Geochemical Perspectives Letters

Ageing of organic materials at the surface of Mars: A Raman study aboard Perseverance

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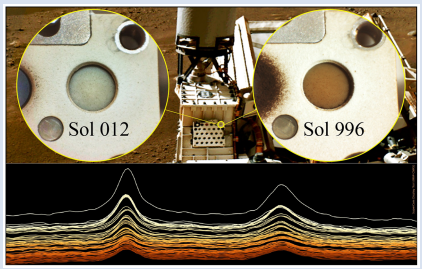
Abstract



The Perseverance rover is exploring Jezero crater on Mars, one of its goals being to collect samples to be returned to Earth to search for organic remains of ancient Martian life. However, the organic content of these rocks has likely suffered from the radiation environment on the surface of Mars to an extent yet to be quantified. For the first time, a 1000 sols long ageing experiment was conducted at the surface of Mars, *i.e.* under actual Martian conditions, relying on the 100 % organic Ertalyte target carried by Perseverance. White at landing, the Ertalyte target has turned brown with time, while its Raman signal changed, with a modification of the background (its maximum has shifted from 1500 to 2000 cm^{-1}) and a reduction of the contribution of the Raman signal of Ertalyte (by a factor of 5 over the first 500 sols). Given the intrinsic resistance of the Ertalyte to UV exposure, which is not anticipated for most Martian organic materials, these results suggest that exposure at the surface of Mars will make the detection of Martian organic molecules challenging.

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Letter

The Perseverance rover was sent to Jezero crater on Mars to explore an astrobiologically relevant site, characterise its biosignature preservation potential, and collect rock samples to be returned to Earth (Farley *et al.*, 2020; Haltigin *et al.*, 2022). Determining the nature and the origin (biogenic or abiotic) of the organic compounds possibly trapped in these rocks will be required to assess whether or not there has been life on Mars (Viennet *et al.*, 2019; Bosak *et al.*, 2021; McMahon and Cosmidis, 2021; Ansari, 2023; Criouet *et al.*, 2023). Yet, the thin CO_2 atmosphere of Mars and the lack of a magnetic field expose the Martian surface to high doses of electromagnetic (UV and γ) and particle (protons, neutrons and high Z atoms) radiation (Patel *et al.*, 2002; Hassler *et al.*, 2014), which may chemically and structurally alter organic compounds. With a present day Martian UV flux similar to that of early Earth (Cockell *et al.*, 2000), a number of studies have investigated the impact of UV exposure, mainly relying on gas chromatography mass spectrometry (GCMS) and/or Fourier transform infrared spectroscopy (FTIR) to monitor the evolution of organic compounds (*e.g.*, Fornaro *et al.*, 2018). These studies have shown that even

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short exposure to UV (corresponding to less than hundreds of sols on Mars) leads to the photodecomposition of organic compounds, such as amino acids (ten Kate *et al.*, 2005), carboxylic acids (Stalport *et al.*, 2009), nucleobases (Fornaro *et al.*, 2013), urea (Poch *et al.*, 2014) or PAHs (Stalport *et al.*, 2019). Such photodecomposition is generally interpreted as the result of UV-driven, Fenton-like reactions producing radicals, as predicted by Benner *et al.* (2000).

Raman spectroscopy has recently become available on Mars with Perseverance carrying both the SuperCam time-resolved Raman (Maurice *et al.*, 2021; Wiens *et al.*, 2021) and the deep UV SHERLOC Raman (Bhartia *et al.*, 2021) spectrometers. This has motivated specific experimental studies to document the effect of UV radiation on the Raman signals of organics and minerals (Megevand *et al.*, 2021; Fox *et al.*, 2023; Clavé *et al.*, 2024; Royer *et al.*, 2024). For instance, combining time-resolved Raman, FTIR and ESR (electron spin resonance) spectroscopies, Megevand *et al.* (2021) showed that exposure to UV leads to the increase of structural disorder and to the creation of electronic defects and/or radicals within the molecular structure of S-rich organic compounds, such as cystine. Yet, such experimental studies may not have completely mimicked Martian conditions. In fact, results of experiments conducted both on Mars and in the laboratory on Earth showed that the Raman signal of the Apatite (TAPAG) SuperCam calibration target changed faster in the laboratory than on Mars when exposed to equivalent doses of UV (Clavé *et al.*, 2024). This is possibly a result of relaxation periods absent in the laboratory but existing on Mars (periods of shades, day/night cycles), and/or from differences in pressure, temperature, humidity or radiation conditions (Clavé *et al.*, 2024). A similar study conducted on organic materials exposed to actual Martian conditions, *i.e.* on board Perseverance, thus appears necessary to properly determine what would/should be expected to be found in Martian rocks using Raman spectroscopy.

One of the onboard SuperCam calibration targets (Manrique *et al.*, 2020; Cousin *et al.*, 2022) is a 100 % organic target made of polyethylene terephthalate (PET - $(C_{10}H_8O_4)_n$, a.k.a. the Ertalyte® target; Fig. 1), which has allowed conducting a 1000 sols long ageing experiment under actual Martian conditions. This target is made of a thermoplastic polymer intrinsically resistant to UV and exhibiting a chemical structure including aromatic, aliphatic and ester/carboxylic functional groups (Fig. 1). Such molecular groups are expected to be found in biogenic organic compounds trapped in ancient rocks (Bernard *et al.*, 2021). The Ertalyte target carried by Perseverance is 100 % crystalline and was machined at IMPMC in Paris out of an Ertalyte® rod provided by Mitsubishi Chemical Advanced Materials. The surface roughness was reduced through gentle polishing, making it the same size as the other SuperCam targets (1 cm diameter, 5 mm thickness). The Raman signal of the Ertalyte target exhibits a number of intense Raman bands (Fig. 1), at 633 cm^{-1} (ring mode), 859 cm^{-1} (ring C-C and C(O)-O), 998 cm^{-1} (C-C), 1095 cm^{-1} (ring C-C, C(O)-O and C-C), 1180 cm^{-1} (C-H and C-C), 1288 cm^{-1} (C(O)-O), 1460 cm^{-1} (C-H), 1613 cm^{-1} (ring mode), 1727 cm^{-1} (C=O), 2966 cm^{-1} (C-H) and 3083 cm^{-1} (O-H) (Manrique *et al.*, 2020; Cousin *et al.*, 2022).

On Mars, the Ertalyte target has been systematically imaged using the SuperCam Remote Micro-Imager (RMI), and analysed using the SuperCam Raman every 40 to 70 sols from sol 26 (after the landing of Perseverance on Feb 18th, 2021) up to sol 996, providing a consistent set of data over almost 1000 sols. Briefly, the SuperCam Raman relies on a pulsed Nd:YAG laser and a frequency doubler to produce a 532 nm pulsed green beam collimated towards the sample. A repetition rate of 10 Hz is used with 9 mJ per pulse deposited on a ~1 cm diameter spot, yielding an irradiance at the sample surface ranging from 1.10^{10} to 5.10^{10} $W.m^{-2}$, *i.e.* well below the threshold for laser-induced damage (Fau *et al.*, 2019). The transmission spectrometer has a compact design including diffraction gratings, an intensifier, and relay optics that focus light onto a CCD, yielding a spectral resolution of about 10–12 cm^{-1} for the Raman window, ranging from 165 to 7065 cm^{-1}. Each spectrum shown in the present study corresponds to the accumulation of 100 laser shots collected using a gate of 100 ns, with the laser powered at 110 A and an intensifier gain of 3200. Of note, given the variability of the total signal collected for each shot, spectra have been normalised to the total signal received on the spectrometers (Figs. S-1 and S-2). SuperCam VIS spectra of the Ertalyte target were also collected every 80 to 100 sols (Fig. S-3). In addition, the Raman spectrum of the sample holder paint has been measured on the same sols from sol 450 to serve as a reference insensitive to UV radiation (the sample holder paint has not degraded over time; Fig. S-4). The strong variability in the total signal collected on the sample holder paint from one sol to another is instrumental as it is directly correlated to the temperature of the primary mirror of the SuperCam telescope, which is directly related to the temperature of the laser (Fig. S-5).

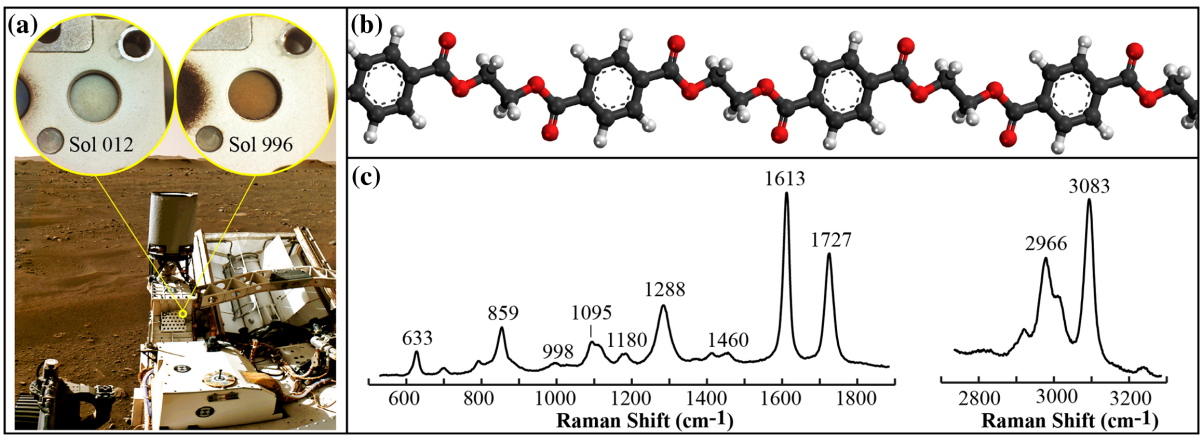

Figure 1 **(a)** Image of the Perseverance rover showing the location of the Ertalyte target and RMI images of the Ertalyte target after 12 and 996 sols since the landing of Perseverance on Mars. **(b)** Schematic molecular structure of the Ertalyte polymer. **(c)** Raman spectrum of the Ertalyte target collected using the customised time-resolved Raman spectrometer built by the Cellule Projet @ IMPMC.

In parallel, irradiation experiments were conducted in the laboratory on Earth, in a dedicated Martian chamber and using a customised time-resolved Raman spectrometer, both built by the Cellule Projet @ IMPMC (Megevand *et al.*, 2021; Clavé *et al.*, 2024; Royer *et al.*, 2024). In this chamber, a spare Ertalyte target was exposed to UV radiation at 0 °C under a primary vacuum (<1 mbar). The UV flux was produced by a 150 W arc lamp equipped with a high pressure Xenon bulb (UXL-150SP - LOT-ORIEL), delivering UV with a pattern similar to that of the Martian surface radiation spectrum (Royer *et al.*, 2024), a DUV Grade Fused Silica window allowing for rejection of most of the IR radiation (down to 2.89 µm). Time-resolved Raman spectra of the pristine and irradiated Ertalyte target were measured directly in the Martian chamber (from 200 to 2000 cm^{-1} with a spectral resolution of about 10–12 cm^{-1}) after 100, 370, 1370 and 2640 minutes. Each spectrum corresponds to the accumulation of 10^6 laser shots collected using a 2 ns ICCD gate centred on the 1.2 ns laser pulse. The irradiance of the laser, distributed over a spot of ~6 mm in diameter at the surface of the Ertalyte target, did not exceed 10^{10} $W.m^{-2}$, thereby preventing laser-induced damage (Fau *et al.*, 2019). To describe more quantitatively the evolution of the Raman signals of the Ertalyte irradiated on Mars and in the laboratory on Earth, the main Raman bands (at 1613 and 1727 cm^{-1}) were fitted (after normalisation to the total signal) with pure Gaussian functions (with the background classically modelled by a linear function in the range 1550–1880 cm^{-1}) using the SuperCam Display Spectra 4.0 developed by the SuperCam team at IRAP.

While white at the time of landing, the Ertalyte target has turned brown with time as revealed by the RMI images (Fig. 1). White balancing the RMIs using the sample holder paint, a clear browning of the Ertalyte target with time can be observed (Fig. 2), consistent with the evolution of its VIS signal (Fig. S-3). The Raman spectrum of the Ertalyte target has also changed with time (Fig. 2), with a modification of the shape of the background and a shift of its maximum from 1500 to 2000 cm^{-1}. No new peak was detected, suggesting that no new Raman active compounds were produced, or were in quantities insufficient to be detectable. Of note, the Raman spectrum of the Ertalyte target still exhibits all the features observed in the spectrum of pristine Ertalyte, even after 996 sols on Mars. This indicates that a certain volume of pristine Ertalyte is still contributing to the signal. Still, the contribution of the Raman signal to the total signal has decreased by a factor of 5 over the first 500 sols (Fig. 2). At first sight, the evolution of the area of the band at 1613 cm^{-1} (normalised to the total signal) follows a first order logarithmic law with time (Fig. 3). Yet, a closer look reveals that three stages of evolution can be identified: an early stage up to sol 280, a second stage from sol 280 to sol 480 and a last stage starting from sol 480 (Fig. 3).

From sol 26 to sol 280, the total signal (dominated by the background) has been increasing while the contribution of the Raman signal has been decreasing (as shown by the evolution of the area of the band at 1613 cm^{-1} normalised to the total signal; Figs. 3 and S-6). This evolution is synchronous with the yellowing/browning of the Ertalyte target, which has likely increased the opacity of the target and limited the penetration of the laser, thereby reducing the contribution of the pristine Ertalyte to the total signal. A similar evolution of the Raman signal of the Ertalyte target is observed for irradiation experiments conducted in the lab, *i.e.* a decrease of the contribution of the Raman signal to the total signal with increasing duration of exposure to UV (Fig. 4). This suggests that the radiative environment on the surface of Mars can be simulated in the laboratory using UV only. Yet, kinetics are very different; the contribution of the Raman signal to the total signal has decreased by a factor of 5 over 500 sols on Mars *versus* over only 1270 minutes in the lab, corresponding to only a few sols assuming day/night cycles. The causes of such discrepancies may reside in the atmospheric pressure and temperature conditions, higher temperature enhancing the effect of exposure to UV (François-Heude *et al.*, 2014; Gogotov and Barazov, 2014), or in some sort of relaxation occurring during periods of non-exposure to UV that remains to be investigated (Clavé *et al.*, 2024).

Mechanistically, such evolutions of the colour and the Raman signal of the Ertalyte target can be explained by the UV-induced production of electronic defects and/or radicals (Grossetête *et al.*, 2000; Sang *et al.*, 2020; Jamalzadeh and Sobkowicz, 2022; Rostampour *et al.*, 2024). In fact, UV exposure of polymers has been shown to induce Norrish type I and type II reactions producing radicals via chain scission (Grossetête *et al.*, 2000; Sang *et al.*, 2020; Jamalzadeh and Sobkowicz, 2022; Rostampour *et al.*, 2024). Norrish type I reactions mainly involve

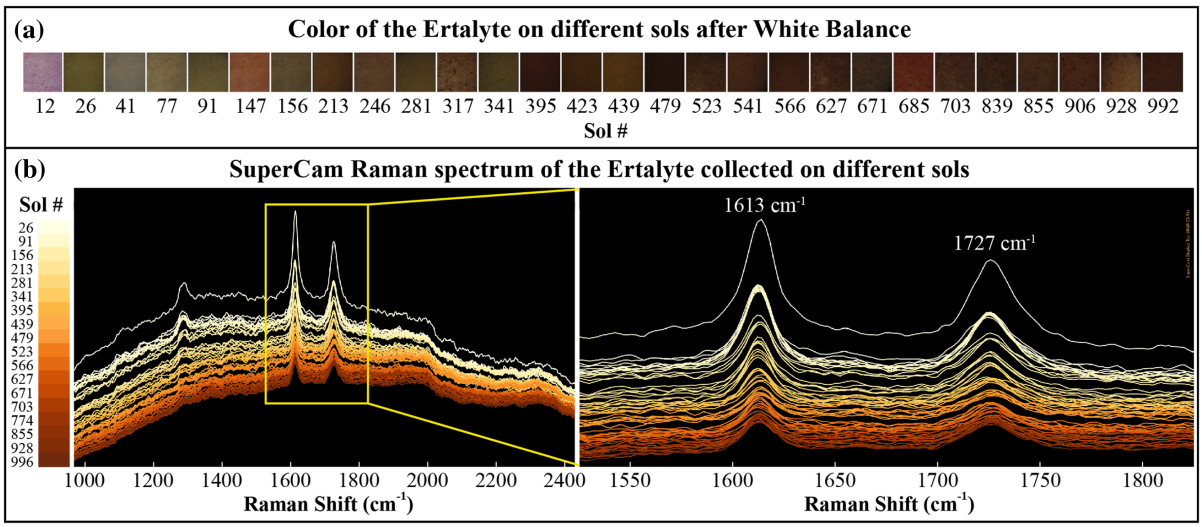

Figure 2 **(a)** Evolution of the color of the Ertalyte target as a function of the time spent on Mars (sol number). **(b)** SuperCam Raman spectra (normalised to the total signal) collected on the Ertalyte target every 40 to 70 sols since the landing of Perseverance. The colour code mimics the true colour evolution of the Ertalyte target from sol 026 to sol 996.

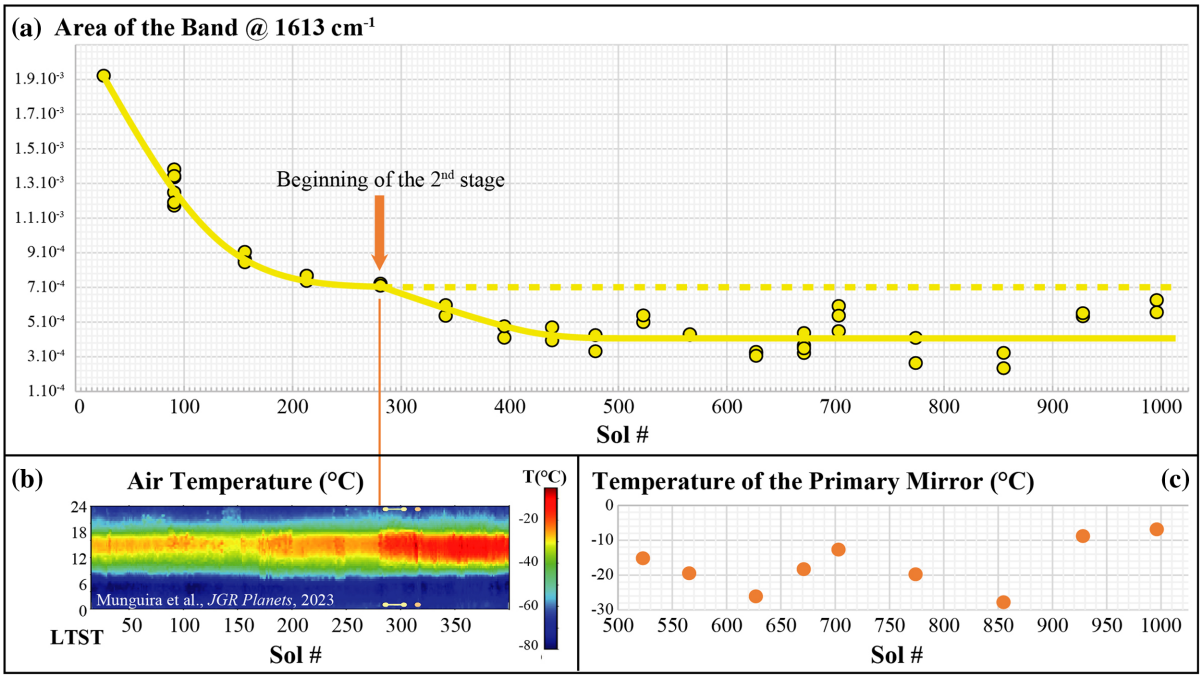

Figure 3 **(a)** Evolution of the area of the band at 1613 cm^{-1} as a function of the time spent on Mars (sol number). **(b)** Air temperature measured at 1.45 m in Jezero crater from sol 020 to sol 400 (from Munguira *et al.*, 2023). **(c)** Temperature of the primary mirror at the time of SuperCam measurements from sol 500 to sol 1000.

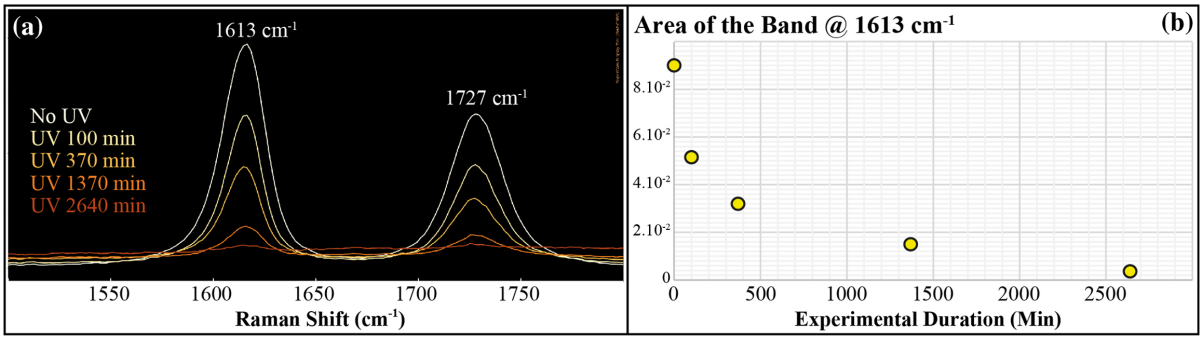

Figure 4 **(a)** Time-resolved Raman spectra (normalised to the total signal) collected using the customised time-resolved Raman spectrometer built by the Cellule Projet @ IMPMC on the spare Ertalyte target before and after exposure to UV radiation in the lab for 100, 370, 1370 and 2640 minutes. **(b)** Evolution of the area of the band at 1613 cm^{-1} as a function of the irradiation duration.

the production of radicals via ester bond cleavage, whereas Norrish type II reactions involve hydrogen atom abstraction via photolysis, eventually inducing a cyclisation process (Grossetête *et al.*, 2000; Sang *et al.*, 2020; Jamalzadeh and Sobkowicz, 2022; Rostampour *et al.*, 2024). Of note, a similar evolution of the colour and the Raman signal was also observed in the laboratory for cystine upon exposure to UV and interpreted as resulting from the creation of electronic defects and/or radicals acting as coloured centres (Megevand *et al.*, 2021). Such an increasing concentration in electronic defects and/or radicals upon exposure to UV explains the yellowing/browning of the Ertalyte target and the evolution of its Raman signal. Consistently, electron spin resonance (ESR) data, collected on Ertalyte irradiated for 2640 minutes, show a new population of defects/radicals absent from the pristine Ertalyte (Fig. S-7).

From sol 290 to sol 480, the data collected on the Ertalyte target define a new stage of evolution. In contrast to the previous stage, the total signal has remained rather constant from sol 280 to sol 480 (Fig. S-6), while the contribution of the Raman signal to the total signal has decreased even more than during the first stage (Fig. 3). This could be an instrumental effect; the temperature of the primary mirror (which is directly related to the temperature of the laser) was rather low on sols 395, 439 and 479, and lower signal is generally collected in such cases (Fig. S-6). Yet, this was not the case for sols 281 and 341 (Fig. S-6). With higher temperature conditions enhancing the effect of exposure to UV (François-Heude *et al.*, 2014; Gogotov and Barazov, 2014), a more likely explanation would be related to the evolution of the mean daily temperature. In fact, air temperature has increased on sol 280 compared to the first several hundred sols, from ~240 K to ~255 K, due to the beginning of the warm season with Mars approaching its perihelion (Fig. 3; Munguira *et al.*, 2023). Such increase in temperature has likely triggered a second stage of UV-induced defect/radical creation, further limiting the contribution of pristine Ertalyte to the total signal. Note that this is definitely not a temperature-related instrumental effect, since higher temperatures lead to higher signals, not the opposite, as demonstrated in the laboratory (Fig. S-8).

Lastly, the Raman signals collected on the Ertalyte target observed during the third stage (from sol 480 to sol 1000) exhibit

a significant variability that is also observed for the signal collected on the sample holder paint on the same sols and seems correlated with the temperature of the primary mirror (Fig. 2 and S-6). Because the relationship between the signal measured and the primary mirror temperature is not linear, it is difficult to take into account this instrumental contribution, preventing the identification of any significant evolution of the Raman signal of the Ertalyte. Instead, as supported by the halting of the browning process from sol 480, it can be assumed that the evolution of the Ertalyte has either stopped or reached a steady state. A steady state would imply that the degradation of the Ertalyte is still ongoing even after 1000 sols on Mars, with some material loss compensating for the progress of radiation damage, resulting in a constant volume of irradiated/damaged Ertalyte contributing to the signal. Determining whether the evolution of the Ertalyte has stopped or reached a steady state cannot be done from the data available.

Altogether, the good agreement between the data collected on Mars and the data collected in the laboratory on Earth suggests that using only UV can be considered as a first order approximation of the radiative environment of the surface of Mars. This means that laboratory studies may provide valuable insights into what organic materials would/should be expected to be found in Martian rocks having been exposed at the surface of Mars. Of note, a Raman signal is still collected after 1000 sols, but 1000 sols remain negligible compared to geological times and, in addition to its intrinsic resistance to UV, the Ertalyte target is a 5 mm thick, 100 % organic target, such quality and quantity not being anticipated for any organic material in Martian rocks. Plus, additional parameters may enhance the degradation of organic compounds upon exposure to UV at the surface of Mars (*e.g.*, Wadsworth and Cockell, 2017; Fornaro *et al.*, 2018). Overall, the present results suggest that prolonged exposure at the surface will eventually cause the severe degradation of organic materials trapped in Martian rocks, making their detection and identification rather challenging.

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Additional Information

Supplementary Information accompanies this letter at https://www.geochemicalperspectivesletters.org/article2509.

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