

1 **Testing the ability of the ExoMars 2018 payload to**  
2 **document geological context and potential habitability on**  
3 **Mars**

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131   **Abstract**

132

133         The future ExoMars rover mission (ESA/Roscosmos), to be launched in 2018, will  
134         investigate the habitability of the Martian surface and near subsurface, and search for traces of  
135         past life in the form of textural biosignatures and organic molecules. In support of this  
136         mission, a selection of relevant Mars analogue materials has been characterised and stored in  
137         the International Space Analogue Rockstore (ISAR), hosted in Orléans, France. Two ISAR  
138         samples were analysed by prototypes of the ExoMars rover instruments used for petrographic  
139         study. The objective was to determine whether a full interpretation of the rocks could be  
140         achieved on the basis of the data obtained by the ExoMars visible-IR imager and spectrometer  
141         (MicrOmega), the close-up imager (CLUPI), the drill infrared spectrometer (Ma\_Miss) and  
142         the Raman spectrometer (RLS), first separately then in their entirety. In order to not influence  
143         the initial instrumental interpretation, the samples were sent to the different teams without any  
144         additional information. This first step was called the “Blind Test” phase. The data obtained by  
145         the instruments were then complemented with photography of the relevant outcrops (as would  
146         be available during the ExoMars mission) before being presented to two geologists tasked  
147         with the interpretation. The context data and photography of the outcrops and of the samples  
148         were sufficient for the geologists to identify the rocks. This initial identification was crucial  
149         for the subsequent, iterative interpretation of the spectroscopic data. The data from the  
150         different spectrometers was, thus, cross-calibrated against the photographic interpretations  
151         and against each other. In this way, important mineralogical details, such as evidence of  
152         aqueous alteration of the rocks, provided relevant information concerning potential habitable  
153         conditions. The final conclusion from this test is that, when processed together, the ExoMars  
154         payload instruments produce complementary data allowing reliable interpretation of the

155 geological context and potential for habitable environments. This background information is  
156 fundamental for the analysis and interpretation of organics in the processed Martian rocks.

157

158

159 **1. Introduction**

160

161 The ExoMars rover mission (ESA/Roscosmos) will be launched in 2018 (ESA, 2013).

162 The science objectives of the mission are as follows: 1) to investigate the habitability of the  
163 landing site; 2) to determine whether the materials present are compatible with the  
164 preservation of potential traces of life; and 3) to search for traces of past or present life,  
165 including prebiotic and abiotic organics (ESA, 2013). To achieve these objectives, the mission  
166 will have to investigate the geological diversity of rocks at the landing site. The ExoMars  
167 rover consists of a suite of complementary instruments for observation and analysis. A  
168 panoramic camera PanCam and the close-up imager CLUPI will be used to obtain context,  
169 structural, and textural information from the kilometre- to the sub-millimetre-scale, while the  
170 ISEM (long range infrared spectrometer), mounted on the mast, will determine the target  
171 soil/rock bulk mineralogical composition. A drill will obtain samples down to two meters  
172 depth. The drill is equipped with an IR spectrometer “Mars Multispectral Imager for  
173 Subsurface Studies” (Ma\_Miss; Coradini *et al.*, 2001) for determining down hole mineralogy  
174 (Fig. 1). The samples will be delivered to the internal laboratory where they will be crushed.  
175 The mineralogy will be investigated with the visual and infrared (IR) imaging spectrometer  
176 MicrOmega and the Raman Laser Spectrometer (RLS), which can also detect the presence of  
177 organic matter. More detailed investigation of the organics will be made by the instrument  
178 Mars Organic Molecule Analyser (MOMA), consisting of laser desorption mass spectrometer  
179 (LDMS) and gas chromatograph mass spectrometry).

180

181        In support of this mission, *Bost et al. (2013)* developed a collection of Mars analogue  
182    rocks and minerals collectively known as the International Space Analogue Rockstore (ISAR,  
183    [www.isar.cnrs-orleans.fr](http://www.isar.cnrs-orleans.fr)), stored at the CNRS in Orléans, France. ISAR contains well-  
184    characterised samples (sedimentary, volcanic and magmatic in origin) available for testing  
185    and calibrating space instruments. Currently, several teams use this collection to obtain  
186    scientific reference data on minerals and rocks.

187

188    However, to date, there has been no comprehensive test of the ExoMars geological suite of  
189    instruments using representative Mars analogue samples. Such studies are essential for  
190    adequately preparing future *in situ* investigations and to develop protocols based on the cross-  
191    correlation of the data. In this contribution, we describe a test of the ExoMars geological  
192    instrument suite consisting of two phases. In the first phase, called the “Blind test”, the  
193    ExoMars instrument teams CLUPI, Ma\_Miss, MicrOmega, and RLS were requested by the  
194    ISAR group to perform mission representative measurements on two rocks selected from the  
195    ISAR collection. They were given small sample aliquots without additional context  
196    information and without data from the other instruments. For interpretation, each instrument  
197    team could only rely on its own measurements. In the second phase, the complete set of  
198    ExoMars instrument results, together with aerial images —comparable to Mars orbital  
199    images— and photographs of the outcrops from which the samples had been obtained in the  
200    field—mimicking PanCam photographs—, were submitted to geologists having no previous  
201    knowledge of the rocks used in this exercise. The aim was, first, to identify the rocks, then the  
202    information from each instrument was cross-correlated in order to re-evaluate the initial  
203    interpretation. Finally, the cross-calibrated data were used to fully characterise the samples,

204 not simply in terms of rock type, but also for any features, such as alteration in the presence of  
205 water, that could provide information on potential habitability.

206

207 **2. Methodology used for the Blind Test**

208 This Blind Test was designed to address the geological capabilities of the ExoMars  
209 payload, so a procedure similar to that defined for the ExoMars mission was used.

210

211 2.1 Sample selection and characterisation during the ExoMars mission.

212

213 The characterisation of rocks and the selection of samples during the ExoMars rover  
214 mission will be made using a specific protocol. The panoramic instruments, including  
215 PanCam (including wide-angle and high resolution cameras) and ISEM, will scan and analyse  
216 the panorama and identify potentially interesting targets. This information will be employed  
217 to decide which target to approach for further investigation. PanCam will be used to study  
218 outcrops, rocks, and soils, while detailed images of these materials will be made by the close-  
219 up imager CLUPI —accommodated on an external wall of the drill box—which has several  
220 viewing modes. CLUPI will observe rock textures in macroscopic mode to understand their  
221 nature and characterise potentially visible morphological biosignatures. The synergetic  
222 combination of PanCam and CLUPI will provide powerful, nested imaging capabilities from  
223 the panoramic to the submillimetric scale. This data is important for interpretation of rock  
224 type, mode of formation, and habitability potential, as well as for identifying lithologies that  
225 could potentially preserve traces of past life.

226

227 Samples will be collected with a drill tool hosting the Ma\_Miss instrument (Fig. 1).  
228 Ma\_Miss can perform spectral measurements to identify subsurface minerals as the drill

229 moves in the borehole. PanCam and CLUPI will also be used to study the powdered fines  
230 produced during drilling, as well as the samples collected by the drill at high resolution, prior  
231 to their delivery to the analytical laboratory.

232 Once a sample has reached the analytical laboratory, it is crushed by the Sample  
233 Preparation and Distribution System (SPDS) to a particle size of approximately 250µm and  
234 delivered to a carrousel for IR and Raman spectrometry (MicrOmega and RLS) investigations  
235 and for analysis using the Mars Organic Molecule Analyser (MOMA) laser desorption mass  
236 spectrometry (LDMS) and gas chromatograph mass spectrometer (GCMS).

237

238 The test described here was designed to address the geological capabilities of the  
239 ExoMars payload and not the organic analyses that form part of a separate study.  
240 Furthermore, the PanCam and ISEM systems were not used because the test concentrated on  
241 the *in situ* measurements, although field photographs obtained with a commercial camera  
242 served as substitute PanCam data.

243

## 244 2.2 Sample selection and preparation for the Blind Test

245

246 Two samples from the ISAR collection were chosen for their analogy with Martian  
247 rocks and their pertinence for astrobiology. These samples, labelled “sample A” and “sample  
248 B”, were previously fully characterised in the laboratory using XRD, Raman spectroscopy, IR  
249 spectroscopy, optical microscopy, Scanning Electron Microscopy (SEM) and Electron  
250 Microprobe (EMP) in the framework of the ISAR project (*Bost et al., 2013*). Powdered  
251 samples with a grain size of ~ 250 µm, similar to those provided by the rock crusher in the  
252 ExoMars SPDS (*Lopez-Reyes et al., 2012; Foucher et al., 2013*), were sent to the RLS and  
253 MicrOmega teams, without any images of the original samples. Both samples were sent as

254 hand specimens to the CLUPI team, and as small slabs and powders to the Ma\_Miss team.  
255 The teams only knew that the samples were representative of Martian rocks and that they  
256 could potentially contain biosignatures. It is important to note that, when the Blind Test phase  
257 was carried out (it started in 01/2012), these samples and the associated data were not  
258 available on the ISAR website and the related publication (*Bost et al., 2013*, submitted in  
259 11/2012) had not yet been published. Consequently, neither the instruments teams nor the  
260 geologists had access to information related to the samples before this exercise.

261

### 262 **3. ExoMars instruments used for the blind test**

263

#### 264       3.1. Close Up Imager (CLUPI)

265

266       The CLUPI (Close-Up Imager) is an instrument developed by the Space Exploration  
267 Institute (Space-X) in Neuchatel, Switzerland. It is a miniaturized, low-mass, low-power,  
268 efficient and highly adaptive imaging system, composed of a colour image sensor (2652 x  
269 1768 x 3 pixels), an optics with focus mechanism and processing electronic boards. The  
270 camera is capable of focusing on any target at distances from 10 cm to infinity. The  
271 functionality of z-stacking is also implemented in order to increase the scientific return.

272

273       CLUPI is positioned on the rover's drill box and replaces the geologist's hand lens.  
274 Its scientific objectives during the ExoMars mission are: (1) Geological environment survey:  
275 determination and characterization of surface and rock types present in the immediate  
276 surroundings of the rover, study of the physical properties of the surface (e.g. compaction  
277 state) by inspection of the rover tracks, contribution to the selection of sites for detailed  
278 investigations (drilling); (2) Close-up outcrop observation: study at high resolution (down to 7

279  $\mu\text{m}/\text{px}$  at 10 cm distance) of the texture, structure, and morphology of outcrops, surface rocks  
280 and particles, as well as potential biofabrics, colour variations and possible layering; (3)  
281 Drilling area observation: characterization of site before drilling; (4) Drilling operation  
282 observation: to provide information about the ejected fines as they are produced, potential  
283 colour changes indicative of geological variation with depth, and the mechanical behaviour of  
284 the drilled surface in contact with the drill tip; (5) Drilled core sample observation: to allow  
285 comparison of the extracted sample with the sampling area and visual examination of the  
286 texture and physical properties of the particles; (6) Drill hole observation: provide information  
287 on the surface state after drilling, the amount of ejected fines, their colour, and their physical  
288 properties.

289

290 The two Blind Test samples were imaged using a CLUPI analogue camera (Sigma  
291 SD15) with the same colour image sensor as the real CLUPI (but 20° field of view optics  
292 instead of real CLUPI's 14° FoV). The samples were deposited on a Martian surface analogue  
293 composed of Permian redbeds (red sandstones from Weitenau, southern Germany), and  
294 illuminated with a Sun simulator (Fig. 2a). Images of each sample were acquired from two  
295 rover-representative working distances, 54 cm (real CLUPI equivalent 76 cm) and 25 cm (real  
296 CLUPI equivalent 35 cm), with a pixel resolution of 60  $\mu\text{m}$  and 28  $\mu\text{m}$ , respectively.

297

### 298 3.2 Mars Multispectral Imager for Subsurface Studies (Ma\_Miss)

299

300 The Mars Multispectral Imager for Subsurface Studies (Ma\_Miss) is developed by the  
301 Institute for Space Astrophysics and Planetology in Rome, Italy (*Coradini et al. 2001*).  
302 Ma\_Miss is a miniaturized near-infrared imaging spectrometer in the range 0.4-2.2  $\mu\text{m}$  with  
303 20-nm spectral sampling. It is positioned in the drill tool a few centimeters above the drill tip,

304 where a sapphire window (characterized by high transparency and hardness) protects the  
305 Ma\_Miss optical head, permitting observation of the borehole wall.

306 The Ma\_Miss optical head performs the double task of illuminating the borehole wall  
307 with a spot of approximately 1-mm diameter and, collecting the scattered light coming from a  
308 0.1-mm diameter region of the target, Ma\_Miss can acquire spectral data from the walls of the  
309 drilled borehole. It is also capable of making spectral images of the borehole by using the drill  
310 rotation and translation movements. This instrument can obtain downhole images of the  
311 excavated borehole wall, performing acquisitions at different depths during vertical  
312 translation—in principle, from 0 to 2-m depth. Ma\_Miss can also create so-called ring  
313 images, performing acquisitions during rotation of the drill at a fixed depth. The Ma\_Miss  
314 breadboard used for this test consists of the following subsystems: a 5W illumination lamp, an  
315 optical fiber illumination bundle, an optical head that focuses the light the sample and  
316 recollects the scattered light, a sapphire window (interface between optical head and the  
317 environment) and a collecting optical fibre (Fig. 2b).

318 The Blind Test samples were analysed using a preliminary version of the breadboard  
319 setup, interfaced with a commercial FieldSpec spectrophotometer. Reflectance spectra were  
320 acquired in the spectral range 0.35–1.8  $\mu\text{m}$ . The sample slabs were placed directly on the  
321 sample holder. The plane surface (cut rock surface, representative of an abraded rock surface  
322 and the external surface of the drill-hole) was oriented perpendicularly to the optical axis of  
323 the spectrometer. Several spectra were acquired in different positions on the samples,  
324 simulating the Ma\_Miss stratigraphic column acquisitions.

325

326 3.3 MicrOmega

327

328 MicrOmega consists of a visible light microscope and a near infrared imaging  
329 spectrometer (*Pilorget, 2012; Pilorget and Bibring, 2013*) (Fig. 2c). The instrument acquires  
330 monochromatic images with a high resolution of 20  $\mu\text{m}$  x 20  $\mu\text{m}$  per pixel at wavelengths  
331 between 0.5  $\mu\text{m}$  and 0.9  $\mu\text{m}$  and with a continuous and high spectral sampling from 0.9  $\mu\text{m}$  to  
332 2.5  $\mu\text{m}$  (now up to 3.5  $\mu\text{m}$ ). In this way MicrOmega acquires the entire spectrum in a spectral  
333 domain for each pixel, thus enabling it to identify the composition of the samples at their  
334 grain scale.

335 The Blind Test samples powders were analysed using the MicrOmega breadboard. *In*  
336 *situ* reflectance hyperspectral spectra were acquired on samples 7.4 x 5.9 mm<sup>2</sup> in size with a  
337 spatial sampling of 23  $\mu\text{m}$ . The samples were illuminated by a monochromator with an  
338 Acousto Optical Tunable Filter (AOTF) in the range of 0.9-2.5  $\mu\text{m}$ , oriented about 20° with  
339 respect to the sample surface. The 320 x 256 pixel-infrared detector (MCT Mars SW1  
340 (Sofradir)) is sensitive in the 0.85-2.5  $\mu\text{m}$  spectral region. The focal plane was cooled down to  
341 190°K.

342

### 343 3.4. Raman Laser Spectrometer (RLS)

344

345 The Raman Laser Spectrometer (RLS) is developed at the Associated Unit of the  
346 University of Valladolid-CSIC-Center of Astrobiology (UVa-CAB), in Spain (*Rull et al.,*  
347 *2011a, b*). The RLS is accommodated in the ExoMars rover's Analytical Laboratory Drawer  
348 (ALD). In automatic mode, the RLS can perform raster analysis of at least 20 points (and up  
349 to 40) of the powdered samples, using a 50- $\mu\text{m}$  spot size and an irradiance level of 0.6–1.2  
350 kW/cm<sup>2</sup> with a 532 nm continuous wave, green laser.

351 The powdered Blind Test samples were analyzed using an RLS ExoMars simulator  
352 that includes an SPDS in order to perform measurements under the operation conditions

353 imposed by the rover-based operation (Fig. 2d; *Lopez-Reyes et al., 2014, Rull et al., 2011a*).  
354 This system provides automatic flattening of the powdered samples, autofocus at each  
355 measurement point, and optical images of the samples. In addition, the system autonomously  
356 optimizes the acquisition parameters (integration time, number of accumulations, etc.) at each  
357 spot with the aid of appropriate algorithms (*Lopez-Reyes et al., 2014*). Thirty spots per sample  
358 were acquired in automatic mode. The spectra were pre-processed to remove the baseline and  
359 instrument artifacts. Mineral identification based on specific Raman lines was performed  
360 using the spectral database developed at the University of Valladolid (*Hermosilla, 2012*).

361

#### 362 **4. Results of the Blind Test phase**

363

##### 364        4.1. Sample A

365

###### 366            4.1.1. CLUPI

367

368        CLUPI images were acquired on rough surfaces, as well as on a fresh cut face  
369 (simulating an abraded surface). Note that a similar system is not planned for the ExoMars  
370 rover mission. The external, uncleaned surface of the rock is characterized by alternating  
371 white and grey layers ranging from mm to cm in thickness. The surface of the rock appears to  
372 be a fracture surface and is coated with a whitish-orange-coloured alteration product (Figs.  
373 4a-1 and a-2). The sharp angles observable on the fracture surface suggest that the rock is  
374 brittle. The cleaned surface of the rock provides a better view of the layering (Figs. 3a-3 and  
375 a-4). The layers are generally parallel to each other although the basal layer exhibits gently-  
376 inclined internal laminae and its surface of the latter layer includes some 0.5 to 1 cm-sized,  
377 rounded protrusions having a whitish cortex and a clear, orange-coloured internal component.

378 The laminated nature of the rock suggests a sedimentary origin and the protuberances on the  
379 surface of the lower layer may be either mineral precipitations or detrital inclusions.

380

381           4.1.2 Ma\_Miss

382

383           Spectral images of the cut rock surface were collected using the Ma\_Miss breadboard  
384 and documented alternating dark and light albedo layers suggestive of a sedimentary rock.  
385 Several different spots were acquired on the sample, both on the dark and bright layers,  
386 simulating the stratigraphic column of the borehole. Also the powder was measured. The dark  
387 and bright layers of the slab have corresponding spectra with very similar shapes and  
388 absorption features, although they show different levels of reflectance and spectral contrast.

389           The spectra acquired on both samples (slab and powder) are characterized by the OH<sup>-</sup>  
390 absorption at 1.4 μm (Fig.3b), indicating the presence of a water-containing mineral. On the  
391 slab, the 1.4-μm band is larger and deeper for the high albedo layers than for the dark layers.  
392 This could be due to real differences in the H<sub>2</sub>O or OH content, or just due to a reduced  
393 spectral contrast on the darker region (Fig.3b). A strong negative slope characterizes the  
394 spectra acquired in the bright region; the spectra of the dark layers show a smaller blue slope.  
395 Spectra of both layers show a clear crystal field (C.F.) absorption at 1 μm, whose wings  
396 extend beyond 1.2 μm, likely due to Fe<sup>2+</sup> absorption (*Burns, 1993; Hunt, 1977; Gaffey, 1985*).  
397 The interpretation of this 1-μm absorption is not unambiguous because the iron responsible of  
398 the absorption could be present in silicates, oxides, sulfides, or carbonates. The succession of  
399 bright and dark spectra with very similar spectral shapes is also suggestive of a layered  
400 (sedimentary) structure.

401

402           4.1.3. MicrOmega

403

404       The IR spectrum (Fig.3c) obtained with the MicrOmega breadboard on the sample A  
405 powder shows absorption features at 1.4  $\mu\text{m}$  and 2.2  $\mu\text{m}$ . There is also a very weak absorption  
406 feature at 1.9  $\mu\text{m}$ . These features reflect the presence of  $\text{H}_2\text{O}$  and  $\text{OH}^-$  in the minerals with  
407 which they are associated. The preliminary identification is a match with the spectrum of  
408 kaolinite, a group of white clays (Fe-poor) containing aluminum.

409

410           4.1.4. RLS

411

412       The thirty Raman spectra acquired on the sample powder permit identification of  
413 quartz, anatase, calcite, muscovite and disordered carbonaceous matter (Fig. 3d). The main  
414 rock-forming mineral is quartz.

415

416

417           4.2 Sample B

418

419           4.2.1. CLUPI

420

421       CLUPI photographed the rough and cut surfaces of Sample B. The rough surface is  
422 brownish in color and characterized by a criss-cross network of indentations (Fig. 4a-1 and a-  
423 2). The cut surface shows that the criss-cross network, resembling buff-coloured acicular  
424 structures, infilled veins, cracks, or crystals, continues into the rock (Fig. 5a-3 and a-4). The  
425 brown surface colour and buff-coloured acicular structures are restricted to the outer portion  
426 of the rock, which contrasts with the uniformly grey colour of the internal portion of the rock

427 in which the acicular texture is still faintly visible. This contrast indicates significant  
428 weathering of the outer part of the rock.

429

430           4.2.2 Ma\_Miss

431

432         Both the rough and freshly cut sample surfaces were observed. As in the previous  
433 sample, acquisition of Ma\_Miss spectra at various points on the slabbed sample simulates the  
434 acquisition of data “downhole” in the drill column. Spectra were obtained both in the “bright  
435 region” (where the buff-coloured acicular structures occur) and in the “dark (grey) region” on  
436 the flat, cut rock surface (Fig.4b). The two regions are characterized by the presence of OH;  
437 the 1.4- $\mu\text{m}$  band in the bright region spectra is larger and deeper than the corresponding band  
438 in the dark region. The spectrum of the dark region appears flat without evident absorption  
439 features. The bright region is characterized by a deep absorption near 1.0  $\mu\text{m}$ , due to  $\text{Fe}^{2+}$   
440 (*Burns, 1993*) and by an absorption at 0.7  $\mu\text{m}$ , likely due to  $\text{Fe}^{2+}$ - $\text{Fe}^{3+}$  intervalence charge  
441 transfer (IVCT, although electronic processes due to transition elements such as Ni, Co, Cr,  
442 Fe, Mn, Ti can occur in certain minerals in this region of the spectrum; *Burns, 1993*).

443         The absorption bands of spectra taken in the bright region are suggestive of the  
444 presence of mafic silicates, iron oxides, and hydrates indicative of possibly extensively altered  
445 mafic or ultramafic rocks with a higher concentration of hydrated mineral phases in the  
446 brighter region (the water OH band suggests alteration of silicates). The spectrum acquired on  
447 powder substantially shows the same absorption bands as the rock sample, *i.e.* the iron  
448 electronic transitions at 0.7-1.0  $\mu\text{m}$ , the OH absorption at 1.4  $\mu\text{m}$ .

449

450           4.2.3. MicrOmega

451

452        The IR spectrum obtained from the powdered sample with the MicrOmega breadboard  
453    show absorption features at about 0.97, 1.43, 1.65, 1.88, 1.95, and 23.3  $\mu\text{m}$ . They are  
454    interpreted to reflect the presence of a mineralogical assemblage composed of saponite,  
455    serpentine and forsterite (the magnesium end-member of olivine) (Fig. 4c).

456

#### 457        4.2.4 RLS

458

459        The Raman analysis of the powdered sample B provided generally fluorescent spectra  
460    with weak and very broad bands (Fig. 4d). This is consistent with a very low degree of  
461    crystallinity. The main bands can be assigned to a combination of magnetite and talc. Other  
462    small bands may also be assigned to clay minerals but more precise identification was not  
463    possible. Brucite is not compatible with the observed spectra; antigorite and/or lizardite  
464    (serpentine minerals) have also low probability. The first is characterised by a strong band at  
465     $1041 \text{ cm}^{-1}$  that is not observed, while the bands of the second occur at higher wavenumbers  
466    than observed. However, the presence of a chloritoid cannot be totally ruled out. In some  
467    cases, the main band at  $668 \text{ cm}^{-1}$  shows a shoulder near  $600 \text{ cm}^{-1}$  that is consistent with the  
468    symmetrical chain vibration of chloritoid.

469

470

### 471        **5. Results of the geological interpretation phase**

472

473        Two geologists specialised in geochemistry (C.R.) and petrology (N.L.B.) interpreted  
474    jointly the bulk observational and analytical data for each sample to identify the rock type. In  
475    order to use the same kind of data set as would be available during the future ExoMars rover  
476    mission outcrop images (Figure 5) corresponding to PanCam images were provided to

477 complement the CLUPI images and the Ma\_Miss , MicrOmega and RLS spectra. Outcrops  
478 images were obtained using commercial cameras (Olympus E410 camera, with a 10.00  
479 Megapixel resolution, for sample A and Olympus OM1 camera for sample B).

480 The data interpretation followed a typical strategy, starting with the geological context  
481 (here given by the satellite observation), following by the optical outcrop and sample  
482 observation (here PanCam and CLUPI images) and finishing with the compositional data  
483 (here given by the Ma\_Miss, MicrOmega and RLS spectroscopic data). Although the  
484 identification of the analogue rocks based on the optical images is a routine matter for  
485 geologists, this study underlined the importance of obtaining a maximum of information by  
486 cross-correlating the data in order to improve and/or re-evaluate the interpretation made by  
487 each instrument separately.

488

489

490 5.1. Data interpretation of sample A.

491

492 The outcrop images show that the rock is massive but highly layered (Fig. 5a). It is  
493 thoroughly crosscut by numerous fractures, which suggests that it is hard and brittle. The mm-  
494 to cm-wide layers consist of alternating grey-white to dark blue-grey beds. The upper and  
495 lower boundaries of the beds are generally linear, sometimes wavy, and could be interpreted  
496 as sedimentary features. Some beds are irregular in thickness because of pinching (either due  
497 to tectonic boudinage or sedimentary features?). Some dark centimetric, rounded to angular  
498 features disrupt the bedding. The massive, competent aspect of the outcrop is compatible with  
499 siliceous beds (cherts or quartzites) or marbles (metamorphosed carbonate beds). Evaporites  
500 are doubtful, given the brittle character of the outcrop. The alternation of beds with different  
501 colours in an apparently homogeneously competent material may either be accounted for by

502 different grain size in beds with similar composition, or by mineralogical differences. If the  
503 rocks are siliceous (*i.e.* chert), the darker beds could be finer grained (light would diffuse at  
504 grain boundaries), whereas the lighter levels could be coarser-grained. Another possibility is  
505 that the darker beds display films of carbonaceous matter at grain boundaries, or contain fine  
506 oxide or sulfide grains.

507

508 CLUPI and Ma\_Miss observations confirm the sedimentary origin of the sample (Fig.  
509 3a and b). The grey-white amorphous layer in the lower part of the sample looks like silica  
510 gel, which would support the hypothesis of a chert. The lobated surface of some beds could be  
511 fine sedimentary structures that have been preserved as they were rapidly covered by the  
512 overlying sediment. Although the rock exhibits a massive appearance suggestive of chert, the  
513 clear Fe<sup>2+</sup> absorption seen by Ma\_Miss (and MicrOmega) at 1.0 µm is typical of igneous rock  
514 silicates. This suggests that the rock was originally volcanic in nature. The low albedo of the  
515 dark region may then possibly be due to the presence of vitrified material (such as a glass).  
516 The dichotomy in albedo and spectral characteristics observed in the different spots indicate a  
517 stratified structure typical of sedimentary rocks. Moreover, the presence of OH absorptions  
518 suggests that the volcanic material making up the rock was altered in the presence of water.

519 The detection of kaolinite by MicrOmega (Fig. 3c) is very surprising and does not fit  
520 with the optical observations of the hard, brittle character of the outcropping rock. It is  
521 concluded that, if kaolinite is present in the sample, it is more likely a very minor phase than a  
522 major component of the sample.

523 The RLS data (Fig. 3d) are in more direct accordance with the optical data. In  
524 particular, the detection of quartz as a major constituent fits well with the previous  
525 interpretation of a chert rock type. The presence of carbonaceous matter is also consistent  
526 with the suggestion that the rock is of sedimentary origin, the carbonaceous compounds being

527 more specifically associated with the darker layers. Since calcite is detected in only a few  
528 analyses, it is interpreted as only a minor rock component. The systematic detection of  
529 anatase and the small amounts of muscovite may indicate a detrital volcanic origin of the  
530 sediment in interaction with hydrothermal processes. Water was involved in the formation of  
531 this sediment. The Raman data help to eliminate the hypothesis of a banded. The kaolinite  
532 hypothesized by MicrOmega, the muscovite proposed by RLS, and the hydrated components  
533 identified by Ma\_Miss can all be associated with dioctahedral smectites.

534 Finally, the interpretation that the rock is a banded chert with anatase (common in  
535 chert) and a small amount of dioctahedral mica is the most likely. The lighter beds are  
536 siliceous, whereas the darker ones could contain carbonaceous matter and/or anatase. Quartz  
537 is the main constituent as shown by Raman analyses. Note that quartz cannot be detected by  
538 IR spectroscopy in the spectral range used by MicrOmega and Ma\_Miss . In order to explain  
539 the detection of kaolinite, which is not in accordance with a chert, the IR data interpretation  
540 was revised. It is concluded that the spectrum most probably corresponds to muscovite, in  
541 accordance with the Raman data and consistent with the fact that the spectra of kaolinite and  
542 muscovite are relatively similar in the 0.9 - 2.5  $\mu\text{m}$  spectral range.

543

544 5.2. Data interpretation of sample B.

545

546 The surface of the rocks at the outcrop appears dark green to red, suggesting they are  
547 iron-rich and partly oxidized (Fig. 5b).

548 The outcrop photographs show that the rock is massive and characterised by what  
549 appear to be cracks. Its twisted structure evokes corodate basalt and in this regard, the rugged  
550 surface could correspond to a scoriaceous lava. The sample surface shows a reddish stain

551 (patina?) which is compatible with lava. Such a patina could also characterize a peridotite  
552 (mantle rock) exposed to weathering. The white dots could be calcite or plagioclase.

553 The CLUPI optical observations confirm the red patina on the sample surface, which  
554 evokes a weathered volcanic rock. The acicular texture is characteristic of the spinifex texture  
555 of komatiites (Fig. 4a).

556 The Ma\_Miss spectra are in accordance with the a volcanic origin of the rock (Fig.  
557 4b), *i.e.* clear crystal field absorption due to  $\text{Fe}^{2+}$  at 1.0  $\mu\text{m}$  indicative of iron silicate-bearing  
558 phases of ultramafic/mafic rocks. Moreover, the  $\text{OH}^-$  band suggests mineral alteration,  
559 indicative of possibly extensively altered mafic or ultramafic rocks, with higher a  
560 concentration of hydrated mineral phases in the brighter region (serpentine group minerals,  
561 olivine/pyroxene alteration products, for example antigorite, see *Clark et al., 1990*). The  
562 spectral signals pointing to  $\text{Fe}^{2+}$  -  $\text{Fe}^{3+}$  IVCT transitions at 0.7  $\mu\text{m}$  and to  $\text{Fe}^{2+}$  C.F. transition  
563 at  $\sim$ 1.0  $\mu\text{m}$ , together with the  $\text{OH}^-$  absorption bands are quite consistent with the fact that such  
564 primitive mafic rocks are unstable in the present day oxidised and hydrated surface  
565 environment. Again, the dark colour of the sample is compatible with a volcanic rock (basalt)  
566 or a peridotite. The exposed surface of the sample has in its centre a massive, light-brown  
567 structure whose periphery has a brecciated structure (Fig 5b). The light-brown central  
568 structure is surrounded by a dense network of dark linear structures, which could correspond  
569 to fractures or, more likely, to skeletal olivine crystals in exhibiting spinifex texture.

570 On the fresh cut surface, the rock appears dark and very massive. It is finely grained  
571 and probably basaltic in nature. White dots on the surface, seen also in the outcrop, could be  
572 either plagioclase or calcite. Yellowish greenish, narrow, specular phases occur in the upper  
573 part of the sample. Two interpretations can be proposed:

574 - The surface of the rock may represent a fracture plane along which the rock is  
575 altered. The yellow crystals would then result from the alteration of a mineral such  
576 as olivine along this plane.

577 - The structure on top of the rock represents magmatic layering. The yellow skeletal  
578 crystals are rooted on a planar surface perpendicular to the observed rock section  
579 (a magmatic floor) and grow perpendicularly or obliquely to this plane. This  
580 evokes skeletal crystal growth from a supercooled, layered magma of low  
581 viscosity. Given the probable basaltic nature of the sample, the yellow acicular  
582 crystals are probably olivine forming a spinifex texture.

583

584 The olivine, serpentine and saponite (a trioctahedral smectite) detected by IR  
585 spectroscopy are quite consistent with a mafic rock (e.g. an olivine-bearing basalt) that has  
586 been hydrothermally altered to serpentine and saponite (Fig. 4c).

587

588 In the final analysis, particular aspects, such as outcrop structure, the macroscopic  
589 aspect of the lava, and IR data, favour the interpretation of an olivine-bearing basalt.  
590 Additional features allow the rock to be characterized as ultramafic. These include primary  
591 magmatic features, such as the spinifex texture of a mineral identified as olivine, possible  
592 magmatic layering, and the Mg- and Fe<sup>2+</sup>-rich character of the rock. Moreover, the presence  
593 of serpentine with iron oxidation-related features on the surface of sample B demonstrates  
594 that the rock has been aqueously altered. However, some index minerals of mafic magmas,  
595 such as pyroxene and plagioclase, were not detected by spectroscopy, although the white dots  
596 visible on the rock surface could be plagioclase. Antigorite was detected in the Ma-Miss IR  
597 spectra and perhaps in the Raman spectrum. Magnetite was detected by Raman and is  
598 typically formed during serpentinization (alteration) of mafic rocks.

599

600

601 **6. Full characterization of the samples**

602

603 In this section, we present the complementary analytical data obtained for the two  
604 samples in the framework of the ISAR collection using a large range of laboratory techniques  
605 and laboratory instrumentation. Detailed data are also available on the ISAR website:  
606 [www.isar.cnrs-orleans.fr](http://www.isar.cnrs-orleans.fr).

607

608       6.1 Sample A

609

610 Sample A is the sample 00AU05 of the ISAR collection (*Bost et al., 2013*). It is a  
611 silicified volcanic sediment (chert) from the 3.446 Gy-old Kitty's Gap Chert in the Panorama  
612 formation of the Warrawoona Group, Pilbara craton, Australia (*de Vries et al., 2004; Westall  
613 et al., 2006*). Although its main constituent is now microcrystalline quartz (SiO<sub>2</sub>), optical  
614 microscopy of thin sections of the rock shows that it consists of volcanic clasts that have been  
615 altered to muscovite and anatase and then largely replaced by silica of seawater and  
616 hydrothermal origin (confirmed by  $\mu$ -Raman spectroscopy and mapping). Structures  
617 observable at outcrop scale (layering) and textures observable at the microscopic scale  
618 indicate that the rock represents volcanic sediments that were deposited in a very shallow  
619 marine environment; such as an infilling tidal channel (*de Vries et al., 2004*). The traces of  
620 carbonaceous matter identified by Raman are related to the presence of fossilized (silicified)  
621 microbial colonies (*Westall et al., 2006; Westall et al., 2011*). Concentrated on the surfaces of  
622 volcanic grains and in the pore spaces between the volcanic grains, these colonies most likely  
623 represent relatively simple microorganisms, such as chemolithotrophs that obtain their energy,

624 nutrients and carbon from inorganic sources. This sample is, thus, particularly relevant in  
625 terms of the search for life on Mars since these volcanoclastic sediments were deposited in a  
626 shallow water aqueous environmental setting that would have been relatively common in the  
627 Noachian period on Mars. The simple, chemotrophic life forms that they contain could  
628 therefore hypothetically reflect the kinds of simple life that may have occurred on Noachian  
629 Mars (*Westall et al. 2011; Westall et al., 2013*). Moreover, during the Noachian,  
630 hydrothermal processes associated with impacts and volcanic activity were likely to have  
631 been important on Mars (e.g. *Schwenzer and Kring, 2009*), and the precipitation of silica and  
632 subsequent silicification of igneous and sedimentary rocks and any life forms that might be  
633 associated is therefore possible. Silica has only recently been detected on Mars (*Bish et al.*  
634 *2013; Blake et al., 2013*), possibly due to technical limitations since quartz has no IR signal in  
635 the spectral range used for Martian exploration.

636

637       6.2 Sample B

638

639       Sample B is the sample 10ZA09 of the ISAR collection (*Bost et al., 2013*). It is a  
640 weathered komatiite from the type locality on the Komati River in the Barberton Greenstone  
641 belt, in South Africa (*Bost et al., 2013*). The main constituents are olivine, antigorite, micas  
642 and clays, as well as traces of hematite, magnetite and talc. Some trace of carbon  
643 (carbonaceous matter) is also observed in this sample. Volcanic rocks, in particular basalts,  
644 are very common on the martian surface (e.g. *McSween et al. 2009*). Although, they are richer  
645 in Fe and Mg than present-day terrestrial volcanics, many volcanic rocks dating back to the  
646 Archaean epoch were also richer in Fe and Mg, especially the komatiites. The possible  
647 presence of komatiite-like rocks from the Noachian epoch on Mars has been evoked by *Nna-*  
648 *Mvondo and Martinez-Frias (2007)*. It is also interesting to note that a recent experiment to

649 produce artificial basalts with a martian composition surprisingly created spinifex-like  
650 textures (*Bost et al., 2012; Chevrel et al., 2013*).

651

652 **7. Discussion and conclusions**

653

654 The results collected during the Blind Test are compared to the ISAR data in Table 1.  
655 There are only a few differences between the analyses made by the Exomars breadboard  
656 instruments (and a CLUPI-like camera) and those made by standard laboratory instruments.  
657 For sample A, calcite was detected by the RLS while it was not observed during the  
658 characterization made for the ISAR collection (although *Orberger et al. (2006)* previously  
659 detected traces of Ca-Mg-carbonates in this sample). This is due to the large area of analysis  
660 (50 µm) of the RLS compared to spot analyses made by laboratory instruments. On the other  
661 hand, goethite and rutile, present in the sample, were not detected by the ExoMars  
662 instruments. For sample B, the ExoMars instruments did not detect phlogopite, hematite and  
663 dolomite.

664

665 Although a trained geologist can identify rock type from observation, it is clear that  
666 cross-correlation between data from different instruments, both observational and analytical,  
667 is essential to fully characterise unknown rock types, as demonstrated by this study. The  
668 iterative approach documented here, refining initial observational and analytical  
669 interpretations through comparison with data obtained by other methods, demonstrates the  
670 force of this interactive process and the complementarity of the ExoMars geological  
671 instrument suite.

672

673 This study thus confirms the ability of the ExoMars geological instruments to carry  
674 out high quality analyses. The panoramic (field camera) and smaller-scale (CLUPI) images of  
675 the geological context provided by the cameras and the mineralogical information obtained  
676 with the RLS, Ma\_Miss and MicrOmega instruments are each necessary and suitably  
677 complementary. The trained geologists were able to determine rock type from the variety of  
678 details obtained from orbit and from the field/hand specimen images. This preliminary  
679 identification was very helpful for interpreting the spectral data. The cross-calibrated spectral  
680 data were essential for the subsequent mineralogical interpretation (Table 2), in particular for  
681 determining the presence of water-bearing mineral species, important for understanding  
682 deposition/weathering/alteration signatures that have a bearing on microbial-scale habitability  
683 and the potential for preserving past traces of life.

684 These results allow a number of important conclusions to be drawn for future Mars and  
685 general planetary *in situ* missions:

- 686 1. Cross-correlation of data obtained with a complementary suite of observational and  
687 analytical instruments, evaluated by trained geologists is essential for the full  
688 characterisation of the rocks.
- 689 2. While the use of pure minerals for space instrument calibration is useful during the  
690 development phase of the instruments, preparation for an *in situ* mission using a suite  
691 of complementary instruments requires cross-testing with suitable analogue rocks  
692 exhibiting heterogeneous structures, textures, and mineralogy.
- 693 3. Interpretation of the data is best made by the multidisciplinary mission team, including  
694 geologists, spectroscopists, geochemists, and engineers (evaluation of the microbial-  
695 scale habitability and eventual biosignatures needs also to include relevant expertise).

696

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698

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704

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797 **Figures caption:**

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799 **Figure 1:** Sketch of the ExoMars rover and the instruments locations. Credit: ESA.

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801 **Figure 2:** (a) CLUPI analogue lab setup. (b) Ma\_Miss breadboard setup photograph. (c)  
802 MicrOmega breadboard setup photograph (modified after *Pilorget and Bibring, 2013*) (d)  
803 RLS ExoMars simulator.

804

805 **Figure 3: Sample A analyses.** (a) CLUPI images of the rough surface and cut face from 54  
806 cm (real CLUPI equivalent 76 cm) (*I* and *2*) and 25 cm (real CLUPI equivalent 35 cm) (*3* and  
807 *4*). The scale bar is 2cm. (b) Ma\_Miss image of the cut slab and VNIR reflectance spectra  
808 acquired on the slab in the dark lower albedo layers (red and blue) and in the bright higher  
809 albedo layers (black, cyan and pink). The colored dots give the positions of spot analyses on  
810 the rock. Spectra have been shifted along the Y-axis for clarity. This series of acquisitions  
811 simulated the analysis of a stratigraphic column. (c) MicrOmega IR spectrum of the powder.  
812 (d) RLS Raman spectra of the powdered sample with mineralogical assignation.

813

814 **Figure 4: Sample B analyses.** (a) CLUPI images of the rough surface and cut face from 76  
815 cm working distance (*i* and *ii* respectively) and at 35 cm working distance (*iii* and *iv*). The  
816 scale bar is 2cm. (b) Ma\_Miss image of the slab and VNIR reflectance spectra acquired on the  
817 slab in the dark lower albedo layers (red) and in the bright, higher albedo layers (containing  
818 buff-colored acicular features) (black and green). The colour of the spectral lines corresponds  
819 to locations marked with the same colour on the rock surface. (c) MicrOmega IR spectrum of  
820 the powder. (d) RLS Raman spectra of the powder with Raman mineralogical assignation.

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822 **Figure 5:** Outcrop photographs of samples A and sample B (Fig. 5a and 5b, respectively)

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