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## The Piancaldoli meteorite: A forgotten primitive LL3.10 ordinary chondrite

Yves Marrocchi, Lydie Bonal, Jérôme Gattacceca, Laurette Piani, Pierre Beck, Richard Greenwood, Jolantha Eschrig, Anne Basque, Pasquale Mario Nuccio, Franco Foresta Martin

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26

27 **Abstract**

28         The Piancaldoli ordinary chondrite fell in northern Italy on 10 August 1968.  
29 Preliminary studies led to its classification as a LL3.4 unequilibrated ordinary chondrite.  
30 However, recent developments in classification procedures have prompted us to re-examine  
31 its mineralogical, petrographic, spectroscopic, chemical, and isotopic features in a multi-  
32 technique study. Raman spectra and magnetic properties indicate that Piancaldoli experienced  
33 minimal thermal metamorphism, consistent with its high bulk hydrogen content and the Cr  
34 contents of ferroan olivines in its type-II chondrules. In combination with findings of previous  
35 studies, our data thus confirm the variability of Cr contents in ferroan olivines in type-II  
36 chondrules as a proxy of thermal metamorphism. Furthermore, our results reveal that  
37 Piancaldoli is less altered than previously reported and should be reclassified as **an** LL3.10  
38 unequilibrated ordinary chondrite. Our results also imply that the bulk deuterium enrichment,  
39 as observed in Piancaldoli (LL3.10), Bishunpur (LL3.15), and Semarkona (LL3.00), is a  
40 specific signature of the most primitive unequilibrated ordinary chondrites. Based on our  
41 results, we propose that, to date, Piancaldoli is the second least-altered unequilibrated  
42 ordinary chondrite fall after Semarkona. This work reiterates the importance of meteorite  
43 collections worldwide as fundamental resources for studying the formation conditions and  
44 evolution our solar system.

45

46 **Keywords:** Ordinary chondrites, meteorite classification, thermal metamorphism, Piancaldoli

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## 51 **1. Introduction**

52

53 On 26 April 1803, thousands of stones fell upon the small city of L'Aigle, France  
54 (~150 km west of Paris). At that time, the extraterrestrial nature of meteorites was not yet  
55 recognized and they were still considered to be ‘thunderstones’ resulting from atmospheric  
56 processes. The precise description of the L'Aigle fall by dozens of people and the unique  
57 nature of the recovered rocks led physicist Jean-Baptiste Biot to conclude their origin to be  
58 unambiguously extraterrestrial. **The extraterrestrial origin of meteorites had already been**  
59 **proposed on the basis of meteor ballistic trajectories and chemical analyses performed by**  
60 **Ernst Florens Friedrich Chladni (see Marvin, 2010 for a review) and Edward Howard**  
61 **(Howard et al., 1802).** Since these works, meteorites have been recognized as objects of  
62 scientific importance, and natural history museums worldwide have established invaluable  
63 meteorite collections. Despite the recent boom in space exploration, most of our current  
64 understanding of the conditions and chronology of the formation of the solar system has been  
65 established thanks to the meticulous work of meteorite collection and curation (Heck et al.  
66 2019).

67 Over the past two decades, the number of meteorites reported to the Meteoritical  
68 Society has increased exponentially thanks to field trips organized to cold and hot deserts  
69 expressly to search for meteorites (e.g., **Harvey, 2003**; Gattacceca et al. 2011; Evatt et al.  
70 2020). These harvests have enabled the characterization of some of the most primitive  
71 meteorites known so far, which are now available to the scientific community. However,  
72 meteorite finds are exposed to terrestrial weathering and contamination, which largely affect  
73 their primitive characteristics (Alexander 2017; Stephant et al. 2018; Vacher et al. 2020). For  
74 instance, Antarctic and Saharan samples are commonly affected by Na loss and/or metal  
75 alteration, and Antarctic samples by oxygen isotopic exchange with Antarctic water

76 (Alexander et al. 2018). Thus, as meteorite falls are collected before any significant terrestrial  
77 alteration can occur, they are of primary importance for deciphering the formation of the solar  
78 system.

79 In addition to terrestrial weathering, most chondrites experienced secondary alteration  
80 on their asteroidal parent bodies, resulting in changes to their primary texture, mineralogy,  
81 and chemical and isotopic compositions (Brearley 2006; Marrocchi et al. 2018). These  
82 petrographic and chemical modifications are the basis of petrologic type assignments (Van  
83 Schmus and Wood 1967) on a scale from 1 to 6, reflecting the progressive roles of low-  
84 temperature aqueous alteration (types 3 to 1) and thermal metamorphism (types 3 to 6). In  
85 theory, type-3 chondrites thus correspond to primitive materials that underwent minimal  
86 modifications after their agglomeration in the protoplanetary disk 4.56 Ga (Amelin et al.,  
87 2010). However, new classification procedures based on thermoluminescence (TL; Sears et al.  
88 1980), chemical characteristics (e.g., Cr content in ferroan olivines in type-II chondrules;  
89 Grossman and Brearley 2005) and Raman spectroscopy (Bonal et al. 2006, 2007, 2016)  
90 permit the quantification of discrete variations among type-3 chondrites. Accordingly, type-3  
91 chondrites are now subdivided into categories ranging from 3.00 to 3.9, with 3.00 being the  
92 most *primitive* extraterrestrial materials, virtually unchanged since their agglomeration in the  
93 disk.

94 Because these advances in the classification of minimally altered chondrites are  
95 relatively recent (Grossman and Brearley 2005; Bonal et al. 2006, 2016), many previously  
96 analyzed chondrites have not been characterized according to the new classification criteria  
97 nor studied in further detail after their initial classification. This is fundamental, however, as  
98 the identification of primitive chondrites with minimal parent-body alteration improves our  
99 understanding of the formation and evolution of solids in the protoplanetary disk. In particular,  
100 Al-Mg chronology requires the characterization of refractory inclusions and chondrules in

101 chondrites of petrologic subtypes  $\leq 3.1$  due to the fast self-diffusion of Mg in minerals and  
102 glassy mesostases (Kita and Ushikubo 2012; Van Orman et al. 2014; Marrocchi et al. 2019).

103 While vacationing in Sicily in 2018, the first author (YM) fortuitously met Franco  
104 Foresta Martin, a geologist and a journalist at the Italian national daily newspaper *Corriere*  
105 *della Sera*. He brought to my knowledge the history of the Piancaldoli meteorite that fell near  
106 Florence in northern Italy on 10 August 1968. A fireball was observed over Yugoslavia and  
107 central and northern Italy, followed by explosions accompanying the break-up of the  
108 meteorite. Three meteorite fragments totaling 13.1 g were found on the roof of a house in  
109 Piancaldoli. The Piancaldoli meteorite was first classified as a L3 chondrite (Carapezza and  
110 Nuccio 1971) before being reclassified as LL3 (Carapezza et al. 1975, 1976). Although no  
111 Raman or TL data were available for Piancaldoli, Rubin et al. (1982) proposed its  
112 reclassification as petrologic type  $3.4 \pm 0.2$  based on silicate compositions. Consequently,  
113 Piancaldoli is listed as a LL3.4 ordinary chondrite in the Meteoritical Bulletin Database. Here  
114 we report a comprehensive description of Piancaldoli and show that its secondary thermal  
115 history was overestimated. Our results indicate that Piancaldoli is the second least-altered  
116 unequilibrated ordinary chondrite fall known to date.

117

## 118 **2. Material and methods**

### 119 **2.1 Mineralogical and petrographic observations**

120 We surveyed two sections of Piancaldoli: thin section USNM 5649 from the National  
121 Museum of Natural History, Smithsonian Institution (Washington D.C., USA) and a thick  
122 section prepared at the Centre de Recherches Pétrographiques et Géochimiques (CRPG-  
123 CNRS, Nancy, France) from a parent sample provided by the Dipartimento scienze della  
124 Terra e del Mare (Palermo, Italy). We also characterized a thin section of Semarkona  
125 (LL3.00; Grossman and Brearley, 2005; Muséum national d'Histoire naturelle, Paris, France)

126 and a thick section of Bishunpur (LL3.15; Grossman and Brearley, 2005; Muséum national  
127 d'Histoire naturelle, Paris, France) for comparison. The sections were imaged by scanning  
128 electron microscopy on a JEOL JSM-6510 equipped with a Genesis energy dispersive x-ray  
129 (EDX) detector at the CRPG using a 3 nA electron beam accelerated at 15 kV. The chemical  
130 compositions of olivine grains were quantified using a Cameca SXFive electron microprobe  
131 at the Université Pierre et Marie Curie (UPMC, Camparis, Paris, France) using a 150 nA  
132 focused beam accelerated at 15 kV. We analyzed Na, Mg, Si, Al, K, Ca, Fe, Ti, Cr, and Mn in  
133 olivine grains. The high beam current allowed detection limits for silicates to be 100 ppm for  
134 Al, Ca, and Ti, 150 ppm for Mn and Si, and 200 ppm for Na, K, Cr, Fe, and Mg. The PAP  
135 software was used for matrix corrections.

136

## 137 **2.2 Raman spectroscopy**

138 Raman spectroscopy was performed at the Ecole Normale Supérieure de Lyon  
139 (Laboratoire de Géologie de Lyon – Terre, Planètes, Environnement, France) using a LabRam  
140 Raman spectrometer (Horiba Jobin-Yvon) equipped with a 600 gr/mm grating and a Spectra  
141 Physics Ar<sup>+</sup> laser ( $\lambda = 514$  nm). Because carbonaceous matter is sensitive to laser-induced  
142 heating and the Raman bands of polyaromatic carbonaceous matter are dispersive, we  
143 followed the analytical procedures of Bonal et al. (2016, summarized here) to avoid any laser  
144 alteration of the carbonaceous matter and to facilitate meaningful comparisons with reference  
145 meteorites from the literature. The laser was focused through a 100× objective to obtain a ~2  
146  $\mu\text{m}$  spot size. The power on the sample was 500  $\mu\text{W}$ . Each acquisition comprised two 30-s  
147 integrations that were averaged to make the final spectrum. Spectra were acquired under  
148 atmospheric conditions over the wavenumbers 500–2,200  $\text{cm}^{-1}$ , covering the first-order  
149 carbon bands. Raman spectra of carbonaceous matter in the matrix of Piancaldoli were  
150 obtained both on isolated matrix fragments (50 spectra) and *in situ* in the thick section

151 prepared at the CRPG (32 spectra). Around 30 matrix fragments (typical apparent diameter  
152 around 30  $\mu\text{m}$ ) were manually selected from a gently crushed raw piece of Piancaldoli (initial  
153 sample of 50 mg) according to their color and texture under a binocular microscope. The  
154 selected matrix fragments were pressed between two glass slides that served as the substrate  
155 for the Raman analyses. The G and D bands were fitted with Breit-Wigner-Fano and  
156 Lorentzian profiles, respectively, to retrieve spectral parameters. We compared the width (full  
157 width at half maximum) of the D band ( $\text{FWHM}_D, \text{cm}^{-1}$ ) and its intensity relative to that of the  
158 G band ( $I_D/I_G$ ) to those of reference samples as these spectral parameters are the most  
159 sensitive to the maturity range of type-3 chondrites (e.g., Bonal et al., 2016).

160

### 161 **2.3 Infrared spectroscopy**

162 Infrared (IR) spectra were obtained with a Bruker HYPERION 3000 infrared  
163 microscope at the Institut de Planétologie et d'Astrophysique de Grenoble (IPAG, Grenoble,  
164 France). The IR beam was focused through a 15 $\times$  objective to a typical spot size on the  
165 sample of  $40 \times 40 \mu\text{m}^2$ . Spectra were acquired at  $4 \text{ cm}^{-1}$  spectral resolution with a MCT  
166 detector cooled with liquid nitrogen. Particular care was devoted to sample preparation, which  
167 is critical in IR microspectroscopy. Samples must be sufficiently thin ( $<100 \mu\text{m}$ ) and their  
168 surfaces flat and parallel to avoid absorption-band saturation and scattering artifacts,  
169 respectively (Raynal et al. 2000). Small matrix fragments (30–50  $\mu\text{m}$ ) were selected under a  
170 binocular microscope and crushed between two diamond windows for analyses in the 4000–  
171 650  $\text{cm}^{-1}$  spectral range. The diamond windows were loaded into an environmental cell,  
172 designed and built at IPAG, capable of achieving temperatures up to 300  $^\circ\text{C}$  under primary or  
173 secondary dynamic vacuum ( $10^{-4}$  to  $10^{-7}$  mbar). Optical access is via both sides of the cell  
174 through KBr windows, enabling measurements in transmission mode. Samples were  
175 progressively heated and analyzed at 20, 100, and 300  $^\circ\text{C}$  for 1 hour at each temperature.



176 The transmission spectra were automatically converted to absorbance ( $A = -\log (T/T_0)$ ,  
177 where  $T_0$  and  $T$  are the background and sample transmittance, respectively). To remove  
178 interferences and scattering effects, a spline baseline was calculated and subtracted from the  
179 raw data. The resulting spectra were normalized to the intensity of the silicate Si-O stretching  
180 band ( $1000 \text{ cm}^{-1}$ ) at  $10 \text{ }\mu\text{m}$  thickness, which does not evolve over the temperature range of  
181 the measurements. Reflectance spectra were obtained on powdered fragments of Piancaldoli  
182 using the SHADOWS instrument (Potin et al., 2018). Spectra were normalized to  
183 spectralon<sup>TM</sup> and infragold<sup>TM</sup> and measured under standard mode.

184

## 185 **2.4 Magnetic properties**

186 Hysteresis measurements were performed on a 122-mg bulk (matrix + chondrules)  
187 fragment of Piancaldoli with a Princeton Micromag vibrating sample magnetometer with a  
188 maximum applied field of 1 T and a sensitivity of  $\sim 5 \times 10^{-9} \text{ A m}^2$ . We analyzed hysteresis  
189 loops to obtain the ratio of saturation remanent magnetization ( $M_{RS}$ ) to saturation  
190 magnetization ( $M_S$ ) and the coercive force ( $B_C$ ).  $M_S$  was determined by correcting the  
191 hysteresis loop for the high-field susceptibility determined by a linear fit of the hysteresis  
192 loops for applied fields  $>0.9 \text{ T}$ . Remanent coercive force ( $B_{CR}$ ) was determined by back-field  
193 experiments performed with the magnetometer. The coercivity spectrum was estimated from  
194 the derivative of the isothermal remanent magnetization by stepwise measurements using the  
195 magnetometer. The evolution of low field magnetic susceptibility at low temperature was  
196 studied using an Agico MFK1 apparatus equipped with a CSL cryostat. All magnetic  
197 measurements were performed at the Centre Européen de Recherche et d'Enseignement des  
198 Géosciences de l'Environnement (Aix-en-Provence, France).

199

200

## 201           **2.5 Hydrogen isotopes**

202           Hydrogen concentrations and isotopic compositions were measured using the Thermo  
203 Scientific EA IsoLink - DeltaV IRMS System at the CRPG according to the procedure  
204 detailed in Lupker et al. (2012), which we summarize here. A small piece of the meteorite  
205 was crushed into powder and two aliquots (5.97 and 7.82 mg, respectively) were loaded into  
206 tin capsules and degassed at 120 °C under vacuum for 48 h to minimize the contribution of  
207 adsorbed atmospheric water (Lupker et al. 2012; Vacher et al. 2016, 2020). Hydrogen isotopic  
208 compositions are expressed relative to that of Standard Mean Ocean Water (SMOW,  
209  $D/H_{SMOW} = 155.76 \times 10^{-6}$ ) as  $\delta D$  [‰] =  $[(D/H_{sample} / D/H_{SMOW}) - 1] \times 1,000$ .  
210 Reproducibilities estimated from reference materials are better than 10% ( $2\sigma$ ) for H  
211 concentration and  $0.5 \times 10^{-6}$  for D/H (or 5‰ for  $\delta D$ ).

212

## 213           **2.6 Oxygen isotopes**

214           High-precision oxygen isotopic measurements were performed at the Open University  
215 (Milton Keynes, UK) using an infrared laser-assisted fluorination system (Miller et al., 1999;  
216 Greenwood et al., 2017). Two individual analyses of whole rock chips of Piancaldoli were  
217 undertaken, with each replicate having a mass of about 2 mg. After fluorination, the O<sub>2</sub>  
218 released was purified by passing through two cryogenic nitrogen traps and over a bed of  
219 heated KBr. O<sub>2</sub> analyses were performed using a MAT 253 dual inlet mass spectrometer.  
220 Analytical precision ( $2\sigma$ ), based on replicate analyses of an internal obsidian standard, is  $\pm$   
221 0.053 ‰ for  $\delta^{17}O$ ,  $\pm$  0.095 ‰ for  $\delta^{18}O$  and  $\pm$  0.018 ‰ for  $\Delta^{17}O$  (Starkey et al., 2016). Oxygen  
222 isotopic analyses are reported in standard  $\delta$  notation, where  $\delta^{18}O$  has been calculated as:  $\delta^{18}O$   
223 =  $[(^{18}O/^{16}O_{sample}/^{18}O/^{16}O_{ref.}) - 1] \times 100$  and similarly for  $\delta^{17}O$  using the  $^{17}O/^{16}O$  ratio.  $\Delta^{17}O$ ,  
224 which represents the deviation from the terrestrial fractionation line, has been calculated as:

225  $\Delta^{17}\text{O} = \delta^{17}\text{O} - 0.525 \times \delta^{18}\text{O}$  in order to compare our results with those obtained by Clayton et  
226 al (1991).

227

### 228 **3. Results**

#### 229 **3.1 Petrographic overview**

230 The Piancaldoli sections contain abundant chondrules (Figs. 1, 2) and chondrule  
231 fragments (Fig. 3) in an optically dark matrix. Type-I chondrules (characterized by abundant  
232 Fe-Ni metal beads, FeO-poor silicates and volatile element depletion) are predominantly  
233 olivine-rich and relatively homogeneous (Fig. 2A), whereas type-II chondrule (characterized  
234 by FeO-rich silicates and more chondritic abundances of volatile elements) are  
235 compositionally variable with normally zoned and relict, Mg-rich, olivine grains (Villeneuve  
236 et al., 2020; Fig. 2B). Some ferroan olivine grains show igneous zoning profiles, with rims  
237 enriched in Cr (Fig. 4), and chromite exsolutions are commonly observed in type-II chondrule  
238 olivines (Fig. 5). Although some type-II chondrules show devitrified mesostases, both types  
239 are characterized by well-preserved glassy mesostases (Figs. 2–4) with no specific evidence  
240 of aqueous alteration. Dusty olivine grains were observed in about 5% of 350 observed  
241 chondrules. Opaque mineral assemblages are rounded and occur predominantly along  
242 chondrule exteriors, but also rarely within the interiors of some chondrules (Fig. 1). Ferroan  
243 olivines in type-II chondrules of Piancaldoli contain 0.2–0.5 wt.%  $\text{Cr}_2\text{O}_3$ , averaging  $0.36 \pm$   
244  $0.20$  wt.%  $\text{Cr}_2\text{O}_3$  ( $1\sigma$ , Fig. 6, Table S1); those of Semarkona and Bishunpur contain  $0.45 \pm$   
245  $0.11$  and  $0.23 \pm 0.13$  wt.%  $\text{Cr}_2\text{O}_3$ , respectively (Fig. 6).

246

#### 247 **3.2 Raman and Infrared spectral characteristics**

248 Every Raman spectrum obtained of the Piancaldoli matrix exhibits the D and G bands,  
249 indicating the presence of polyaromatic carbonaceous matter. We obtained mean values of

250  $\text{FWHM}_D (\text{cm}^{-1}) = 173.4 \pm 20.2$  and  $I_D/I_G = 0.912 \pm 0.089$  for *in-situ* thin-section analyses ( $n =$   
251  $32$ ) and  $\text{FWHM}_D (\text{cm}^{-1}) = 159.2 \pm 37.8$  and  $I_D/I_G = 0.996 \pm 0.145$  for matrix fragments ( $n =$   
252  $50$ ; the errors reflecting the variable structural order of the polyaromatic carbonaceous matter).

253 These spectral parameters are thus variable throughout Piancaldoli: the structural order of the  
254 polyaromatic carbonaceous matter in the CRPG section of Piancaldoli reflects a metamorphic  
255 grade similar to that of Bishunpur (LL3.15), whereas the spectral parameters obtained for the  
256 matrix fragments are more dispersed and tend to reflect a slightly higher metamorphic grade  
257 (Fig. 7). This discrepancy might be explained by the brecciated nature of Piancaldoli  
258 (discussed in section 4).

259 The presence of aliphatic bands in the  $2800\text{--}3000 \text{ cm}^{-1}$  range of IR spectra (Fig. 8)  
260 confirms our manual selection of matrix fragments because the matrix is the only  
261 petrographic component containing organics in chondrites. These IR spectra mostly exhibit  
262 bands related to Si-O stretching around  $1000 \text{ cm}^{-1}$ , indicating olivine to be the dominant  
263 silicate. The stretching mode of molecular water is poorly visible at  $\sim 3400 \text{ cm}^{-1}$ , and the OH  
264 band at  $\sim 3670 \text{ cm}^{-1}$  is absent (Fig. 8).

265 The reflectance spectrum obtained for Piancaldoli presents the two typical absorption  
266 bands at 1 and 2- $\mu\text{m}$  diagnostic of Fe-bearing silicates (olivine + pyroxene). The shape bands,  
267 in particular the reflectance maxima around  $1.6 \mu\text{m}$ , is typical of LL chondrites (Fig 9).  
268 However, the depth of the two bands is lower than typical ordinary chondrites, which is likely  
269 related to the unequilibrated nature of the sample. The visible reflectance at  $550 \text{ nm}$  is lower  
270 than type 4-6 ordinary chondrites, and in low-side of the range of value measured for type 3  
271 ordinary chondrites.

272

273

274

### 275           **3.3 Magnetic properties**

276           The hysteresis properties of the bulk Piancaldoli fragment are  $M_S = 6.79 \text{ A m}^2/\text{kg}$ ,  $M_{RS}$   
277  $= 0.216 \text{ A m}^2/\text{kg}$ ,  $B_C = 9.02 \text{ mT}$ , and  $B_{CR} = 62.8 \text{ mT}$ , in agreement with observed values for  
278 LL3 chondrite falls (Gattacceca et al. 2014). The  $M_S$  value indicates a metal content similar to  
279 other LL3 chondrites. The coercivity spectrum of Piancaldoli (Fig. 10) is difficult to fit with a  
280 typical combination of log-normally distributed components, and is notably shifted toward  
281 low coercivities compared to other LL chondrite falls, including LL3 chondrites. In particular,  
282 the absence of a peak in the high coercivity range ( $>500 \text{ mT}$ ) precludes the presence of  
283 ordered tetrataenite in cloudy zone microstructures of zoned taenite (Gattacceca et al. 2014).  
284 Such microstructures form only by the cooling of Fe-Ni metal from above  $320 \text{ }^\circ\text{C}$  at cooling  
285 rates slower than  $\sim 1000 \text{ }^\circ\text{C}/\text{Myr}$  (Gattacceca et al. 2014; Maurel et al. 2019) and can be  
286 disordered by impact-induced thermal events at or above shock stage S3/S4 (Gattacceca et al.  
287 2014). In the case of Piancaldoli (shock stage S1), the absence of ordered tetrataenite  
288 constrains the peak metamorphism temperature to  $<320 \text{ }^\circ\text{C}$ . LL chondrites with broadly  
289 similar coercivity spectra include Bishunpur, Krymka, and Chainpur, although Piancaldoli has  
290 the lowest coercivities. Conversely, Vicencia (LL3) and all other equilibrated LL chondrites  
291 have high-coercivity components attributable to ordered tetrataenite in the cloudy zone  
292 structure (Fig. 10). It is therefore likely that the classification of Vicencia as LL3.2 (Keil et al.  
293 2015) should be revised to a higher petrologic subtype, consistent with the observed Raman  
294 spectral parameters (see their Fig. 8, and Fig. 7 herein). Indeed, Vicencia appears to be more  
295 metamorphosed than Chainpur (LL3.4) and less than Tieschitz (H/L3.6).

296           The low temperature magnetic measurements performed on Piancaldoli do not reveal a  
297 Verwey transition at 120K that would indicate the presence of magnetite. Magnetite has been  
298 detected through low temperature magnetic measurements in a few primitive LL3 falls, such  
299 as Semarkona and Krymka (Keil et al., 2015), and through petrographic observation in, e.g.,

300 Semarkona (Hutchison et al., 1987). The absence of the Verwey transition in Piancaladoli  
301 confirms the absence of significant aqueous alteration.

302

### 303 **3.4 H and O isotopes**

304 The two analyzed aliquots of Piancaldoli contain 0.124 and 0.127 wt.% H, equivalent  
305 to 1.12 and 1.14 wt.% H<sub>2</sub>O, respectively, and have respective D/H values of  $193.2 \times 10^{-6}$  ( $\delta D$   
306  $= -57.7\text{‰}$ ) and  $187.8 \times 10^{-6}$  ( $\delta D = -57.6\text{‰}$ ). These values are broadly typical of mildly  
307 metamorphosed LL3 chondrites (Robert et al. 1979; McNaughton et al. 1982; Yang and  
308 Epstein 1983; Alexander et al. 2012; Vacher et al. 2020). Although the hydrogen  
309 concentrations are on the higher end of those measured for LL3 chondrites similar to  
310 Semarkona (~0.04–0.12 wt.% H), the D/H ratios are intermediate among the range observed  
311 for LL3 chondrites ( $158 \times 10^{-6}$  to  $505 \times 10^{-6}$ ; Fig. 11).

312 The results of oxygen isotope measurement of Piancaldoli are plotted in Fig. 12. The  
313 two individual analyses show consistent results with  $\delta^{18}\text{O} = 5.16 \pm 0.21 \text{‰}$  ( $2\sigma$ ) and  $\delta^{17}\text{O} =$   
314  $3.63 \pm 0.18 \text{‰}$  ( $2\sigma$ ). Compared to other ordinary chondrites, Piancaldoli plots at the edge of  
315 the LL chondrite field with a relatively low  $\Delta^{17}\text{O}$  value of  $0.95 \pm 0.08 \text{‰}$  ( $2\sigma$ ; Fig. 12).

316

## 317 **4. Discussion**

318 The Piancaldoli chondrite contains sharply defined type-I and type-II chondrules (Fig.  
319 2) with a mean apparent diameter of  $901 \pm 445 \text{ }\mu\text{m}$ , and a median apparent diameter of  $804 \text{ }\mu\text{m}$   
320 ( $n=352$ ). Rubin et al. (1982) described a Piancaldoli clast with ~100 radial-pyroxene micro-  
321 chondrules 0.2–64  $\mu\text{m}$  in apparent diameter. Chondrule olivine and pyroxene crystals show  
322 heterogeneous compositions with (i) olivine varying between 66 and 98 mol% of forsterite  
323 and (ii) pyroxene ranging from 2 to 24 mol% of ferrosilite (Carapezza et al., 1976; Rubin et  
324 al., 1982). The chemical composition of Piancaldoli is typical of LL ordinary chondrites with

325  $\text{FeO}/\text{SiO}_2 = 0.48$ ,  $\text{SiO}_2/\text{MgO} = 1.62$ , and  $\text{Fe}^0/\text{Fe}_{\text{tot}} = 0.12$  (Carapezza et al. 1976; Wasson and  
326 Kallemeyn, 1988). In addition, the chemical compositions of the fine-grained opaque matrix  
327 and the whole rock were used to propose that Piancaldoli is an unequilibrated LL ordinary  
328 chondrite of petrologic type  $3.4 \pm 0.2$  (Carapezza et al. 1976; Rubin et al. 1982). However, all  
329 data collected herein indicate that Piancaldoli is more primitive than this estimation. Thus, in  
330 this section, we (i) assess the extent of thermal alteration experienced by Piancaldoli and (ii)  
331 propose a new classification.

332         The distribution of Cr contents in ferroan olivines in type-II chondrules is considered a  
333 proxy of incipient thermal metamorphism. We obtained a mean value of  $0.36 \pm 0.19$  wt.%  
334  $\text{Cr}_2\text{O}_3$  (Fig. 6; Table S1) for type-II chondrule olivines in Piancaldoli, and our measurements  
335 of those in Semarkona and Bishunpur during the same analytical session are in good  
336 agreement with previous reports (Grossman and Brearley 2005; Fig. 6). This suggests that  
337 Piancaldoli experienced minimal thermal metamorphism corresponding to petrologic type  
338 3.10 (Fig. 6; Grossman and Brearley 2005), supported by the occurrence of igneous Cr zoning  
339 patterns (i.e., rims enriched in Cr) in some type-II chondrule olivines (Fig. 4). Such primary  
340 textures are extremely sensitive to thermal metamorphism as they are replaced by complex,  
341 subparallel, needle-like exsolutions of Cr-rich phases (likely chromite; Fig. 5) upon heating  
342 (Grossman and Brearley 2005). As the separation of Cr-rich phases from FeO-rich olivine  
343 grains is nearly complete by petrologic type 3.2 (Grossman and Brearley 2005), the observed  
344 Cr zonings in Piancaldoli olivine are a strong argument in favor of Piancaldoli's primitive  
345 nature.

346         Tetrataenite dominates the magnetic properties of all but the most primitive LL  
347 chondrites (and those that experienced shock stage S4 or greater). The absence of tetrataenite  
348 revealed by the magnetic properties of Piancaldoli sets it among the most primitive LL  
349 chondrites and constrains the peak metamorphic temperature to  $<320$  °C. This low degree of

350 thermal metamorphism is supported by the structural order of the polyaromatic carbonaceous  
351 matter in Piancaldoli. Indeed, the Raman spectral parameters of polyaromatic carbon bands of  
352 Piancaldoli are comparable with those of Bishunpur (LL3.15; Fig. 7). Interestingly, spectra  
353 obtained on matrix fragments selected from a piece of bulk Piancaldoli exhibit a wider  
354 spectral variability, and, on average, a higher maturity (higher  $I_D/I_G$  and lower  $FWHM_D$ , Fig.  
355 7). Therefore, some parts of Piancaldoli appear to have experienced a higher peak  
356 metamorphic temperature consistent with a petrologic type closer to 3.4. This variable  
357 metamorphic grade is consistent with previous petrographic observations and could be related  
358 to the brecciated nature of Piancaldoli (as attested by the presence of light- and dark-colored  
359 matrix domains in section USNM 5640; Rubin et al. 1982). Indeed, the fine exsolutions of Cr-  
360 rich phases commonly observed in Piancaldoli (Fig. 5) suggest that it is more altered than  
361 Semarkona, in which such textures are absent (Grossman and Brearley 2005).

362 Infrared spectra of Piancaldoli's matrix indicate, if not the absence, the really low  
363 abundance of secondary hydrated minerals, and thus that Piancaldoli did not experience  
364 significant aqueous alteration. This is in contrast to the matrix of Semarkona (LL3.00), which  
365 clearly contains hydrated material, most likely smectite (Alexander et al. 1989; Quirico et al.  
366 2003).

367 The hydrogen content and isotopic composition of Piancaldoli show important  
368 similarities with the two least-altered ordinary chondrite falls, Semarkona and Bishunpur (Fig.  
369 11). The hydrogen content of Piancaldoli (~ 0.125 wt.%) is among the highest observed in  
370 ordinary chondrites (despite the removal of adsorbed atmospheric water before measurement  
371 in our methodology; Vacher et al. 2016, 2020) and is similar to the highest values reported for  
372 Semarkona (0.12 wt.% H; Alexander et al. 2012), further indicating that Piancaldoli  
373 experienced minimal thermal metamorphism, which would have resulted in hydrogen loss  
374 (Fig. 11). As neither phyllosilicates nor magnetite were observed in Piancaldoli, the H budget



375 is likely controlled by: (i) insoluble organic matter and (ii) chondrules (silicates and glassy  
376 mesostases). However, a detailed TEM study should be performed to clearly rule out the  
377 presence of phyllosilicates (Hutchison et al., 1987). Although enriched in deuterium  
378 compared to more metamorphosed ordinary chondrites, the rather low D/H signature of  
379 Piancaldoli relative to Semarkona and Bishunpur may be a consequence of the D/H  
380 heterogeneities observed among unequilibrated LL chondrites. Indeed, D/H values reported  
381 for Semarkona and Bishunpur are highly heterogeneous, ranging from  $393$  to  $609 \times 10^{-6}$  and  
382 from  $253$  to  $504 \times 10^{-6}$ , respectively (McNaughton et al. 1982; Yang and Epstein 1983;  
383 Alexander et al. 2012; Piani et al. 2015). Therefore, it seems that heterogeneous H isotopic  
384 compositions can be taken as an argument for the primitive nature of unequilibrated LL  
385 ordinary chondrites.

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## 388 **5. Concluding remarks**

389 We studied the Piancaldoli chondrite via scanning electron microscopy, electron microprobe  
390 analyses, hydrogen and oxygen isotopic analyses, magnetic measurements, and Raman and IR  
391 spectroscopy. Characteristics of the Raman spectra and magnetic properties of Piancaldoli are  
392 consistent with its primitive nature as inferred from the variability of Cr contents in ferroan  
393 olivines in type-II chondrules. Furthermore, the high bulk H content of Piancaldoli indicates  
394 that it experienced only minimal thermal metamorphism. Our petrographic and chemical  
395 results therefore suggest that Piancaldoli should be reclassified as a LL3.10 unequilibrated  
396 ordinary chondrite, rather than a LL3.4 as previously proposed. We further suggest that the  
397 deuterium observed in Piancaldoli, Bishunpur, and Semarkona are specific signatures of the  
398 most primitive unequilibrated LL ordinary chondrites. Our results thus demonstrate that (i)  
399 Piancaldoli is the second least-altered unequilibrated ordinary chondrite fall after Semarkona,

400 and (ii) primitive meteorites are most likely sitting, unstudied, in meteorite collections  
401 worldwide.

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450 **Figure captions**

451

452 **Fig. 1:** Composite back-scattered electron images of Piancaldoli: (A) USNM 5649 and (B)  
453 Piancaldoli-CRPG.

454

455 **Fig. 2:** Back-scattered electron images of representative (A) type-I and (B) type-II chondrules  
456 with well-preserved mesostases.

457

458 **Fig. 3:** Back-scattered electron image of a fish-shaped chondrule fragment.

459

460 **Fig. 4:** EDX maps of (A) Si + Mg and (B) Cr in euhedral ferroan olivine grains in a type-II  
461 chondrule of Piancaldoli. Olivine rims are enriched in Cr, representing an igneous zoning  
462 profile.

463

464 **Fig. 5:** Back-scattered electron image of a ferroan olivine grain in a type-II chondrule  
465 showing complex, subparallel, needle-like exsolutions of a Cr-rich phase (likely chromite).

466

467 **Fig. 6:** Standard deviation on Cr<sub>2</sub>O<sub>3</sub> content *versus* mean Cr<sub>2</sub>O<sub>3</sub> content of ferroan olivines in  
468 type-II chondrules in Piancaldoli compared to Semarkona and Bishunpur (data from this  
469 study and Grossman and Brearley, 2005). Piancaldoli lies between Semarkona and Bishunpur,  
470 suggesting it is of petrologic subtype 3.10.

471

472 **Fig. 7:** Spectral parameters (FWHMD vs. ID/IG, **averages and one standard deviations**) of  
473 Raman bands attributed to carbonaceous materials in Piancaldoli compared to other

474 meteorites (see Bonal et al., 2016). Data for Vicencia (LL), Axtell (CV3) and ALHA 77307  
475 (CO3) are also reported (see section 3.3).

476

477 **Fig. 8:** Baseline-corrected transmission IR spectra (normalized to the height of the Si-O band  
478 at  $1000\text{ cm}^{-1}$ ) of matrix fragments of Piancaldoli, acquired at  $20\text{ }^{\circ}\text{C}$  under dynamic vacuum.  
479 A vertical offset was applied to ease comparison. Notably, the water band is very weak and  
480 olivine is the dominant silicate.

481

482 **Fig. 9:** Left: Reflectance spectra of Piancaldoli powder compared to average ordinary  
483 chondrites spectra from the RELAB database. Spectra were normalized at  $550\text{ nm}$ . Right:  
484 Reflectance value at  $550\text{ nm}$  of Piancaldoli compared to ordinary chondrites, as a function of  
485 petrographic type.

486

487 **Fig. 10:** Coercivity spectra, i.e., the derivative of the isothermal remanent magnetization, IRM,  
488 (normalized to saturation remanence) as a function of the acquisition field, of LL chondrite  
489 falls. The petrologic types and masses of each chondrite is indicated.

490

491 **Fig. 11:** Hydrogen content (reported as  $1/H_{\text{wt}\%}$ ) and isotopic composition of Piancaldoli  
492 compared to H, L, and LL ordinary chondrites. The hydrogen concentration of Piancaldoli  
493 plots in the range of poorly metamorphosed LL3 chondrites. Error bars represent  $1\sigma$  standard  
494 deviations for chondrites having multiply reported hydrogen compositions (data from Robert  
495 et al. 1979; McNaughton et al. 1982; Yang and Epstein 1983; Alexander et al. 2012; Vacher  
496 et al. 2020).

497

498 **Fig. 12:** The oxygen isotopic composition of Piancaldoli compared to H, L, and LL ordinary  
499 chondrites (data from Clayton et al., 1991 and Greenwood et al., 2020) . Piancaldoli plots at  
500 the edge of the LL chondrite field with a relatively low  $\Delta^{17}\text{O}$  value.

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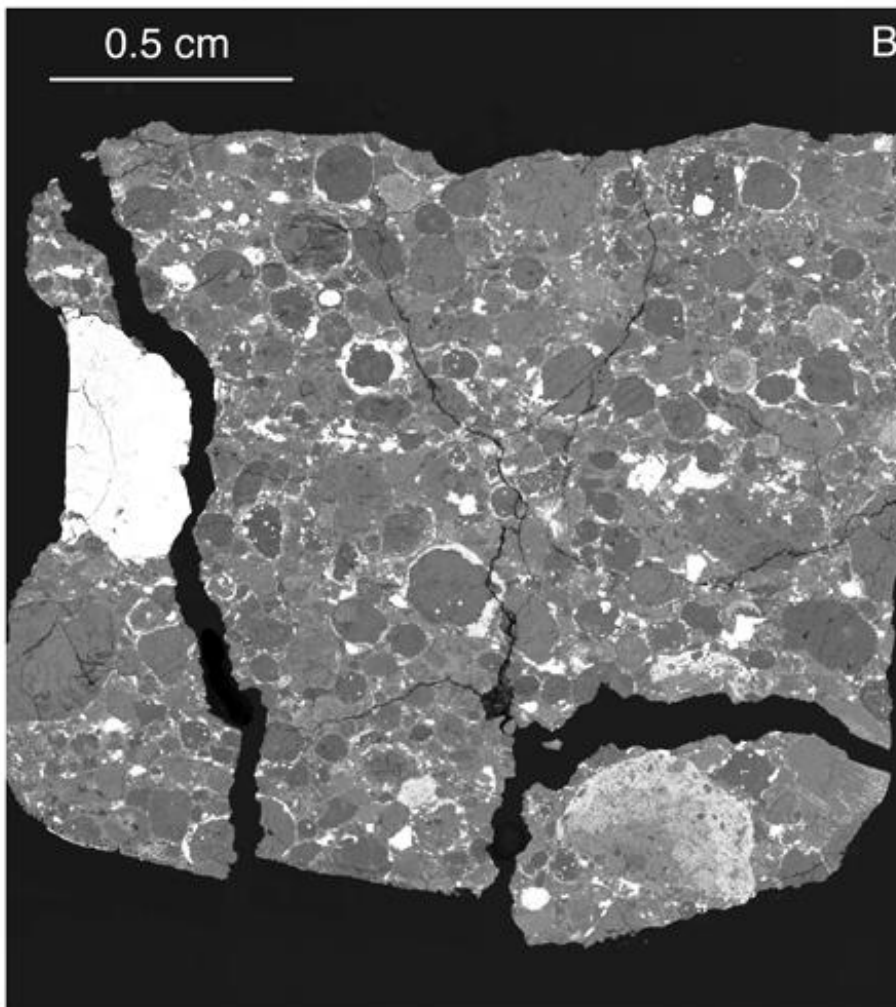
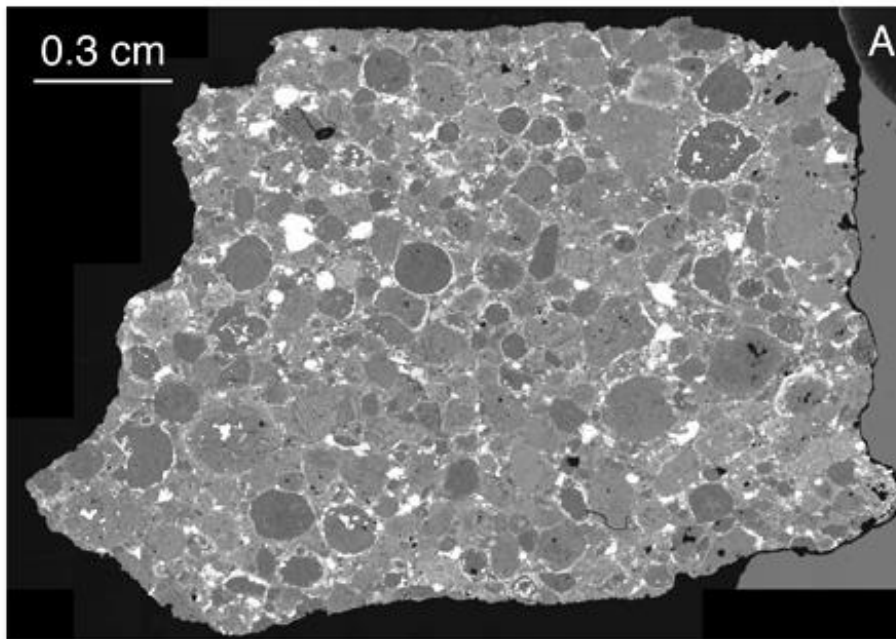
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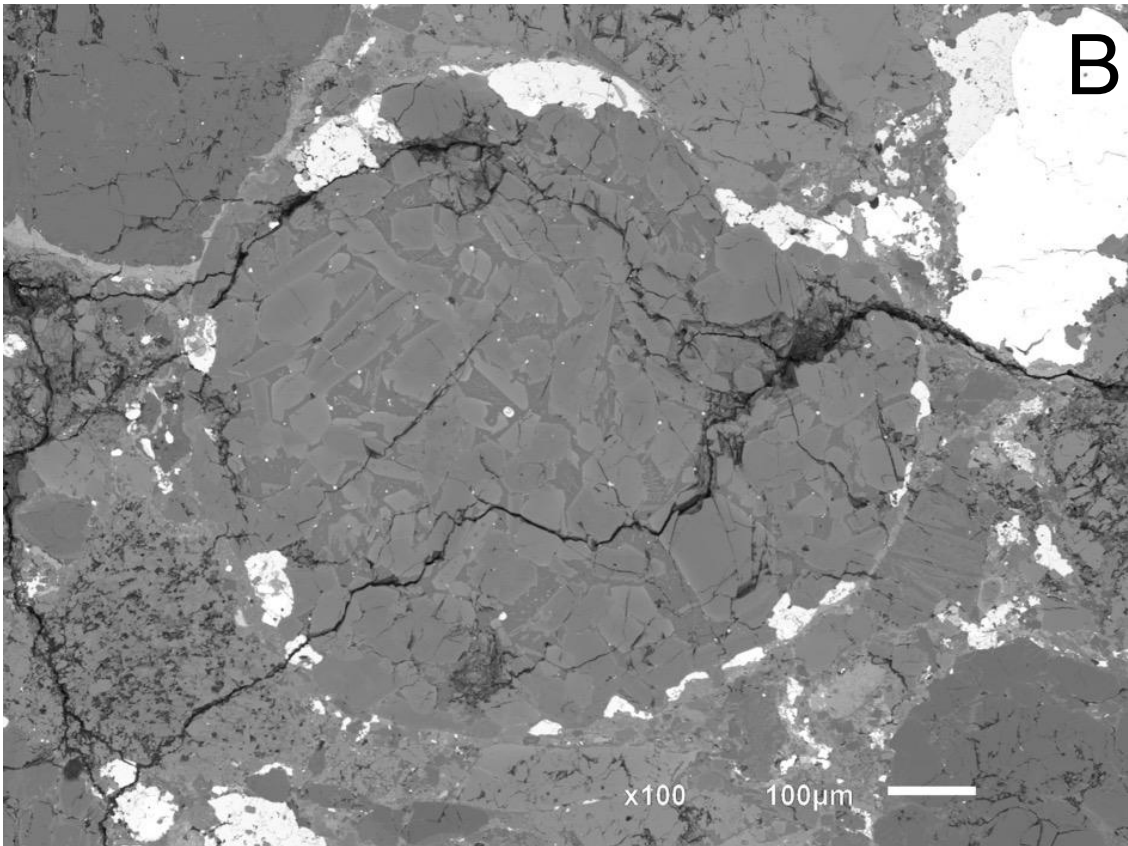
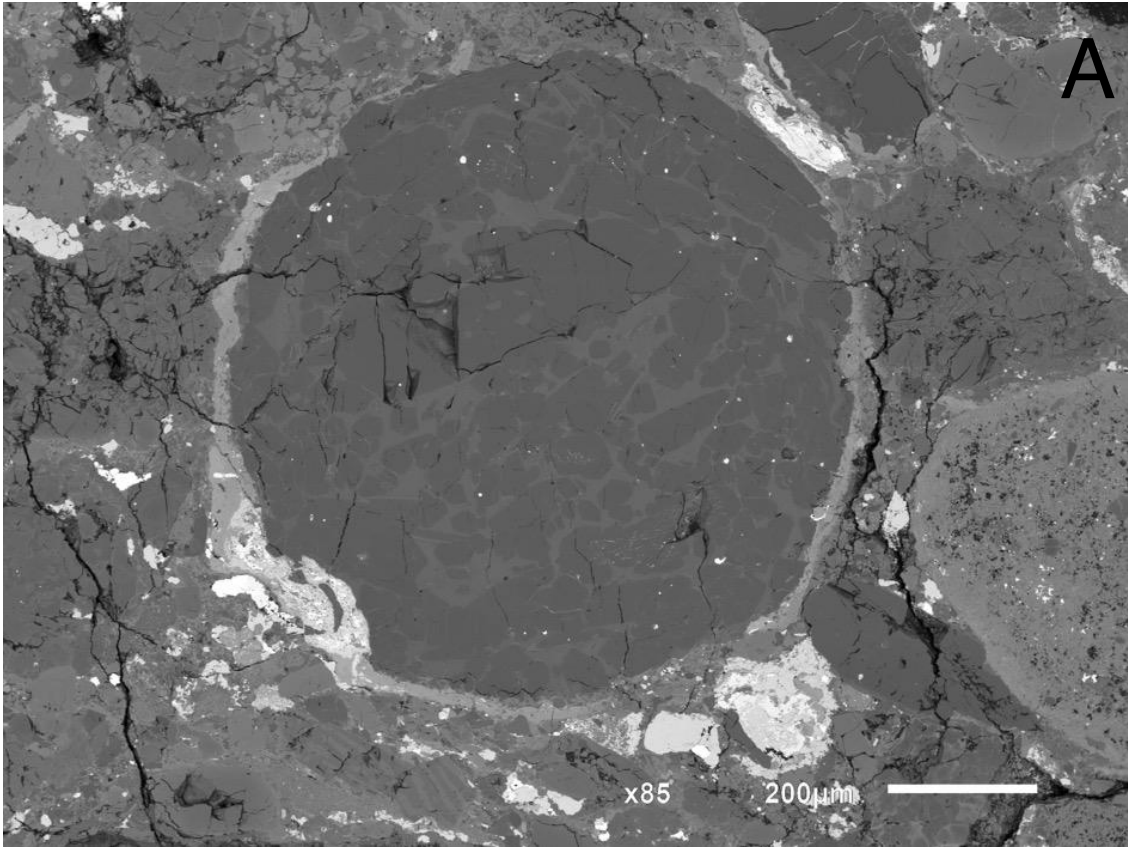
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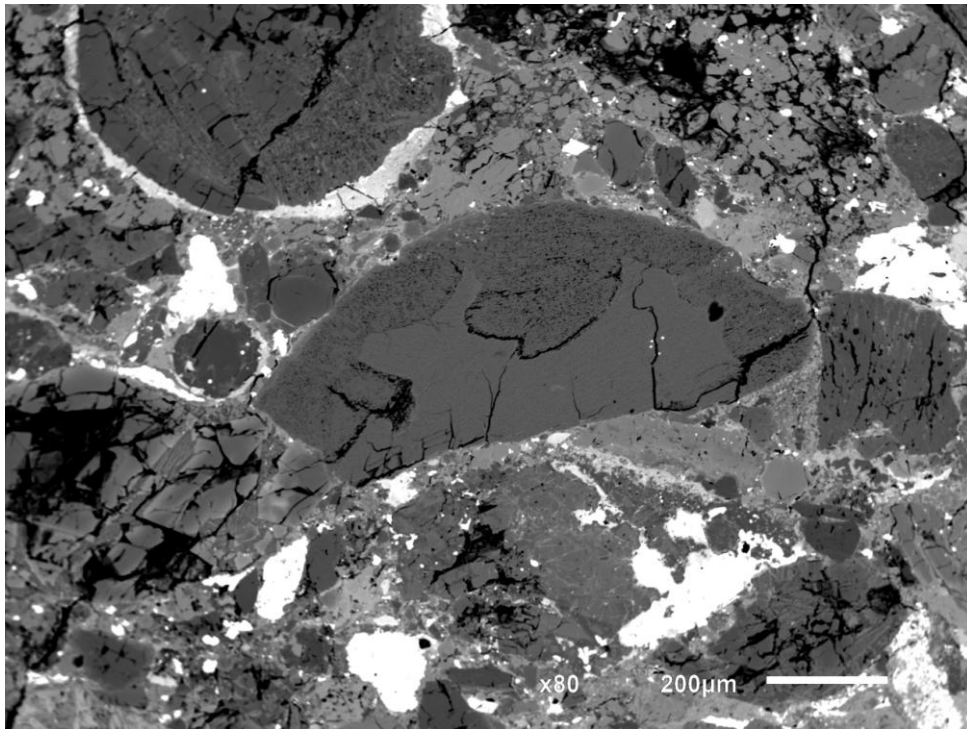
Fig. 1



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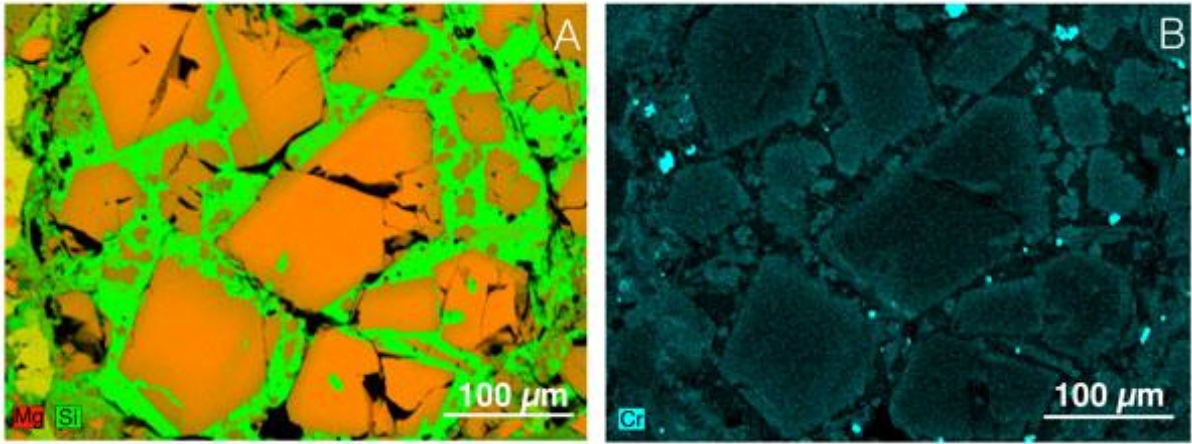
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Fig. 4

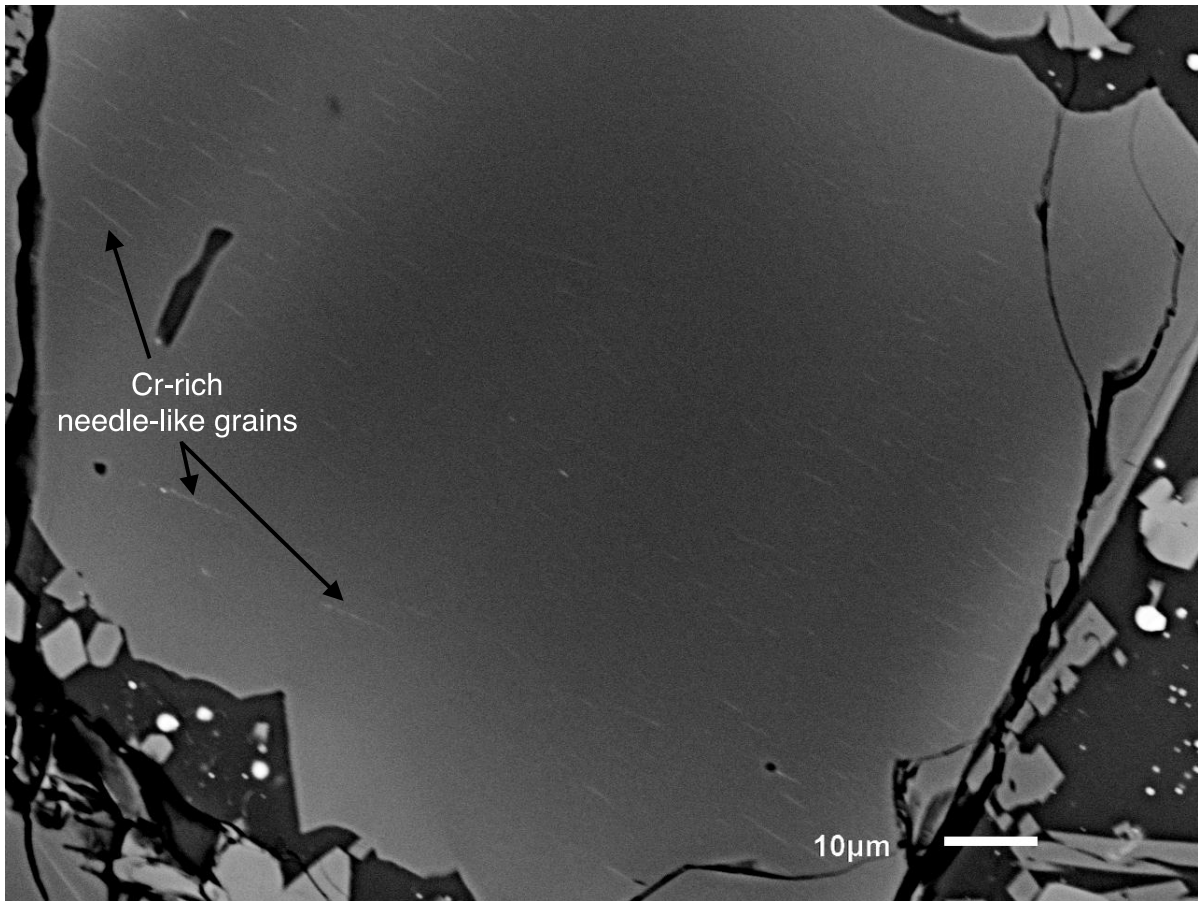
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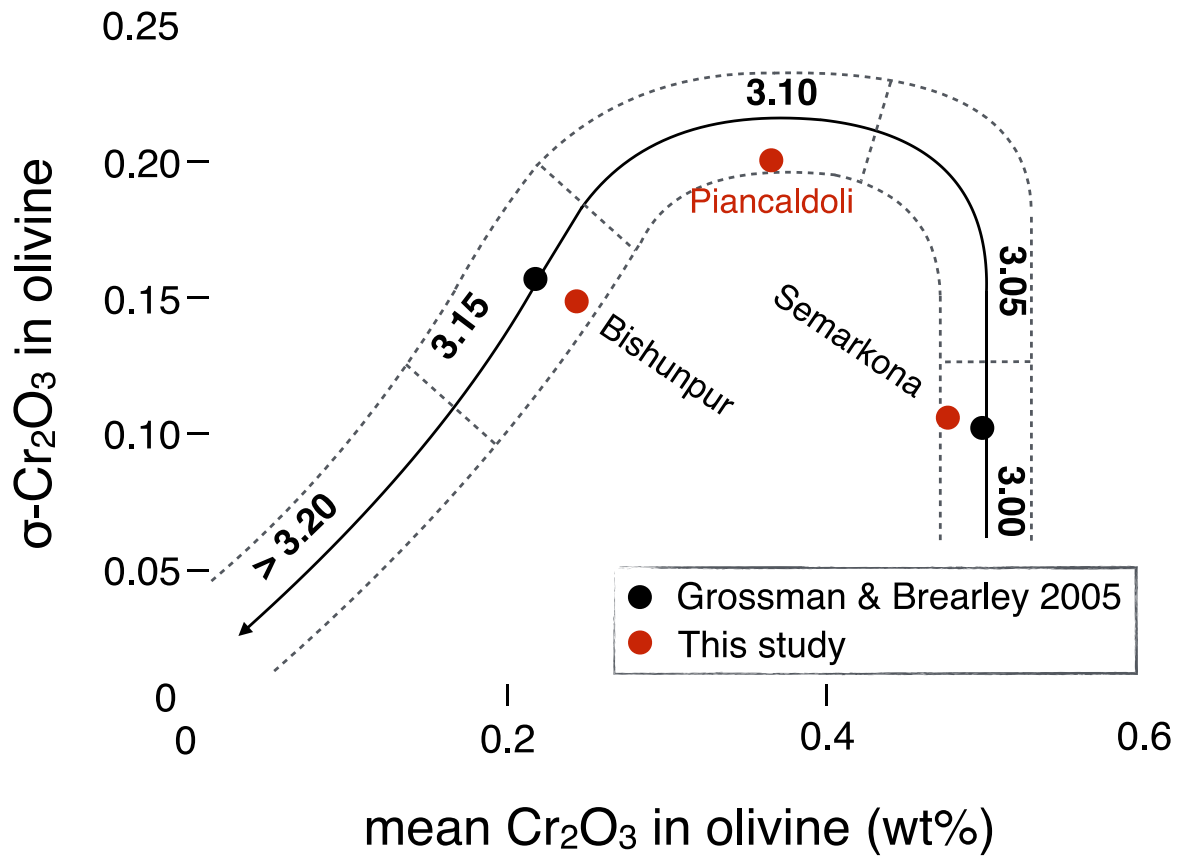


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Fig. 5

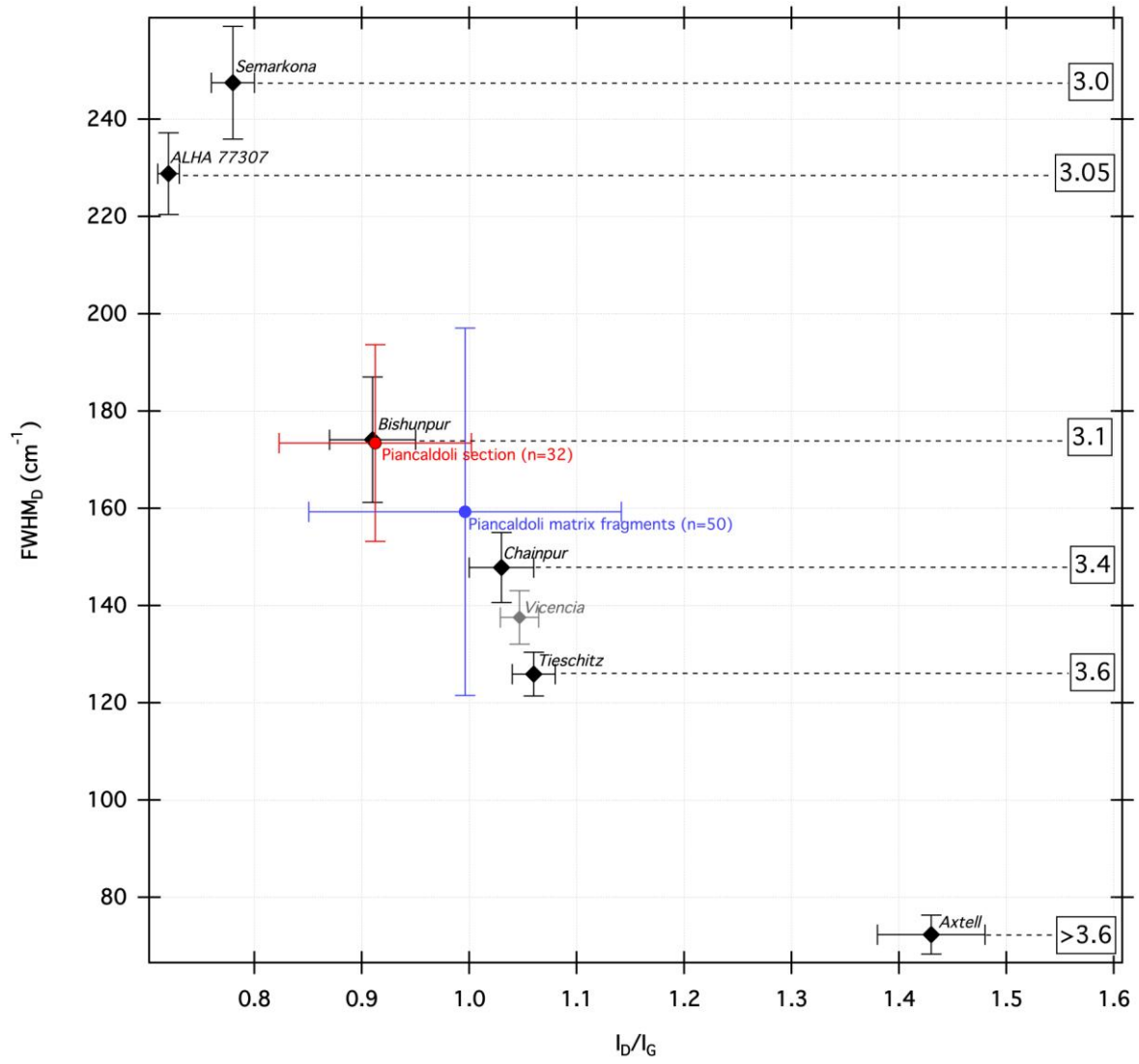




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Fig. 6

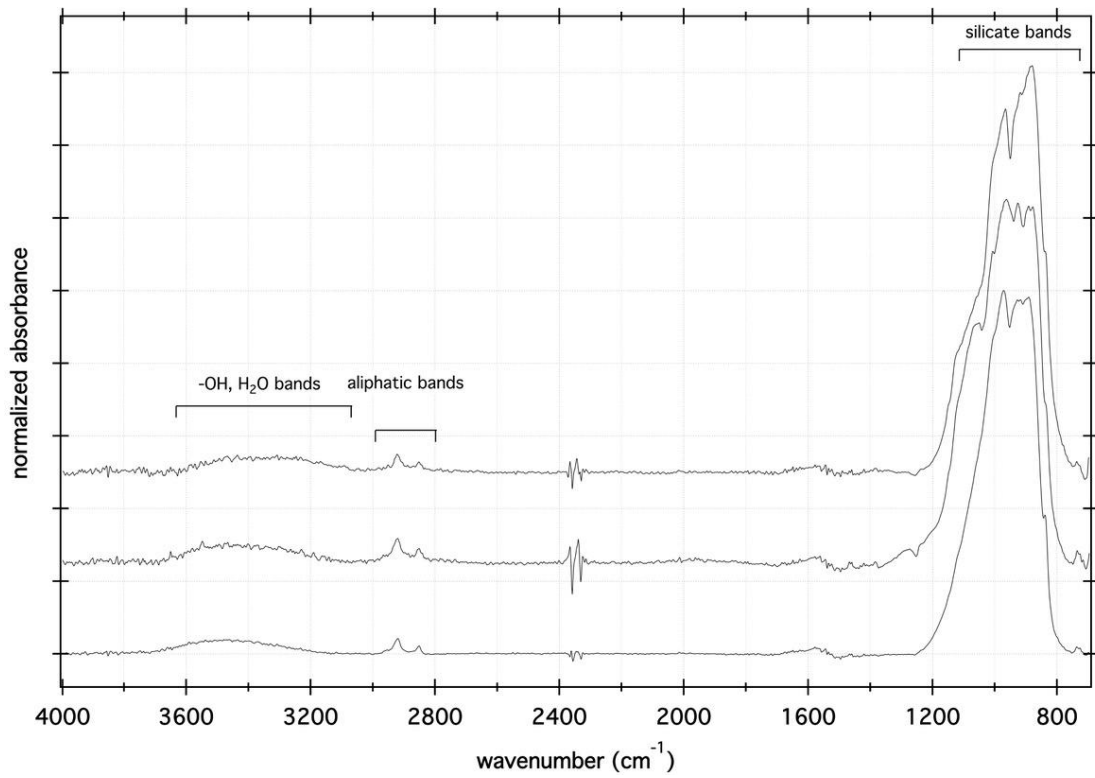


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Fig. 7



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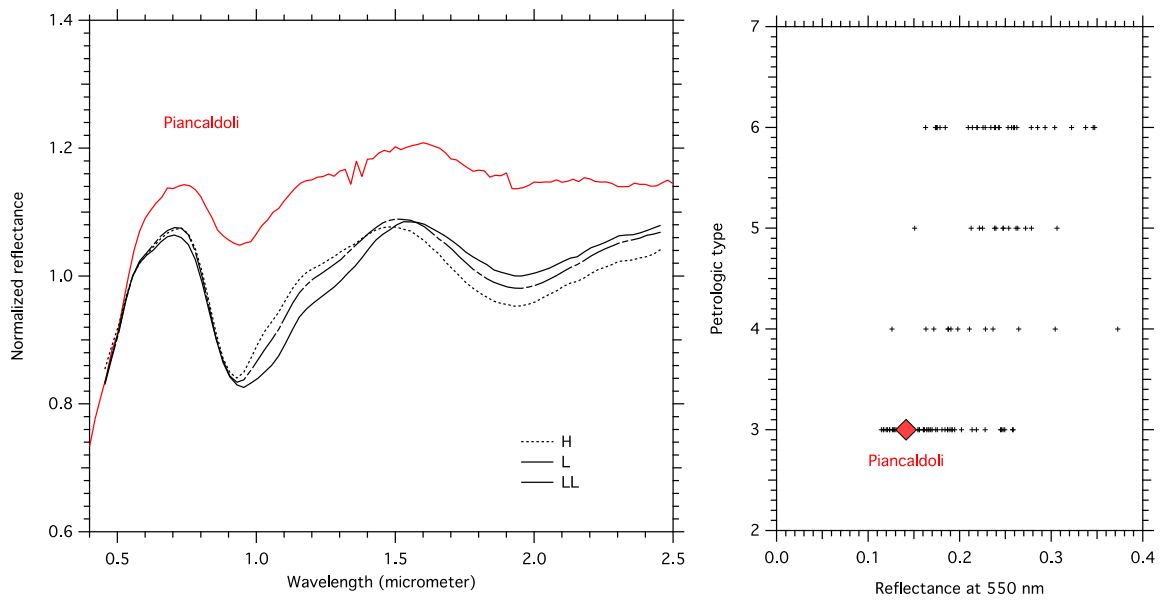
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Fig. 8



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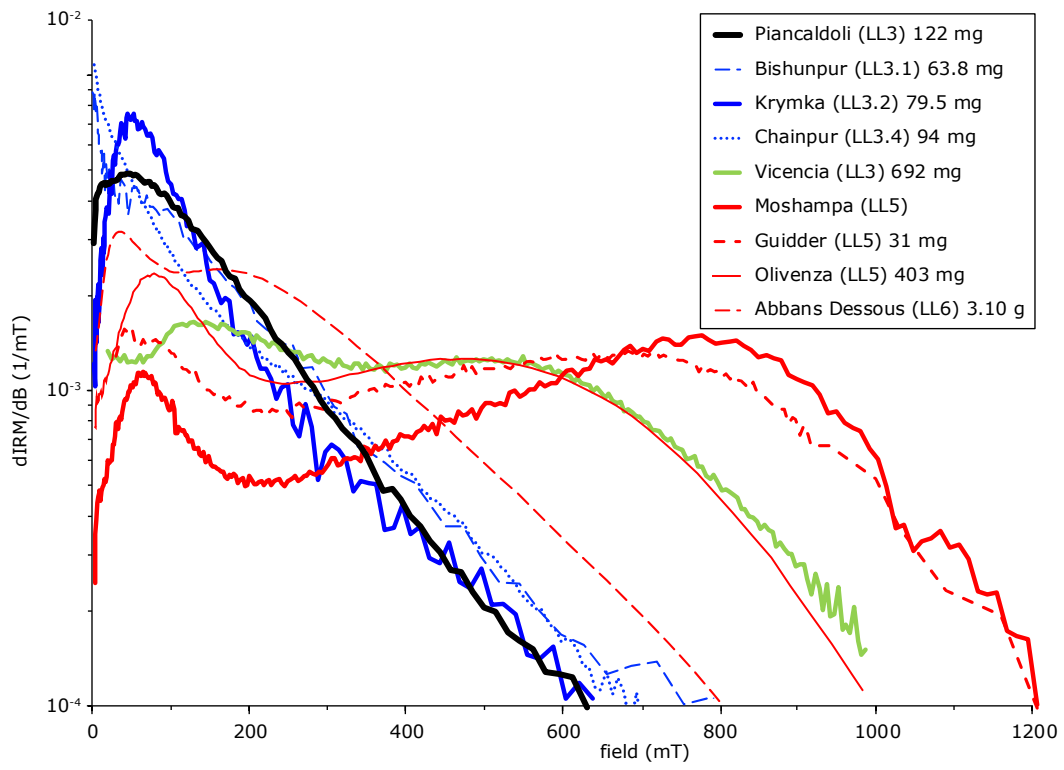
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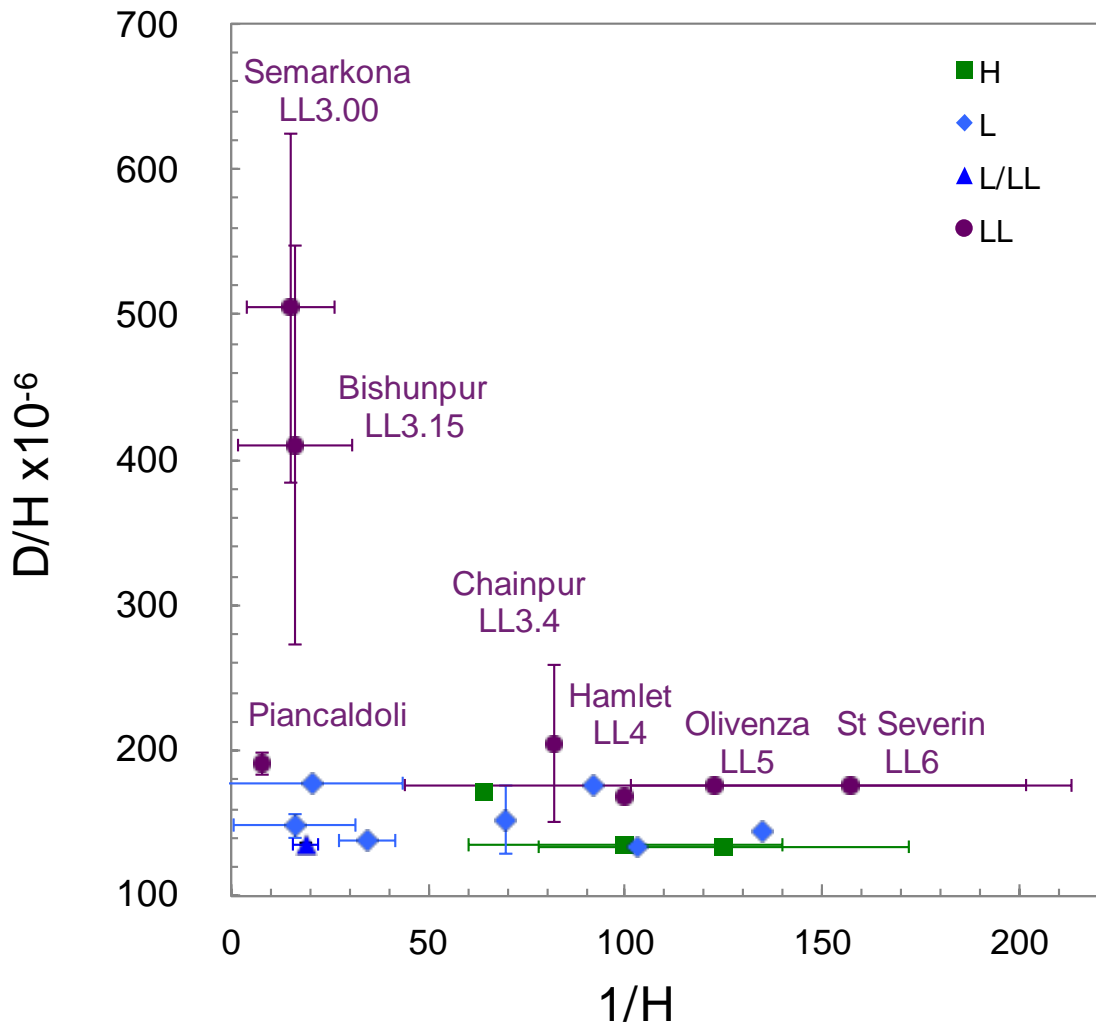


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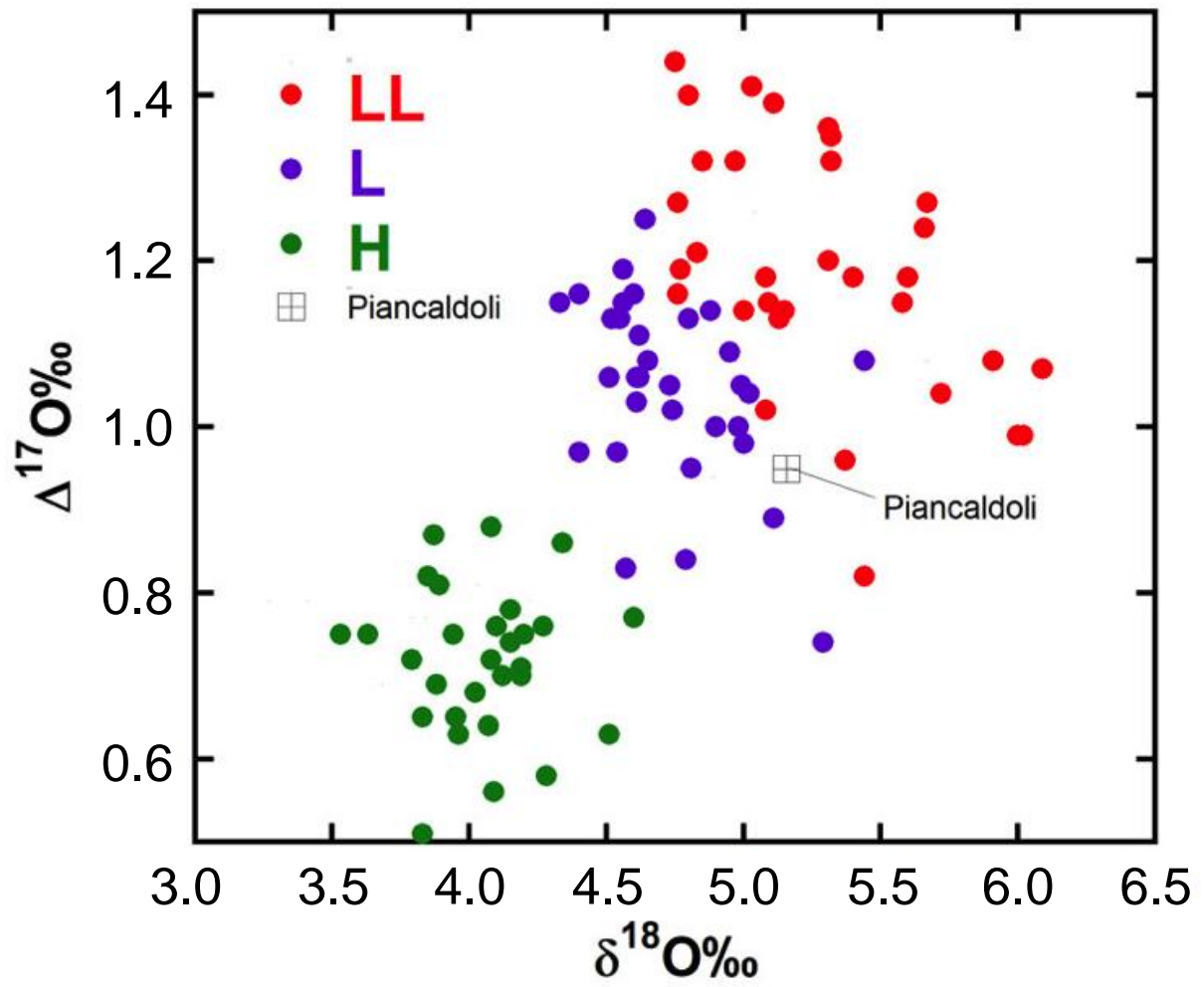
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Fig. 11



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Fig. 12

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