

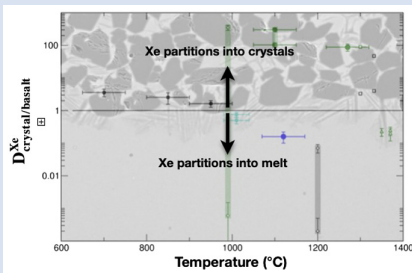
Xenon compatibility in magmatic processes: Hadean to current contexts

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Abstract



Xenon (Xe) behaviour in petrological processes, albeit essential to constrain mantle ingassing and degassing models, is elusive due to its volatile nature, and lack of direct investigation at the pressures (P) and temperatures (T) relevant to magma formation and crystallisation at depth. Xenon stands out amongst noble gases due to its unique reactivity with silicates of the lower crust and upper mantle, which could at least partially explain that published mineral/melt partitioning coefficients span up to six orders of magnitude. We report partition coefficients of Xe using *in situ* X-ray fluorescence at high P and T , and mass spectrometry analyses. Xenon is found to be moderately incompatible in anorthite-clinopyroxene mix in equilibrium with basalt (partition coefficient value of 0.16 ± 0.06), and compatible in olivine in equilibrium with basalt (partition coefficients in the range 88 ± 22 to 302 ± 46). While Xe is, thus, concentrated in basaltic melts coexisting with crystallising pyroxenes and feldspars, it is strongly retained in olivine at depth. Consequently, Xe originally contained in solid Earth has been preferentially retained at depth throughout Earth's history, from the magma ocean stages to present day partial mantle melting processes.

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Introduction

Noble gases provide unique clues to unravel the geochemical evolution of volatile elements upon Earth's formation to present day geodynamics (Ozima *et al.*, 2002 and references therein), assuming their inertness and volatile behaviour. Xenon is also unique for its enigmatic atmospheric depletion relative to lighter noble gases (Anders and Owen, 1977), known as the 'Xe paradox', and its strong depletion in light isotopes (Krummenacher *et al.*, 1962). Atmospheric escape and trapping-at-depth scenarios have been proposed to explain both observations (Ardoin *et al.*, 2022; Broadley *et al.*, 2022; Rzeplinski *et al.*, 2022 and references therein), and are not exclusive. Atmospheric escape models stem from the fact that Xe is the easiest to ionise amongst noble gases and to investigate regarding how this could be triggered by extreme UV radiation of the young Sun if sufficient amount of hydrogen or organic matter were present. However, the main challenge is how to lift the heavy Xe through the atmosphere up to levels where it can be lost (Zahnle *et al.*, 2019). Trapping-at-depth scenarios, in turn, suffer from a lack of knowledge on Xe petrological behaviour. Noble gases partitioning between major minerals and melt is indeed a debated issue, with Xe spanning the largest range amongst noble gases crystal/melt partitioning data (noted as $D_{\text{crystal/melt}}^{\text{Xe}}$), with up to 6 orders of magnitude from 6×10^{-4} to 351 for $D_{\text{olivine/basalt}}^{\text{Xe}}$ (Hiyagon and Ozima, 1986; Broadhurst *et al.*, 1992; Heber *et al.*, 2007).

The published experimental $D_{\text{crystal/melt}}^{\text{Xe}}$ values were obtained from samples brought to high T , equilibrated with a noble gas medium either at atmospheric P or ~ 110 MPa, and quenched to room conditions for chemical analysis. A few experiments were carried out at higher P up to 1.5 GPa (Hiyagon and Ozima, 1986) but with only adsorbed air on starting sample as noble gas source. After correction for eventual melt inclusions in crystals, the very large range of $D_{\text{crystal/melt}}^{\text{Xe}}$ values partly results from the interpretation of bubbles in minerals, *i.e.* whether they should be excluded from measurements or not. Indeed, while noble gas content in melts is homogeneous, such is not the case in minerals, with almost systematic reports of heterogeneous distribution of heavy noble gases, often at the micron or sub-micron scale (Hiyagon and Ozima, 1986; Broadhurst *et al.*, 1992; Heber *et al.*, 2007). Some data were discarded on this ground, despite sometimes clear elemental fractionation from the original gas (Hiyagon and Ozima, 1986), which is not expected for passively trapped gas. Indeed, Xe was observed to retro-diffuse out of olivine in high P experiments upon T -quenching (Sanloup *et al.*, 2011), an exsolution process that could explain at least part of the bubbles observed on quenched samples. It is therefore very challenging to interpret the heterogeneous distribution of Xe in quenched minerals recovered from experiments.

Another source of controversy arises from the impact of noble gases adsorption on minerals, *i.e.* whether or not it is significant in experiments, and therefore if it should be corrected

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for or not. Step heating experiments show that heavy noble gases are tightly bound in minerals, with largest fractions being released above 1000 °C (Hiyagon and Ozima, 1986), arguing against physical adsorption.

Last but not least, datasets are difficult to compare due to the different compositions used for both melts and crystals (Table 1). For instance, olivine–melt experiment compositions range from synthetic Fe-free forsterite/61 % SiO₂-rich melt (Heber *et al.*, 2007) to natural olivine/basalt (Hiyagon and Ozima, 1986). It is, nonetheless, established that melt composition strongly affects trace element partitioning (Schmidt *et al.*, 2006), with one order of magnitude difference between gabbroic and granitic melts, an effect also expected to be strong for noble gases based on solubility values in melts (Carroll and Stolper, 1993; Schmidt and Keppler, 2002).

Facing these experimental controversies, the peridotitic database shows an enrichment in Xe over other noble gases in xenoliths (Hennecke and Manuel, 1975; Poreda and Farley, 1992; Czuppon *et al.*, 2009). Consistently, natural measurements of Xe mineral/basalt partitioning obtained by analysing parent and partially crystallised magmas (Batiza *et al.*, 1979), or coexisting magma and olivine crystals (Kaneoka *et al.*, 1983), show Xe compatibility with $D_{\text{crystal/basalt}}^{\text{Xe}}$ a few-fold above unity. Natural measurements must, nonetheless, be considered with caution (Carroll and Draper, 1994) due to potential magma degassing processes if minerals and melt did not re-equilibrate, and/or if crystals contain vapour inclusions (Kaneoka *et al.*, 1983), which as for experiments are difficult to interpret.

To circumvent these problems, we have recently developed a new method combining synchrotron X-ray fluorescence and diffraction techniques with large volume presses (Chen *et al.*, 2022). The method was first tested on crystal/felsic melt Xe partitioning, and Xe was found to be moderately incompatible to compatible, with a T -dependent behaviour (0.50 ± 0.20 for $D_{\text{plagioclase/melt}}^{\text{Xe}}$ at 1010 °C to 3.46 ± 0.25 for $D_{\text{jadeite/melt}}^{\text{Xe}}$ at 700 °C). This method is applied here to a Paris-Edinburgh press energy-dispersive set-up, which has the additional advantage of being optimised for the observation of diffuse X-ray scattering signal from melts.

Mineral/Melt Xenon Partitioning Measurements

We measured Xe partition coefficient between olivine and basalt, and between feldspar-clinopyroxene mix and basalt, in order to target Xe petrological behaviour in early Hadean contexts (*e.g.*, magma oceans in planetary embryos) and present day subduction zone contexts respectively. Xenon crystal/melt partition coefficients were measured by means of *in situ* synchrotron X-ray diffraction and X-ray fluorescence simultaneously collected on the same spectrum, using an energy-dispersive set-up and a Paris-Edinburgh press to generate high P - T conditions (Supplementary Information). The starting sample (Supplementary Information) is a synthetic glass relevant for lunar-like magma ocean at the stage of anorthite crystallisation, doped by high P - T synthesis with 0.05 wt. % Xe (Table S-1), *i.e.* well below Xe solubility in tholeiitic melt (Schmidt and Keppler, 2002) of 0.41 wt. % at 2 GPa to avoid supersaturation, while being high enough for the Xe fluorescence signal to be significantly above noise level. The starting sample is used either pure to mimic planetesimal magma ocean stages or mixed with 10 wt. % labradorite feldspar to get an analogue of high alumina basalts, relevant for present day subduction zone settings. At each targeted P , T was first raised until

recrystallisation, further raised until full remelting, and lowered until crystals grew in equilibrium with melt to ensure chemical equilibrium. The sample was then scanned perpendicularly to the X-ray beam in order to probe either pure melt or crystallised areas. For the olivine/basalt experiments, crystals were efficiently segregated from melt due to their density difference. This was not the case of the pyroxene-feldspar/basalt experiments for which it was not possible to probe only crystals, and the fraction of melt *vs.* crystals in crystal-rich areas was determined from the X-ray diffraction signal (Supplementary Information). Xenon content in pure melt or crystals was obtained from the Xe K_α fluorescence intensity signal in the fully molten sample pattern and the relevant pattern, either pure melt or crystals, at crystal-melt equilibrium (Supplementary Information). If a pure crystal pattern could not be obtained, Xe content in crystals from mixed crystals + melt pattern was obtained using mass-balance calculation from Xe K_α fluorescence intensity in crystal-rich area and local melt fraction obtained from X-ray diffraction signal (Eq. S-3).

For basaltic melt in equilibrium with feldspar and pyroxene, Xe was enriched in the melt as seen by the stronger Xe fluorescence signal (Fig. 1) and the derived partition coefficient (0.16 ± 0.06 ; Table 2). Crystal growth partly occurred upon quenching to room T (see microlithic texture of sample in Fig. S-3), hence it is difficult to have a proper estimate of feldspar *vs.* pyroxene at high P - T conditions, and oriented crystal growth prevents estimate from *in situ* energy-dispersive X-ray diffraction data. Nonetheless, due to the low global D value, Xe had to be incompatible with both pyroxene and feldspar at our experimental conditions, consistent with the reported T -dependence of D for clinopyroxene/felsic melt (Chen *et al.*, 2022). In contrast, Xe fluorescence signal was not detected in basalt coexisting with olivine (Fig. 1), which implies a lower limit of 80 for $D_{\text{olivine/basalt}}^{\text{Xe}}$ taking the noise oscillations as a maximum for Xe fluorescence signal in melt. Xenon X-ray fluorescence signal in olivine crystals is strongly affected by quenching T at high P , with a 2.9 factor decrease in intensity (Fig. 1a), indicating important—although incomplete—Xe exsolution back to room T , consistent with previous observation from *in situ* X-ray diffraction (Sanloup *et al.*, 2011).

To further constrain Xe concentration in melt coexisting with olivine, pieces of glass recovered from synchrotron experiments and an additional sample synthesised at similar P - T conditions (Supplementary Information) were analysed by noble gas mass spectrometry. From the additional sample, three fragments of glass and two fragments of mostly crystals could be separated, as olivine crystals had sedimented at the bottom of the capsule. Mass spectrometry analyses were done on bulk sample fragments fully melted by laser heating (Supplementary Information), revealing systematic Xe enrichment in olivine-rich fragments compared to glass. Olivine content in olivine-rich fragments is estimated at 83 ± 10 % from analyses of SEM images (Fig. S-3) using ImageJ software. Our reported values of $D_{\text{olivine/basalt}}^{\text{Xe}}$ 88(22)–302(46) (Table 2), exceed 80 as expected from the lack of Xe X-ray fluorescence signal in the melt. The lower range value was obtained using Xe content in crystals as measured from energy-dispersive X-ray data, and Xe content in glass as measured by mass spectrometry (Table S-2), which is near the 104(16) value obtained on the additional sample using mass spectrometry results only. The upper range corresponds to that value times the intensity ratio between crystals at high P - T and quenched crystals at high P (*i.e.* 2.9 GPa), to account for Xe exsolution from olivine crystals upon quenching.



Table 1 Compilation of literature datasets. Chemical compositions have been rounded at unity, given in wt. % for glass and mol % for gas. Analytical techniques: UV laser ablation (Heber et al., 2007), step heating (Hiyagon and Ozima, 1986; Broadhurst et al., 1992). Data discarded by authors due to contamination of crystals with bubbles are given in italics. For Hiyagon and Ozima (1986), glass composition is bulk sample starting composition. Data where lithium borate was added to lower melting T are not included, as borate modifies melt polymerisation which controls noble gases solubility. Error bars on D are given when available; * error equals D values.

Run (ref.)	Thermal treatment (duration @ °C)	P (GPa)	Glass composition (wt. %)	Crystal	Gas composition (mol %)	D ^{Xe} (error)
olivine-melt partitioning						
V (B1992)	18 d @ 1300	10 ⁻⁴	54SiO ₂ -15Al ₂ O ₃ -14MgO-17CaO	natural forsterite	5Ne-93Ar-1Kr-1Xe	90
RB587 (H2007)	1 h @ 1165 + 3 h @ 1045 + 6 h @ 990	0.1	61SiO ₂ -15Al ₂ O ₃ -23Na ₂ O	Mg ₂ SiO ₄	25He-23Ne-25Ar-22Kr-5Xe	3.9 × 10 ⁻³ -351
RB588 (H2007)	1 h @ 1165 + 3 h @ 1045 + 6 h @ 990	0.1	61SiO ₂ -16Al ₂ O ₃ -24Na ₂ O	Mg ₂ SiO ₄	25He-23Ne-25Ar-22Kr-5Xe	6 × 10 ⁻⁴ -148
RB589 (H2007)	1 h @ 1165 + 3 h @ 1045 + 6 h @ 990	0.1	58SiO ₂ -16Al ₂ O ₃ -23Na ₂ O	Mg ₂ SiO ₄	25He-23Ne-25Ar-22Kr-5Xe	0
BH-257 (H1986)	3 h 23 min @ 1370	10 ⁻⁴	48SiO ₂ -15Al ₂ O ₃ -10FeO-15MgO-7CaO-1Na ₂ O	(Mg,Fe) ₂ SiO ₄	11He-1Ne-88Ar-0.3Kr-0.1Xe + CO ₂ -H ₂ (0.5 to 1 ratio)	0.197(0.080)
BH-258 (H1986)	7 h 36 min @ 1370	10 ⁻⁴	48SiO ₂ -15Al ₂ O ₃ -10FeO-15MgO-7CaO-1Na ₂ O	(Mg,Fe) ₂ SiO ₄	11He-1Ne-88Ar-0.3Kr-0.1Xe + CO ₂ -H ₂ (0.5 to 1 ratio)	0.240(0.069)
BH-276 (H1986)	14 h 20 min @ 1350	10 ⁻⁴	48SiO ₂ -15Al ₂ O ₃ -10FeO-15MgO-7CaO-1Na ₂ O	(Mg,Fe) ₂ SiO ₄	115He-1Ne-88Ar-0.3Kr-0.1Xe + CO ₂ -H ₂ (0.5 to 1 ratio)	0.222(0.059)
HPP-1 (H1986)	10 min @ 1350	1.0	48SiO ₂ -15Al ₂ O ₃ -10FeO-15MgO-7CaO-1Na ₂ O	(Mg,Fe) ₂ SiO ₄	air adsorbed on powder	0.14(0.05)
HPP-3 (H1986)	4 h @ 1360	1.5	48SiO ₂ -15Al ₂ O ₃ -10FeO-15MgO-7CaO-1Na ₂ O	(Mg,Fe) ₂ SiO ₄	air adsorbed on powder	0.046(0.016)
pyroxene-melt partitioning						
IV (B1992)	18 d @ 1300	10 ⁻⁴	56SiO ₂ -11Al ₂ O ₃ -15MgO-19CaO	natural diopside	5Ne-93Ar-1Kr-1Xe	3.3
V (B1992)	7 d @ 1332	10 ⁻⁴	56SiO ₂ -11Al ₂ O ₃ -15MgO-19CaO	natural diopside	5Ne-93Ar-1Kr-1Xe	47
VI (B1992)	9 d @ 1332	10 ⁻⁴	56SiO ₂ -11Al ₂ O ₃ -15MgO-19CaO	natural diopside	5Ne-93Ar-1Kr-1Xe	4.0
RB586 (H2007)	0 h 30 min @ 1290 + 1 h @ 1272 + 6 h @ 1200	0.1	65SiO ₂ -14Al ₂ O ₃ -4MgO-8CaO	(Mg,Ca) ₂ SiO ₃	25He-23Ne-25Ar-22Kr-5Xe	0.2-70 × 10 ⁻³ *
HH355 (C2022)	2 h 30 min @ 950 + 3 h 45 min @ 700	1.6	67SiO ₂ -15Al ₂ O ₃ -1CaO-5Na ₂ O	jadete	Xe	3.58
HH357 (C2022)	4 h 20 min @ 850	1.7	60SiO ₂ -18Al ₂ O ₃ -1MgO-2CaO-8Na ₂ O	omphacite	Xe	2.54
feldspar-melt partitioning						
Cell5 (C2022)	6 h 30 min @ 1010	2.0	63SiO ₂ -24Al ₂ O ₃ -5CaO-8Na ₂ O	plagioclase	Xe	0.76



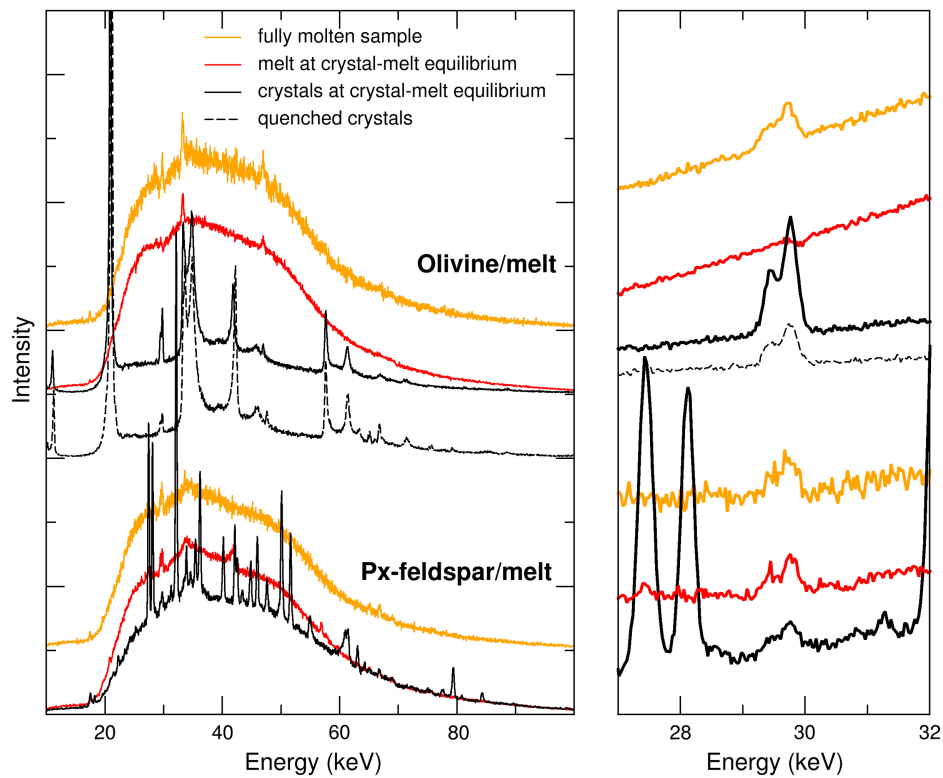


Figure 1 Energy-dispersive X-ray data sets. (Left) Full data sets collected at 10.031°. For each crystal/melt system, data collected at different T are vertically spaced for clarity: fully molten sample (orange), partially molten sample (red for molten zone, black for crystalline or crystal-rich zone), and quenched crystals (black dashed). Peaks at 33 keV and 47 keV are MgO diffraction peaks from the cell-assembly. (Right) Zoom on the Xe $K_{\alpha 1}$ and $K_{\alpha 2}$ fluorescence lines (29.4 keV and 29.7 keV). For olivine-melt experiments, data sets in the zoomed panel were collected at 4.0285° to avoid diffraction peaks from crystals overlapping with Xe fluorescence lines.

Table 2 Run conditions, coexisting phases, crystalline fraction in crystal-rich areas, Xe content in each phase (wt. %), and partitioning coefficients. Note that crystal and melt fractions do not apply to the whole sample but only to the volume probed by the X-ray beam. *Mass spectrometry measurement. Values in parentheses are errors on the last reported digits.

Run	P (GPa)	T (°C)	Phases	X_{crystals}	$[\text{Xe}]^{\text{crystals}}$ (wt. %)	$[\text{Xe}]^{\text{melt}}$ (wt. %)	$D_{\text{crystal/melt}}^{\text{Xe}}$
Cell2	2.0	1270	Ol + melt	100(5)	0.145(36)	$1.65(15) \times 10^{-3*}$	88(22)
PC265	1.3	1100	Ol + glass	83(10)	0.015(6)*	$1.5(8) \times 10^{-4*}$	104(16)–302(46)
Cell3	1.2	1120	An + Di + melt	34(4)	0.007(2)	0.044(9)	0.16(6)

Olivine Retains Xe in Magmatic Processes

Clinopyroxene-feldspar/melt data are broadly consistent with the T -trend reported for pyroxene/felsic melt under similar P (Chen *et al.*, 2022), and intermediate within the range of literature values at ambient or near ambient P (Broadhurst *et al.*, 1992; Heber *et al.*, 2007). Olivine/melt data (Fig. 2) confirm the compatible nature of Xe in olivine predicted from *ab initio* calculations (Crépeisson *et al.*, 2018), are similar to ambient P data from Broadhurst *et al.* (1992), and correspond to the higher range from Heber *et al.* (2007). The latter study may underestimate $D_{\text{olivine/basalt}}^{\text{Xe}}$ as 1) the melt was enriched in silica (Table 1) while Xe solubility increases by a factor of five, for instance, between a MORB and a haplogranite (Leroy *et al.*, 2019), and 2) crystals with bubbles were discarded on the basis of potential contamination while, at least, some of the bubbles are expected to form upon T -quenching, although this effect could be restricted to high P conditions.

Considered together, the present high P - T clinopyroxene-feldspar/melt and olivine/melt data are consistent with the natural reports of bulk peridotite/melt partitioning coefficient a few-fold above unity from oceanic island basalts (Batiza *et al.*, 1979; Kaneoka *et al.*, 1983). The P effect on $D_{\text{crystal/melt}}^{\text{Xe}}$ could not be investigated here due to the limited P - T stability field of the mineralogical parageneses investigated. It further cannot be inferred from the comparison of the present and previous datasets (Fig. S-5) as data are either at ambient or near ambient P (10^{-4} and 0.1 GPa) or in a restricted P -range between 1.0 and 2.0 GPa. Additionally, the only high P previous dataset for olivine/melt partitioning was obtained with only air adsorbed on samples as a source of noble gases (Hiyagon and Ozima, 1986). Pressure is, nonetheless, expected to be important as Xe retention in silicates is P -induced. Importantly, Xe retention does not extend to lower mantle silicates (Shcheka and Keppler, 2012), consistent with the plume source being depleted in Xe compared to MORB source (Parai, 2022).

As for any element, Xe crystal chemistry controls its partitioning behaviour, and its knowledge is key to understand its

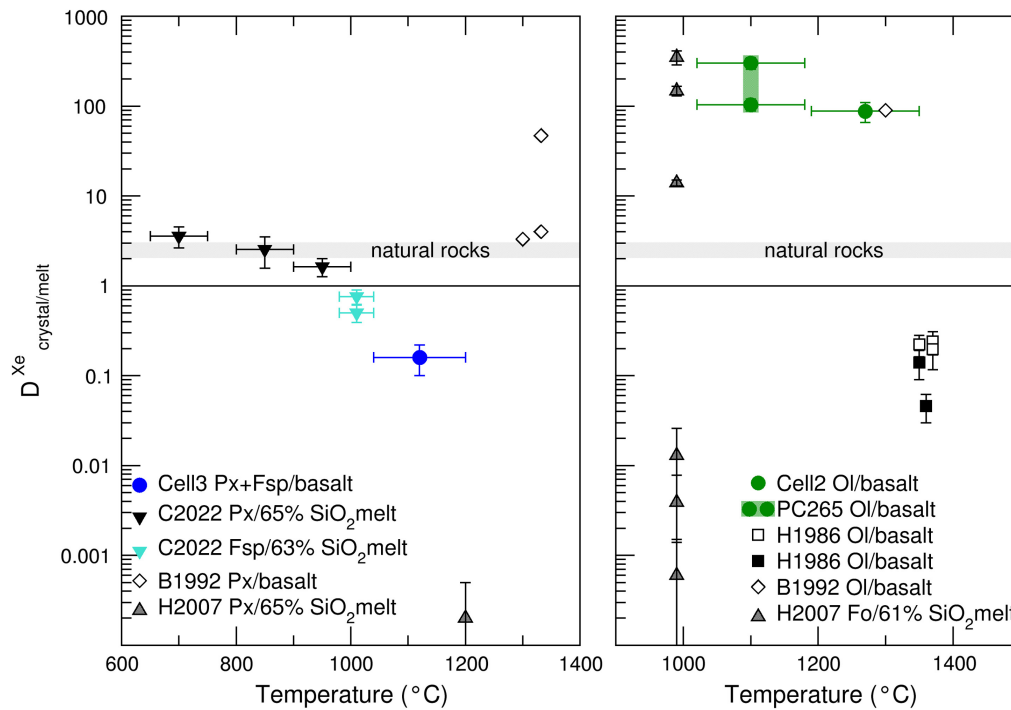


Figure 2 Summary of Xe crystal/melt partition coefficients. Data abbreviations: H1986, Hiyagon and Ozima (1986); B1992, Broadhurst *et al.* (1992); H2007, Heber *et al.* (2007); C2022, Chen *et al.* (2022); PC265, Cell2, and Cell3 are data from this study (Table 2). Empty symbols indicate room P data; filled symbols indicate data collected between 1.0 and 2.0 GPa (*cf.* Table 1). Note that Heber *et al.* (2007) discarded all values above unity due to the observation of gas bubbles in crystals, while at least some might form from exsolution upon quenching. Natural rocks dataset: residual rock/basalt partition coefficients from Batiza *et al.* (1979).

repartition in planetary envelopes. In magmas, noble gases enter the ring structure of silicate melts (Carroll and Stolper, 1993), eventually leading to oxidation in the case of Xe in compressed silica-rich melt (Leroy *et al.*, 2019). In minerals, the tightly bound nature of Xe leads to the suggestion that either interstitial sites or crystal vacancies are possible sites for the Xe atoms (Hiyagon and Ozima, 1986). Theoretical calculations have shown that Xe can chemically bond to oxygen in quartz (Probert, 2010; Crépisson *et al.*, 2019) and olivine (Crépisson *et al.*, 2018) under modest P by substituting a Si atom. This precludes the use of mineral/melt partitioning models considering Xe as a ‘zero’-charge species (Brooker *et al.*, 2003). The radius of chemically bonded Xe is, indeed, much smaller than that of inert Xe, with three oxygen atoms located at 2.0 Å in a planar configuration in olivine (Crépisson *et al.*, 2018), and two nearest oxygen atoms at 2.0 Å, and two further ones at 2.3 Å in quartz (Crépisson *et al.*, 2019). Xenon crystal chemistry has not been reported for pyroxene nor feldspar but for the latter it might be similar to that in quartz, both being tectosilicates. The contrasting $D_{\text{crystal/melt}}^{\text{Xe}}$ values between olivine/basalt and pyroxene-feldspar/basalt may relate to the different local environments of Xe in these minerals.

Over the course of Earth’s history, Xe should have been strongly retained in olivines crystallising from magma oceans, and in olivine-rich residues in present day mantle partial melting processes. Preferential release to the atmosphere is instead expected from high T (>1000 °C) crustal processes, turning to a moderate retention in pyroxenes and feldspars equilibrated with lower T melts such as evolved hydrous magmas in continental crust and arc contexts (Chen *et al.*, 2022). The petrological behaviour of Xe hence supports the trapping-at-depth scenario (Rzeplinski *et al.*, 2022), whereby a succession of collisions between pre-planetary embryos led to the depletion in terrestrial and Martian Xe light isotopes due to Xe trapping and oxidation in crystallising magma oceans, while over 99 % (Harper and

Jacobsen, 1996) of the initial budget was expelled from growing planetesimals by exsolution from melt at low P in convecting magma oceans followed by atmospheric losses on a few Myr timescale. The late veneer chondritic input to the atmosphere followed by partial-only Xe degassing concomitantly with the emplacement of the continental crust led to the rising Kr/Xe ratio and progressively heavier Xe observed in Archean atmospheric samples (Broadley *et al.*, 2022) until the late veneer got overprinted, while lighter Xe in the mantle (Peron and Moreira, 2018) would result from mixing mass fractionated Xe with subducted Archean atmospheric Xe. This Archean evolution is not observed on Mars where such events did not occur. That the retention of Xe differs in minerals while its impact on isotopic fractionating is similar (Rzeplinski *et al.*, 2022) implies that Xe elemental and isotopic evolution cannot be modelled by the same Rayleigh distillation law. Indeed, Rayleigh-predicted Kr/Xe ratios for the Archean atmosphere disagree (Broadley *et al.*, 2022), a mismatch attributed to variable Xe loss over time which is challenging to reconcile with a continuous isotopic evolution.

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Additional Information

Supplementary Information accompanies this letter at <https://www.geochemicalperspectivesletters.org/article2413>.



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