

# Martian Atmospheric Noble Gas Measurements

Subjects: Others

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Martian Atmospheric Noble Gas Measurements refer to technologies to measure Martian atmospheric noble gases in situ by entry probes and in laboratory in Martian meteorites.

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## 1. Introduction

Noble gases, namely, helium, neon, argon, krypton, and xenon (He, Ne, Ar, Kr, and Xe), have been used as tools addressing a wide spectrum of scientific questions associated with terrestrial and extraterrestrial materials<sup>[1][2]</sup>. They are chemically inert; their abundances in the atmospheres of terrestrial planets are mainly controlled by the outgassing of solid materials throughout their geological history<sup>[3]</sup>. On one hand, the noble gas abundances and their isotopic ratios in planetary atmospheres can only be addressed by in situ measurements made by entry probes, because of the absence of radio signals in the planetary atmospheres<sup>[4]</sup>. Precise measurements are therefore necessary and represent a primary focus in planetary atmosphere exploration.

On the other hand, in addition to space missions, the history of Mars can be also unraveled from Martian meteorites. The Martian meteorites were first identified after the analysis of the Antarctic meteorite Elephant Moraine (EETA) 79001<sup>[5]</sup> (Figure 1a). This meteorite clearly shows the presence of glasses which probably formed by an impact on Mars that ejected EETA 79001. Similar features are observed in the Martian meteorite Tissint (Figure 1b). It is believed that these glasses encapsulated the composition of the Martian atmosphere at the time of the impact. Therefore, trapped noble gases (Ne, Ar, Kr, and Xe), as well as nitrogen N<sub>2</sub> and CO<sub>2</sub> abundances in black glasses separated from EETA 79001 showed a 1:1 correlation (on a log-log plot) with the volume abundances measured for the Martian atmosphere by the Viking mission (Figure 2)<sup>[5]</sup>.



**Figure 1.** (a) The Martian meteorite Elephant Moraine (EETA) 79001 and the presence of black glass which contains trapped Martian atmosphere. Credit from NASA. (b) A fragment of Tissint meteorite with shock-produced glass pockets (black), photo credit Meteor-Center (scale is represented by the 1-inch scale cube).

Both elemental abundances and isotopic ratios of a single noble gas are likely to be modified due to the escape of a primary atmosphere in the early history of the Solar System<sup>[6]</sup>. Physical processes leading gas to escape to space preferentially remove the lighter isotopes, thereby leaving the remaining atmosphere enriched in the heavier. These processes, therefore, lead to the progressive isotopic fractionation of planetary atmospheres, with heavy isotopic enrichments being consistent with atmospheric loss to space. Such fractionation processes in noble gas isotopic ratios and abundances were identified by, e.g., Viking or Curiosity. As an example, the Curiosity Ar isotopic measurements revealed fractionated  $^{36}\text{Ar}/^{38}\text{Ar}$  ratios<sup>[7]</sup> compared to Solar  $^{36}\text{Ar}/^{38}\text{Ar}$  ratio<sup>[2]</sup>, and assuming that Mars originally condensed

with this value. It, therefore, suggests a loss of Ar to space by pickup-ion sputtering, preferentially removing lighter atoms, i.e.,  $^{36}\text{Ar}$ <sup>[8]</sup>. In addition, Xe heavy isotopic enrichments observed in the Martian atmosphere by Viking and Curiosity can be explained by a preferential loss of light Xe isotopes to space by hydrodynamic escape<sup>[9][10][11]</sup>. Extreme UV exposure to the early Martian atmosphere by the young evolving Sun can account for the dissipation of primary planetary atmospheres around low-gravity planetary bodies<sup>[6][7][8][12][9][10][11]</sup>.

## 2. Noble Gases in Martian Atmosphere and In Martian Meteorites Measured In Situ or In the Laboratory: Technical Aspects

### 2.1. The Atmospheric Noble Gas Composition of Mars: The Latest Significant Missions

Over the last five decades, four space missions to Mars measured the composition of the Martian atmosphere, and particularly noble gas abundances and isotopic compositions: Viking, Curiosity, the Mars Atmosphere and Volatile Evolution (MAVEN), and the Mars Orbiter Mission (MOM). Here, we provide a technical summary of the noble gas measurements of the Martian atmosphere in situ and in laboratories on Earth.

#### 2.1.1. The Viking Project (1976–1982)

NASA's Viking Project was the first U.S. mission to land a spacecraft safely on the surface of Mars. The Viking 1 lander landed on the western slope of Chryse Planitia on 19 June 1976, while the Viking 2 lander touched down at Utopia Planitia on 3 September 1976, performing the very first geochemical exploration of Mars. Measurement of the Martian atmospheric gas composition was made by mass spectrometers onboard the Viking landers (Table 1) <sup>[13][14][15]</sup>. The abundances of CO<sub>2</sub>; H<sub>2</sub>O; N<sub>2</sub>; O<sub>2</sub>; the noble gases; as well as the isotopic ratios of hydrogen, carbon, oxygen, nitrogen, argon, krypton, and xenon were measured. The mass spectrometer onboard the Viking molecular analysis experiment had a high sensitivity, a high mass range, and a high resolution which facilitated a better characterization of minor atmospheric components. Such characteristics were suitable for the measurement of the noble gas abundances in the Martian atmosphere. Thus, two methods were adopted for the atmospheric analyses; the atmosphere was either directly inlet into the mass spectrometer for the measurement, or gas scrubbers were used to reduce the partial pressures of species such as CO and CO<sub>2</sub>, which otherwise could compromise the measurement. In the latter case, a mixture of Ag<sub>2</sub>O and LiOH was used as an absorber. After the elimination of 99% of the CO<sub>2</sub>, the concentrations of the noble gases were increased by successively repeating the ingestion and purification procedures. By these successive enrichments and by increasing the electron multiplier gain, Kr and Xe peaks were observed. A combination of 15 cycles measured with the electron multiplier after chemical purification lead to increase the signal-to-noise ratio by a factor of 5.3 over the nominal value<sup>[13][14][15]</sup>. A lower ionization energy was also used to reduce the interference caused by the double ionized species, particularly problematic for the Ne measurements (i.e.,  $^{40}\text{Ar}^{++}$  for  $^{20}\text{Ne}$  and  $^{44}\text{CO}_2^{++}$  for  $^{22}\text{Ne}$ ). By using the chemical scrubbers to reduce the CO<sub>2</sub> partial pressure, the observed contribution of interfering CO<sub>2</sub> at  $m/e = 22$  was less than one third.

**Table 1.** Composition of the Martian lower atmosphere<sup>[13]</sup>.

Gas	Abundance
CO <sub>2</sub>	95.32%
N <sub>2</sub>	2.7%
Ar	1.6%
O <sub>2</sub>	0.13%
CO	0.07%
H <sub>2</sub> O	0.03%
Ne	2.5 ppm

Kr	0.3 ppm
Xe	0.08 ppm
O <sub>3</sub>	0.03 ppm

### 2.1.2. The Mars Science Laboratory Mission (2012)

The Mars Science Laboratory (MSL) onboard the Curiosity rover landed in Gale Crater on 5 August 2012. MSL is equipped with a suite of scientific instruments including the Sample Analysis at Mars (SAM). The SAM suite onboard the Curiosity rover has three instruments dedicated to the study of gas and/or solid samples, including a quadrupole mass spectrometer (QMS), a tunable laser spectrometer (TLS), and a 6-column gas chromatograph (GC) system<sup>[16][17]</sup>. The combination of both QMS and TLS enables multiple and high-precision composition measurements throughout the mission.

Prior to any measurement, both QMS and TLS were evacuated using turbomolecular pumps. The Martian atmospheric samples were then inlet into a pre-evacuated (at 50 °C) manifold. Chemical scrubbers and zeolites helped reduce the partial pressure of active gases (e.g., CO<sub>2</sub> and H<sub>2</sub>O) by more than 95%, which would otherwise compromise any measurements. The post-purification gas consists of a gas mixture enriched in N<sub>2</sub>, Ar, Kr, and Xe. The gas is then channeled through a cooled hydrocarbon trap which traps Xe, whereas other gases passed through.

The ingestion and purification of gases are repeated several times until reaching a noble gas density sufficient enough to achieve high signal over background ratios. Background measurements are performed when the QMS is opened to the evacuated manifold. The Martian atmosphere is subsequently inlet for ~30 s to a portion of the manifold. A small fraction of this gas is finally inlet into the QMS in a dynamic sampling mode.

### 2.1.3. MAVEN (2014)

The main aim of the Mars Atmosphere and Volatile Evolution (MAVEN) mission was to study the Martian atmosphere, the geological evolution, and the potential habitability of the planet<sup>[8][18]</sup>. The MAVEN mission objectives particularly aimed at evaluating the amount of atmospheric gas which has been lost to space over geological time through, among others, the ratio of <sup>38</sup>Ar/<sup>36</sup>Ar. Argon can be removed from the Martian atmosphere by only solar-wind-induced sputtering (pick-up ion sputtering)<sup>[8]</sup>: the higher scale height of <sup>36</sup>Ar relative to <sup>38</sup>Ar implies that the lighter isotope is more abundant in the upper atmosphere, thus proportionally more <sup>36</sup>Ar is removed by sputtering. Therefore, measuring precisely the <sup>38</sup>Ar/<sup>36</sup>Ar ratio is of great significance as it can give information to determine the sputtering loss of other atoms and molecules.

The composition, structure, and variability of the upper atmosphere of Mars were characterized by the Neutral Gas and Ion Mass Spectrometer (NGIMS) onboard the MAVEN mission<sup>[18]</sup>. The NGIMS instrument, a direct heritage from e.g., Galileo GPMS or Pioneer Venus ONMS, consists of a quadrupole mass spectrometer with open and closed sources, for neutral and ions, and for non-reactive neutrals (i.e., Ar, He, CO<sub>2</sub>, N<sub>2</sub>, and CO), respectively. Its design enables neutral masses ranging from ~2 amu to ~150 amu to be measured. The incoming signal is reduced by a factor of ~10 or ~100, enabling the QMS to detect densities of gas at much higher concentrations than in normal operating mode. This is particularly suitable for the measurement of high concentrations of Ar, CO<sub>2</sub>, or CO, and O in the Martian atmosphere, which would normally saturate the detector at nominal attenuation. See Mahaffy et al., 2015 for further details about the NGIMS instrument and characteristics<sup>[18]</sup>.

### 2.1.4. MENCA Onboard MOM (2014)

India's first mission to Mars, Mars Orbiter Mission (MOM), was inserted into the Martian orbit on 24 September 2014. The Mars Exospheric Neutral Composition Analyzer (MENCA) is one of the science instruments onboard the MOM mission; it was equipped with a quadrupole mass spectrometer which is capable of total pressure measurements, thanks to a Bayard Alpert gauge<sup>[19][20]</sup>. Similar to the NGIMS in MAVEN mission, the main objective of MENCA was to understand the escape of the Martian atmosphere over geological time; in addition, <sup>40</sup>Ar being one of the minor constituents of the Martian atmosphere, its variability has direct implications regarding atmospheric evolution processes on Mars. The MENCA was equipped with two detectors, a Faraday cup, and a channel electron multiplier, in a dynamic range of 10<sup>10</sup>. The quadrupole mass spectrometer was therefore also sensitive enough to measure the tenuous exosphere of Mars. MENCA measured the relative abundances of neutral constituents in the mass range of 1 to 300 atomic mass units<sup>[19][20]</sup>.

The goals and achievements of these missions to Mars, in terms of noble gas elemental abundance and isotopic ratio measurements, are summarized in Table 2.

**Table 2.** List, goals, and achievements of the latest missions to Mars for the measurement of noble gas elemental abundances and isotopic ratios.

Mission	Goals	Payloads	Achievements
<i>Viking</i>	<ul style="list-style-type: none"> <li>–Detect and identify organic compounds</li> <li>–Determine the composition of the lower atmosphere</li> </ul>	Mass spectrometer coupled with gas chromatograph	–Atmospheric elemental abundances, including noble gases
<i>Curiosity</i>	<ul style="list-style-type: none"> <li>–Understand the potential of the present or past Martian environments to support life</li> <li>–Chemical and isotopic composition of the atmosphere</li> </ul>	Sample Analysis at Mars (SAM): quadrupole mass spectrometer, tunable laser spectrometer, gas chromatograph	–Measurements of all of the stable isotopes of the heavy noble gases in the Martian atmosphere
MAVEN	<ul style="list-style-type: none"> <li>–Study the Martian atmosphere, and the geological evolution and potential habitability of the planet</li> <li>–Evaluate the amount of atmospheric gas which has been lost to space over geological time</li> </ul>	Neutral Gas and Ion Mass Spectrometer (NGIMS), a quadrupole mass spectrometer	<ul style="list-style-type: none"> <li>–Characterization of the Martian upper atmospheric structure</li> <li>–Fractionation of argon</li> </ul>
MOM	Understand the escape of the Martian atmosphere over geological time	Mars Exospheric Neutral Composition Analyser (MENCA), a quadrupole mass spectrometer	<ul style="list-style-type: none"> <li>–Exospheric composition of Mars</li> <li>–Altitude profiles of <math>^{40}\text{Ar}</math> in the Martian exosphere</li> </ul>

## 2.2. Noble Gas Mass Spectrometry Analyses in Laboratory

A noble gas mass spectrometer is mostly used to measure noble gases (from He to Xe). Noble gas mass spectrometry determines accurate measurements of noble gas concentrations and isotopic ratios of various terrestrial and extraterrestrial materials (cf. introduction and references therein). In addition, the study of Martian meteorites provides even higher precision in the noble gas elemental abundances and isotopic ratios compared to spacecraft.

Noble gases are extracted from meteorite samples through heating (pyrolysis or combustion) in a single step at a temperature high enough to release all noble gases at once or by incremental heating in discrete temperature steps<sup>[21]</sup>. Based on the science question and the type of meteorite, one can choose the method of heating. General heating methods involve resistance furnace heating and laser ablation. In some special types of meteorites, gas extraction can be done through vacuum crushing and combustion coupled with pyrolysis heating. Here, we will briefly describe and explain the state-of-the-art technical aspects of noble gas mass spectrometry on meteorite samples. In some cases, noble gases and nitrogen have been measured simultaneously. In addition, the Open University, UK, is, so far, the unique facility capable of measuring carbon, nitrogen, and noble gas isotopes simultaneously.

### 2.2.1. Sample Preparation

Prior to any noble gas measurement of meteorite specimens, samples are first cleaned with ethanol in an ultrasonic bath, dried, weighed, and then loaded into a sample chamber connected to a noble gas extraction and purification line. Typically, after any sample loading, the whole system (sample chamber volume and extraction and purification line) is

baked and evacuated for ~1–3 days at ~120 °C in order to remove adsorbed atmospheric gases. In the case of Martian meteorites, the sample mass can range between a few mg to hundreds of mg, depending upon the method of gas extraction.

The best samples to search for Martian atmospheric signatures are shock-induced glasses separated from, e.g., the shergottites. For example, signatures of Martian atmosphere, based on N<sub>2</sub>, Ar, and Xe isotopes, were determined in separated millimeter-sized pockets of shock-melted glass in the meteorite Zagami<sup>[22]</sup>. In addition, noble gases were measured in glass samples from the latest Martian meteorite fall, Tissint<sup>[23][24]</sup>. In all of these studies, glass samples were prepared through hand picking. When a complete separation is not possible, glass separates with some matrix minerals were also used for noble gas measurements.

### **2.2.2. Pyrolysis: Total Fusion or Stepwise Heating**

As mentioned above, pyrolysis is done either in a single step or in multi-steps, based on the type of sample and the scientific objectives. If the sample size is restricted to a few mg (e.g., less than 10 mg), laser heating can be adopted for total fusion. However, in the cases of relatively large samples (tens of mg to a few grams), total extraction can be achieved by heating in a crucible made of a metal with high melting temperature (Ta, Mo, or W)<sup>[21]</sup>. Crucibles and samples are heated by radiofrequency induction, by applying a current through a resistance, or by electron bombardment. Single grain analysis is usually done by laser ablation. In addition, lower instrumental blanks used in furnace heating are an advantage for laser heating. In some cases, noble gases are extracted in different steps, due to some specific scientific objectives such as (1) the selective identification of a particular component of interest, (2) to decipher spatial distribution of noble gas components in mineral grains, (3) to infer noble gas diffusion coefficients, and (4) to avoid terrestrial contamination in some meteorites<sup>[21]</sup>. After the samples are loaded in the preparation system, and before sample measurements, calibrations are regularly performed using standard gases of known noble gas isotopic composition or air. Instrumental mass discriminations are as well derived from calibration measurements. Instrumental blanks are repeatedly measured, usually before and after each sample, using the same extraction procedure as for the samples. Noble gases are extracted by heating the samples in either a single extraction step or in stepwise heating for a duration of several minutes to ~30 min. The released gases are subsequently cleaned from all reactive gases which would compromise the measurements such as CO<sub>2</sub>, H<sub>2</sub>O, or hydrocarbons, etc. using a series of getters, operating at various temperatures<sup>[25][26]</sup>.

### **2.2.3. Combustion**

In order to measure noble gases released during progressive outgassing of the mineral host phases, differential combustion in molecular oxygen is usually adopted as an extraction method for Martian meteorites and carbonaceous chondrites<sup>[22][27]</sup>. Stepped combustion helps to resolve different carbon-bearing components on the basis of thermal stability under oxygen. This method is usually adopted when noble gases are measured simultaneously with C and N isotopes. As volatile compounds such as atmospheric adsorbed gases or organic species are released at low temperature (~300 °C), combustion at a low temperature helps to identify the composition of trapped noble gases. For example, in such a study, N, Ar, and Xe were released from separated glass samples from the Zagami meteorite, through combustion in pure O<sub>2</sub> at a pressure of 5 torrs (up to 800 °C), followed by pyrolysis (up to 1500 °C)<sup>[22]</sup>.

### **2.2.4. Crushing Technique**

In order to separate trapped and cosmogenic noble gas components, vacuum crushing is an efficient method. It was demonstrated for example in Martian meteorites<sup>[28]</sup>, especially to have access to the trapped gases implanted from the Martian atmosphere, which usually reside in the glass vesicles of the meteorites.

However, prolonged crushing might induce heating, which may also release matrix sited cosmogenic He as observed in some previous studies<sup>[29]</sup>. Nevertheless, <sup>3</sup>He is only lost during hot conditions, with temperatures >300 °C<sup>[29]</sup>. Noble gases released using this technique appeared to be quite different from the Martian atmospheric signature. In particular, Kr and Xe isotopic signatures are indistinguishable from terrestrial and/or Martian atmosphere<sup>[30]</sup>. Vacuum crushing is extensively used in, e.g., terrestrial mantle materials when searching for noble gases trapped in fluid inclusions, where usually a few grams of sample are crushed at a pressure of 1500–2000 psi (1 psi = 51.715 torrs)<sup>[25][31]</sup>. However, in the cases of Martian meteorite samples, the sample masses are lower—usually 100–200 mg<sup>[32]</sup>. Note that after crushing, the samples can be recovered and subsequently loaded into a furnace or in a laser chamber for further stepwise heating combustion or pyrolysis<sup>[32]</sup>.

### **2.2.5. Separation and Purification**

After gas extraction (e.g., by pyrolysis, combustion, or crushing), all active gases have to be removed before introducing noble gases into the mass spectrometer. The usual method for removing active gases requires getters, made of chemically reactive metals or metal alloys such as ZrAl or ZrTi<sup>[21]</sup>. In order to have a maximum surface area, these materials are often porous.

Purification procedures may vary slightly between laboratories depending on the types of materials used in various parts of the preparation system. From the purified gas mixture, the He-Ne fraction is separated from the Ar-Kr-Xe fraction using activated charcoal held at the temperature of liquid nitrogen (LN<sub>2</sub>) for 20 min. Subsequently, the Ne fraction is separated from He by using a cryogenic cold trap held at 35 K for ~10 min<sup>[21]</sup>. Generally, the trapping medium is charcoal, but note that the trapping temperature may vary depending on the medium. After having trapped Ne, a fraction of the He gas is then inlet into the mass spectrometer for measurement. After He measurements, the Ne fraction is released at 80 K for ~30 min<sup>[21]</sup>, and inlet into the mass spectrometer for subsequent measurement. During Ne measurements, the remaining background gases, especially <sup>40</sup>Ar<sup>++</sup> and CO<sub>2</sub><sup>++</sup> interfering at  $m/e = 20$  and  $m/e = 22$ , respectively, are further reduced using a charcoal trap held at LN<sub>2</sub> temperature, adjacent to the mass spectrometer source. At the same time, all heavy noble gases are released (here a mixture of Ar, Kr, and Xe) from a cold trap with active charcoal at ~420–480 K. The Ar fraction is then usually separated from the Kr and Xe fractions using a cold trap (charcoal or in some cases a cryogenic trap) at a temperature on the sorption-desorption characteristics of the considered trap. It varies between 150 K and 155 K for cold trap with active charcoal<sup>[32]</sup> and around 85 K for nude cryogenic traps<sup>[33]</sup>, and by doing so, only Kr and Xe remain trapped; all of the Ar will remain as a gas and can be further trapped and subsequently measured. The Kr fraction can then be separated from the Xe fraction at the given temperature (~198 K for cold trap with active charcoal, and around ~95–100 K for nude cryogenic traps), and subsequently inlet into the mass spectrometer. Finally, after Kr measurement, the remaining Xe is released at ~420–480 K for ~20 min and is further inlet into the mass spectrometer. However, in most cases, (Ar) Kr and Xe are not separated and measured consecutively, in order to avoid some possible isotopic fractionation processes as well as separation loss<sup>[34][35]</sup>. After the first measurement, second extractions at slightly higher temperatures are systematically performed in order to ensure good first extraction rates. Additional extractions are performed as long as the gas amount from the successive extractions remains significant.

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