



Predominantly non-solar origin of nitrogen in lunar soils

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Abstract

Simultaneous static-mode mass spectrometric measurements of nitrogen, carbon, helium, neon, and argon, extracted from the same aliquot of sample by high-resolution stepped combustion, have been made for a suite of five lunar soils.

Noble gas isotope ratios show that the majority of noble gases are derived from a solar wind source; for example, at peak release temperatures of 500–600 °C, $^{21}\text{Ne}/^{22}\text{Ne} = 0.0313 \pm 0.0007$ to 0.0333 ± 0.0007 , and $^{20}\text{Ne}/^{22}\text{Ne} = 11.48 \pm 0.05$ to 12.43 ± 0.07 , with values at the lowest temperature steps less fractionated during implantation from, and therefore even closer to, solar values ($^{21}\text{Ne}/^{22}\text{Ne}_{\text{SW}} = 0.03361 \pm 0.00018$ and $^{20}\text{Ne}/^{22}\text{Ne}_{\text{SW}} = 14.001 \pm 0.042$ (Pepin et al., 2012)). Despite the co-release of nitrogen and solar wind argon, measured nitrogen isotopic signatures at each temperature step, whilst variable, are significantly more enriched in ^{15}N compared to the measured solar wind nitrogen value from the Genesis mission. Therefore, mixing between a ^{15}N -enriched non-solar planetary nitrogen source with solar wind nitrogen is required to explain the measured isotopic values from the stepped combustion analysis of lunar soils. Binary mixing calculations, made under different assumptions about the degree of loss of solar wind ^{36}Ar , reveal that the majority (up to 98%) of the nitrogen released is derived from a non-solar source. The range of modelled non-solar end-member nitrogen compositions required to satisfy the measured $\delta^{15}\text{N}$ values varies between samples and temperature steps from +5‰ up to +300‰, or between +87‰ and +160‰ for bulk samples. This range of modelled isotopic compositions for the non-solar source of nitrogen encompasses measured values for several different groups of carbonaceous chondrite, as well as IDPs.

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1. INTRODUCTION

For decades, there has been debate about the source(s) of nitrogen in lunar soils; since noble gases in lunar soils are predominantly solar, and nitrogen is both well correlated with these solar components, and likewise, co-located on soil grain surfaces (Wieler et al., 1999), a predominantly solar source for nitrogen was expected. However, stepped heating analyses of lunar regolith have

consistently revealed a similar release profile across all Apollo and Luna soils, with low temperature, ^{15}N -enriched nitrogen preceding a mid-temperature, ^{15}N -depleted component, which is then followed at the highest temperature steps by a second ^{15}N -enriched release (e.g. Brilliant et al., 1994; Assonov et al., 2002). This pattern of heavy-light-heavy (‘V-shaped’) nitrogen, with large-scale variations in nitrogen isotopic composition of around 300‰ (Becker and Clayton, 1975; Kerridge, 1975, 1993; Clayton and Thiemens, 1980; Frick et al., 1988; Brilliant et al., 1994; Wieler et al., 1999; Assonov et al., 2002; Furi et al., 2012), coupled with the observation that nitrogen isotopic variations correlate with $^{40}\text{Ar}/^{36}\text{Ar}$ ratios (which are taken as a proxy for soil antiquity, or when in

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the past a soil was exposed at the lunar surface) (Füri et al., 2012), led many to suggest that the composition of the solar wind must have changed over time, becoming progressively more enriched in ^{15}N (Kerridge, 1975; Becker and Clayton, 1975; Clayton and Thiemens, 1980).

In contrast, solar noble gases as measured in lunar soils display very minor variations in comparison to nitrogen isotopic compositions (e.g. Kerridge, 1989, 1993), inconsistent with a secular variation hypothesis for nitrogen. Further, $\text{N}/^{36}\text{Ar}$ ratios measured in lunar soils (Frick et al., 1988; Kerridge, 1993; Wieler et al., 1999; Assonov et al., 2002) are around ten times greater than the solar elemental abundance ratio of 37 (Anders and Grevesse, 1989). Whilst ^{15}N -depleted ('light') nitrogen is correlated with the release of solar wind D-depleted hydrogen from the outer rims of lunar soil grains, ^{15}N -enriched ('heavy') nitrogen correlates with the release of D-enriched hydrogen from a planetary source (Hashizume et al., 2000). Thus, the observed trend of higher average $\delta^{15}\text{N}$ values with decreasing soil antiquity may not be caused by a secular variation in solar wind composition, but instead may be recording a change in the relative contribution of a planetary nitrogen source to the lunar surface (Hashizume et al., 2002). Therefore, current evidence suggests that nitrogen in lunar soils represents mixing between a 'heavy' planetary nitrogen component and a solar wind component of 'light' composition (Geiss and Bochsler, 1982; Wieler et al., 1999; Hashizume et al., 2000, 2002; Assonov et al., 2002; Marty et al., 2003; Ozima et al., 2005; Füri et al., 2012). This has been supported by recent direct measurements of solar wind nitrogen implanted into the Genesis Solar Wind Concentrator target, which gave a modern-day $\delta^{15}\text{N}$ value for solar wind of $-407 \pm 7\text{‰}$ (Marty et al., 2011), comparable to the composition of protosolar nitrogen ($-383 \pm 8\text{‰}$ (Marty et al., 2011)), and to that of the Jovian atmosphere (Owen et al., 2001).

In light of this, Füri et al. (2012) calculated that most of the nitrogen released from Luna 24 soil grains is from a non-solar source with a planetary composition of between $+100\text{‰}$ and $+160\text{‰}$, assuming binary mixing with ^{15}N -depleted solar wind, with the highest proportional contribution from a non-solar source being found in recently exposed Apollo soils (with $^{40}\text{Ar}/^{36}\text{Ar}$ ratios ≤ 1.5).

Characterising the source(s) of volatiles being delivered to the lunar surface is essential for a broader consideration of how volatiles can be transported around the Solar System and added to the surfaces and atmospheres of other rocky Solar System bodies after initial planetary formation. Indeed, the volatile elements included in this study form the majority of most terrestrial planet atmospheres. Thus, to begin to understand planetary atmospheric evolution, any likely exogenous volatile source(s) must be identified and their contributions taken into account. Further, volatiles present at the surface of the Moon and other airless bodies, such as larger asteroids, may prove vital to the further human exploration of the Solar System, providing resources to support life beyond Earth. From this perspective, knowledge of which volatiles are present must be coupled with information about where they originated, thereby

enabling resource reservoir estimation and replenishment rate consideration.

To this end, a diverse set of Apollo lunar soils have here been analysed by stepped combustion, with the released gases simultaneously analysed for nitrogen, carbon, and noble gases (He, Ne, and Ar) in order to better constrain the contribution of non-solar nitrogen by taking into account multiple isotope systems, and their variations over discrete temperature increments.

2. SAMPLES

Five Apollo lunar soils, representing both extremely mature (e.g. $I_s/\text{FeO} = 94$) and immature (e.g. $I_s/\text{FeO} = 5.7$) samples, and covering a range of collection locations, from Apollo 12, 14, 15, 16, and 17, and including both shaded soils and samples collected at the surface of the regolith in unshaded locations, were chosen for stepped combustion analyses. Note that sample 12070 was analysed twice. (Table 1). Soil maturity is expressed as the ratio of observed ferromagnetic intensity of the sample (I_s) divided by its total Fe content (FeO); ferromagnetic intensity increases as a sample is exposed at the lunar surface *via* the production of non-charged Fe microparticles, termed nanophase Fe or np-Fe⁰, resulting from reduction or sputtering processes acting upon lunar regolith materials to remove oxygen from ferrous Fe. Thus, the larger the ratio of I_s/FeO , the more mature the soil.

3. METHODS

For stepped combustion analyses, samples were weighed out and transferred into 4 mm × 4 mm clean platinum foil buckets in a Class 100 cleanroom (Pt foil cleaning methods are as described in Abernethy et al., 2013). Tweezers and spatulas used to transfer the sample fines from their respective vials to the platinum buckets were cleaned before use, and wiped with acetone using lint-free cloths between usages with separate samples.

The 'Finesse' mass spectrometric instrument used in this study is a custom-built mass spectrometer system, consisting of three dedicated static-mode mass spectrometers (one for carbon, one for nitrogen and argon, and a quadrupole for helium and neon), all linked *via* high vacuum lines to a common sample inlet and gas clean-up sections, and combustion furnace (e.g. Mikhail et al., 2014, and references therein).

3.1. Stepped combustion procedure

Samples were combusted in oxygen, supplied from CuO, in a double-walled quartz-ceramic furnace for 30 min at each temperature step, followed by 15 min for oxygen desorption, before transfer of the released gases to the clean-up section. For Run 1 of soil 12070, twelve combustion steps were used, in 100 °C steps from 300 to 1400 °C. However, for Run 2 of soil 12070, fifteen combustion steps were employed to acquire higher-resolution data, heating in 100 °C steps from 200 to 700 °C, then in 50 °C steps from

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