereas the triple O isotopes of magnetite from the deposits in the Yazd area apparently predominantly record the interaction between magmatic fluids and evaporite rocks, magnetite from the deposits in the Sirjan area, in contrast, apparently predominantly records the interaction between magmatic fluids and carbonate rocks (Fig. 3). This conclusion demonstrates that the triple O isotope composition of magnetite, in general, may help us to better understand the geological and lithological contexts within which iron-oxide deposits formed.

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