

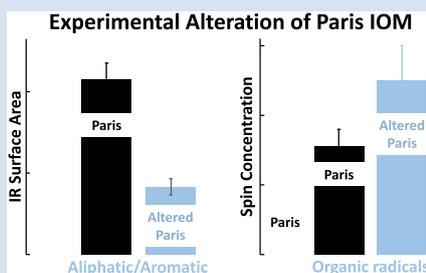
■ Preservation of the isotope signatures in chondritic IOM during aqueous alteration

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Abstract



Mighei-type carbonaceous chondrites (CM) figure among the most primitive objects in the solar system. Yet, they all have experienced various degrees of aqueous alteration having modified their insoluble organic matter (IOM), in a sequence that remains to be accurately constrained. Here, we exposed the IOM of Paris, the least altered CM available, to hydrothermal conditions at 150 °C for 49 days and compared the experimental residue to the IOM of two altered CMs likely originating from the same parent body as Paris, namely Aguas Zarcas and Mukundpura. The experimental residue shows a chemical and isotopic composition similar to those of Aguas Zarcas and Mukundpura IOMs, confirming that these CMs can be seen as altered counterparts of Paris. The abundance of organic radicals also increases significantly during the experiment. Isotopic hotspots do not seem to have been lost

during the experiment, suggesting that the hotspots generally observed within the CM IOMs may date back from pre-accretion era. Of note, the Raman signature of the residue differs from that of the CM IOMs, highlighting the need for further experiments better mimicking asteroidal-like conditions.

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Letter

Carbonaceous chondrites are likely fragments of C-complex asteroids (Vernazza *et al.*, 2017), containing a significant amount of organic matter which may have contributed to the organic inventory of the early Earth. Representing 25 % of the carbonaceous chondrites collected so far (Gounelle *et al.*, 2005), the CMs are among the most primitive objects in the solar system at our disposition. Yet all CMs have experienced various degrees of aqueous alteration, as evidenced by the presence of numerous secondary minerals (Brearley, 2006; Le Guillou *et al.*, 2012; Howard *et al.*, 2015), yielding both weakly altered objects, such as Paris (Hewins *et al.*, 2014) or Asuka (A)-12236 (Glavin *et al.*, 2020), and moderately to totally altered objects in which primary phases have all been replaced. Therefore, a deeper knowledge of the effect of aqueous alteration processes is essential to build a reliable vision of the nature of the primordial, unaltered organic reservoir of CMs.

The CMs exhibit the highest organic carbon contents of all chondrites, with values ranging from 1.5 to 3.4 wt. % (Vacher *et al.*, 2020). Most of this organic component consists of an insoluble organic matter (IOM); macromolecules with rather small aromatic units showing a high degree of crosslinking (Derenne and Robert, 2010; Orthous-Daunay *et al.*, 2013). Of note, the IOM of the most altered CMs are chemically different from

those of the least altered CMs known so far (*i.e.* Paris and Asuka (A)-12236; see Vinogradoff *et al.*, 2017; Glavin *et al.*, 2020). This suggests that the IOM could serve as a chemical tracer of aqueous alteration, and that its early nature can eventually be reconstructed.

Laboratory experiments constitute the most appropriate way to properly constrain the evolution of chondritic IOM during fluid circulation in the asteroidal stage. IOM has been previously shown to experience aromatisation if submitted to high temperature (Okumura and Mimura, 2011; Remusat *et al.*, 2019), as well as significant deuterium depletion (Yabuta *et al.*, 2007; Oba and Naraoka, 2009; Foustoukos *et al.*, 2021), with final D/H values depending on that of the fluid if present. This evolution is significant above 300 °C (Oba and Naraoka, 2009), although the temperature dependence remains limited between 250 and 450 °C (Foustoukos *et al.*, 2021). While these high temperatures certainly reconcile laboratory limitations and astrophysical processes by potentially simulating very slow processes using short duration experiments, they are well beyond the peak temperature undergone by CMs. Although some authors concluded that carbonates were formed at temperature as high as 350 °C, the peak temperature undergone by CMs may not be that far above 100 °C (Guo and Eiler, 2007; Verdier-Paoletti *et al.*, 2017). In that context, we subjected the IOM isolated from the weakly altered Paris (CM2.7) to hydrothermal conditions (150 °C in the

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presence of liquid water) for 49 days and compared the experimental residue (hereafter referred to as ‘experimentally altered Paris’) to IOM of newly recovered and extensively altered CMs, namely Aguas Zarcas (CM2.2; [Martin and Lee, 2020](#)) and Mukundpura (CM2.0; [Rudraswami et al., 2019](#)). Alteration indices for CMs follows classification from [Rubin et al. \(2007\)](#) based on chemistry and mineralogical evidence, with subtypes defined from totally altered (2.0) to not altered (3.0). The molecular structure of the experimental residue was analysed using X-ray absorption near edge structure (XANES) spectroscopy, Fourier transform infrared (FTIR) spectroscopy and time-of-flight secondary ion mass spectrometry (ToF-SIMS). The radicals in the IOM structure were detected through electron paramagnetic resonance (EPR). The distribution of D- and ^{15}N -rich hotspots was imaged by NanoSIMS. The limited amount of recovered IOM only allowed elemental ratios determination via NanoSIMS imaging method for the residue ([Remusat et al., 2019](#)). Finally, the degree of carbon organisation was probed using Raman microspectroscopy.

The elemental composition of the Paris IOM experienced modifications during the experiment. Although the S/C value of the residue is not that different from that of the starting material, its N/C value is lower (by about 15 %) and its O/C value is significantly lower (by about 30 %) as indicated by NanoSIMS ([Fig. S-1](#)). The stability at elevated temperature of S-rich groups in IOM was previously established ([Oba and Naraoka, 2009](#); [Okumura and Mimura, 2011](#)). From the XANES spectra, normalised to the total carbon atoms probed, all IOM here are dominated by aromatic groups and contain various amounts of carbonyl, carboxylic and aliphatic carbons ([Fig. 1a](#)). The widening of the aromatic peak towards 285.5 eV observed in the spectrum of the experimental residue indicates the presence of conjugated rings ([Bernard et al., 2010](#)), evidencing ongoing graphitisation. The experimental residue seemingly contains as low oxygen as the IOM of Aguas Zarcas and Mukundpura ([Fig. 1a](#)).

These results are confirmed by infrared spectroscopy ([Fig. 1b,c](#); IR spectra are reported in [Fig. S-2](#)). The experimental residue contains about as many carbonyl groups as the Aguas Zarcas and Mukundpura IOM, *i.e.* significantly fewer than the

Paris IOM ([Fig. 1b,c](#)). Its aliphatic content is low compared to Paris IOM, but higher than in the Aguas Zarcas and Mukundpura IOM. Yet, the CH_2/CH_3 of the residue is only slightly lower than that of the Paris IOM, while Aguas Zarcas and Mukundpura IOM have the lower values. The increase of aromaticity during the experiment is also confirmed by TOF-SIMS, with a relatively higher concentration of aromatic cycles in the residue compared to that of the Paris IOM ([Fig. S-3b](#)). Altogether, these spectroscopy and mass spectrometry investigations illustrate that, when exposed to hydrothermal conditions, Paris IOM chemically evolves towards the Aguas Zarcas and Mukundpura. The EPR spectra of the IOMs and of the experimental residue also provide information on the concentration of radicals ([Fig. 2a](#)). Concomitantly to the increase of aromaticity, the abundance of radicals detected increases significantly during the experiment, with a 1.6 times higher content in the experimental residue than in the Paris IOM near room temperature ([Fig. 2b](#)). The concentration of radicals in the experimental residue is systematically higher than in the Aguas Zarcas and Mukundpura IOM, Aguas Zarcas having the lowest abundance.

The IOM of the CM here show identical bulk hydrogen isotopic compositions within error bars ([Fig. 3b](#) for δD values of 773 ± 56 ‰ for Paris, 836 ± 116 ‰ for Aguas Zarcas and 780 ± 91 ‰ for Mukundpura). The experimental residue is slightly smaller but remains similar within error bars ($\delta\text{D} = 640 \pm 105$ ‰), and consistent with experimental study on IOM analogues ([Kebukawa et al., 2021](#)). D-rich hotspots usually observed in IOM of CMs ([Busemann et al., 2006](#); [Remusat et al., 2009](#)) are still observed after the experiment ([Fig. 3a](#)), with a distribution of δD values closer to that of Aguas Zarcas and Mukundpura than to that of Paris, as exemplified by δD values ranging from 2800 and 3600 ‰ ([Fig. 3a](#)). This effect does not arise from the hotspot distribution, which show only little modification ([Fig. 3c,d](#)). Nevertheless, the present results differ from results of pyrolysis experiments having evidenced that D-rich hotspots could not withstand high temperature ([Remusat et al., 2009, 2019](#)). If hotspots are unaffected by fluid interactions at 150 °C, then their measured isotopic compositions may reasonably correspond to pre-accretion composition of organic grains exposed to various conditions in the protosolar nebula ([Remusat et al., 2010](#)).

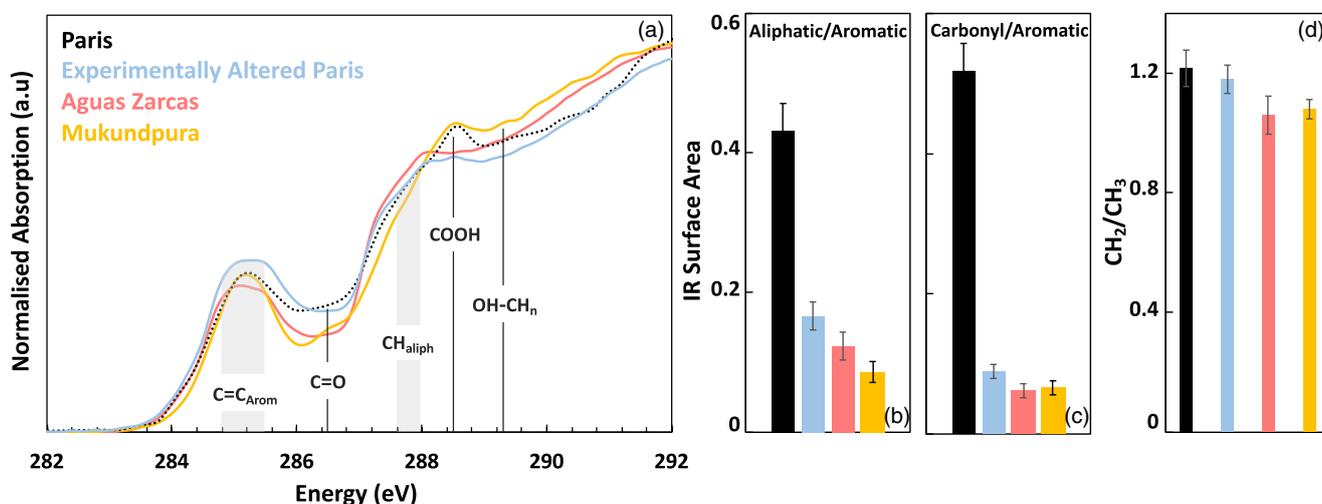


Figure 1 Spectroscopy investigations. (a) Speciation of organic carbon using near edge X-ray absorption fine structure for Paris (black), the experimentally altered Paris (‘Altered Paris’, blue), and altered CM2 Aguas Zarcas (pink) and Mukundpura (yellow) IOMs. The spectra are dominated by aromatic, carbonyl or carboxylic functional groups, with only a minor contribution of aliphatic groups. (b, c), Relative evolution of infrared surface area for aliphatic and carbonyl bands, relative to aromatic carbon. (d) Intensity ratio for aliphatic CH_2 (2925 cm^{-1}) and CH_3 (2960 cm^{-1}) bands.

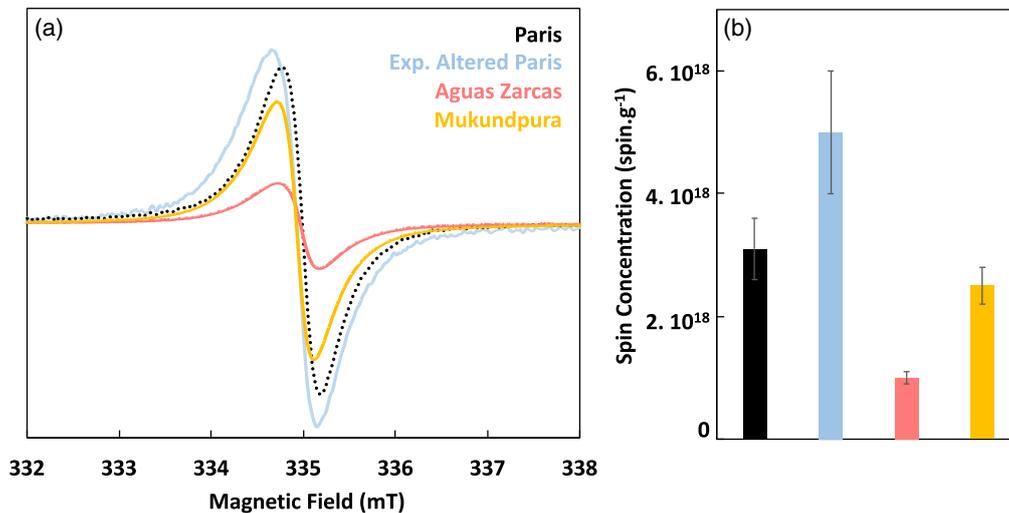


Figure 2 Detection of organic radicals after experimental alteration. (a) Spectra (microwave absorption derivative) at 280–285 K for experimentally altered Paris (blue), CM2.7 Paris (black) and CM2.2 Aguas Zarcas (red) and CM2.0 Mukundpura (yellow). (b) Concentrations of radicals at 280–285 K.

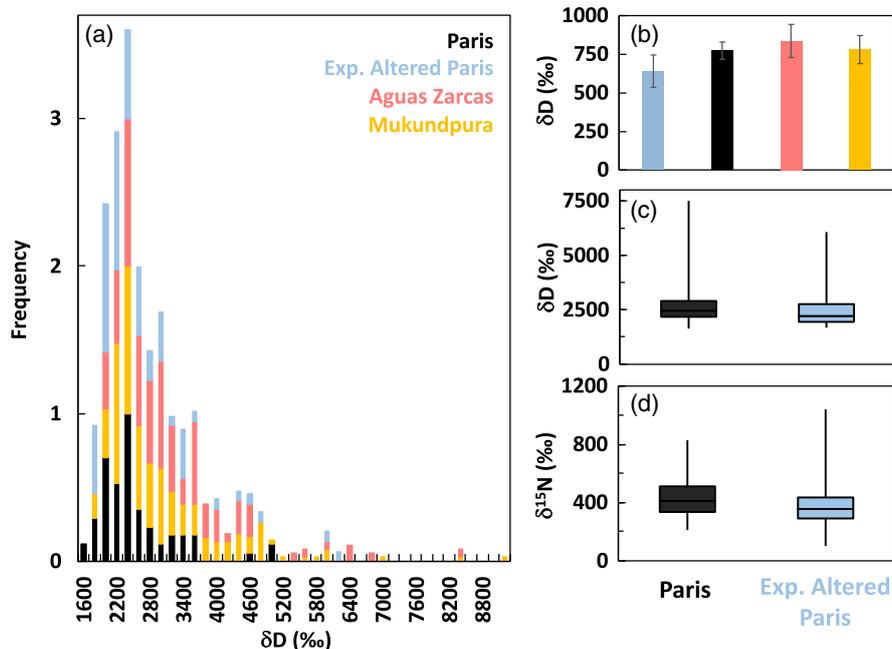


Figure 3 NanoSIMS investigations. (a) Normalised deuterium-rich hotspot distributions in IOM and bulk hydrogen isotopic compositions of Paris (black), experimentally altered Paris CM2 Aguas Zarcas (pink) and Mukundpura (yellow). Distribution are normalised to the number of occurrences and stacked. (b) The bulk hydrogen isotopic compositions of the different CMs and the experimental residue. (c, d) Evolution of the hotspot signatures for hydrogen (analysed on $3600 \mu\text{m}^2$ surface) and nitrogen isotopes (analysed on $2400 \mu\text{m}^2$ surface; images are reported on Fig. S-4).

The Raman spectra of the experimental residue and from the natural objects exhibit two main bands, the so-called defect (D, 1355 cm^{-1}) and graphite (G, 1595 cm^{-1}) bands (Fig. 4a). These two bands are broad for all the samples, and correspond to a highly disordered carbon structure, typical of low thermal alteration (Quirico *et al.*, 2018). All CMs here present similar band widths ($\text{FWHM}_{\text{D,G}}$) and surface area ratios ($A_{\text{D}}/A_{\text{G}}$; Fig. 4b,c). However, the Raman spectrum of the experimental residue is different and rather corresponds to carbonaceous material with an apparent lower degree of carbon organisation, even though it contains more conjugated cycles as indicated by its XANES spectrum.

All considered, the experimental residue is chemically similar to the IOM of CMs having undergone aqueous alteration (Fig. 1). In contrast to the common belief that radicals should be destroyed during alteration, the residue also contains an additional population of radicals easily detected at room temperature (Fig. 2b). Its isotopic composition is not that different from that of the Paris IOM, while its microstructure (*i.e.* its degree of carbon organisation) differs from that of both the Paris, Aguas Zarcas and Mukundpura IOM. Based upon the present results, we suggest that aqueous alteration is not solely responsible for the differences existing between the CM, hence pre-accretion signatures are still detected in natural objects. Nevertheless, we

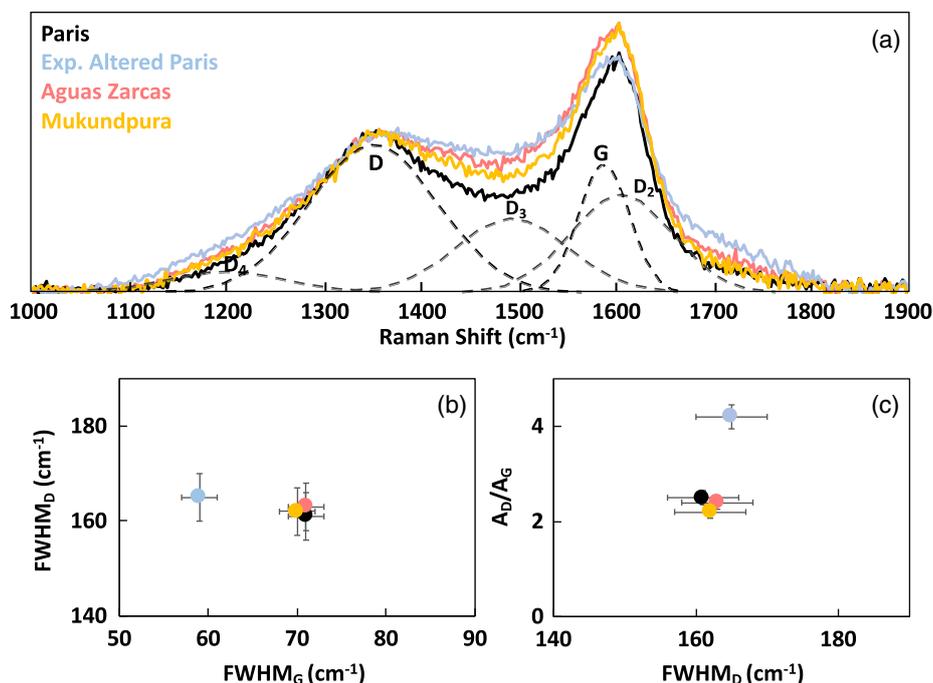


Figure 4 Structural evolution of Paris IOM under asteroidal conditions. (a) Raman spectra of Paris (black), experimentally altered Paris H₂O ('Altered Paris', blue), and altered CM2 Aguas Zarcas (pink) and Mukundpura (yellow). Positions of G, D and minor defect bands are also reported. (b) Comparison of Raman D and G band widths for Paris (black), experimentally altered Paris (blue) and altered CM2 Aguas Zarcas (pink) and Mukundpura (yellow). (c) Evolution of band surface area ratio as function D band width for altered Paris, and the natural objects.

cannot exclude that the present experimental conditions do not perfectly mimic those of natural settings. For these experiments to be closer to the conditions existing in the parent body of CMs, additional parameters should have been considered, such as the presence of mineral phases. Clay minerals constitute the main phase of aqueously altered chondrites (Beck *et al.*, 2010) and are closely associated with organic matter in chondrites (Le Guillou *et al.*, 2014). Clay minerals can in fact adsorb, trap and/or store organic molecules (Lagaly *et al.*, 2013), and possibly inhibit the production of IOM under asteroidal conditions (Viennet *et al.*, 2022). A potential key limitation here could be the strict closed system conditions under which the present experiments have been conducted. While it is consistent with the isochemical alteration undergone by primitive chondrites (Bland *et al.*, 2009), the diversity of d¹⁸O values of the minerals phases comprising CM2s (*e.g.*, Kimura *et al.*, 2020) pleads in favour of a relatively open system. Hence, while showing chemical and isotopic composition congruent with altered chondritic matter, the present study highlights the need for further experiments to better constrain the effect of aqueous alteration on the chemical, isotopic and structural evolution of IOM during asteroidal aqueous alteration and eventually build a reliable vision of the nature of the primordial, unaltered organic reservoir of CMs.

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Additional Information

Supplementary Information accompanies this letter at <https://www.geochemicalperspectivesletters.org/article2233>.



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