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A Portable Micro-SORS Prototype for Cultural Heritage: New Capabilities and Advantages

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Abstract: The combination of Spatially Offset Raman Spectroscopy (SORS) with microscopy has led to the development of micro-SORS, a technique enabling non-invasive analysis of subsurface layers at the microscale, specifically optimized for Heritage Science applications. Here, we present the capabilities and advantages of a recently developed advanced portable micro-SORS prototype, designed to overcome the main limitations of current commercial and custom-built systems. The prototype offers high sensitivity, wide spectral window ($100\text{--}3050\text{ cm}^{-1}$) and a simultaneous acquisition of multiple offset spectra via a custom fibre bundle, which also reduces mechanical vibrations. Moreover, an integrated digital microscope allows for an accurate point selection during in-situ analysis of artworks. The system was tested during two analytical campaigns within the MOLAB platform at the Italian node of E-RIHS-ERIC (European Research Infrastructure for Heritage Science): the study of Andrea Solario's paintings at the Museo Poldi Pezzoli (Milan) and the Book of the Dead of Kha at the Museo Egizio (Turin). The results demonstrate four key capabilities: (i) non-invasive identification of pigments with diagnostic Raman bands in the low-frequency region (e.g., lead-tin yellow type I, anatase); (ii) detection of ground layers, revealing insights into painting techniques; (iii) reconstruction of stratigraphies, including the identification of pigment layering in the papyrus; and (iv) characterization of surface heterogeneity, such as elemental sulphur crystallization linked to arsenic sulphides degradation. These findings confirm the prototype potential as a powerful tool for heritage science, enabling high-quality, depth-resolved Raman analysis directly on artworks. Limitations observed during one year of field use are also reported, highlighting opportunities for future developments.

Keywords: raman spectroscopy; micro-SORS; portable non-invasive in-situ analysis; heritage science

1. Introduction

Scientific interest in the development of portable and handheld Raman spectrometers, and their potential applications in the field of cultural heritage, started in the early 2000s [1,2] and has been steadily increasing ever



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since. From then on, nearly every year reviews or critical assessments have been published, focusing either on technological performance or on novel in-situ applications [3–15]. Most of these studies have involved commercially available spectrometers, although some custom-built devices have also been developed, offering greater flexibility and tailored features.

Over the past decade, a significant methodological advancement has emerged with the integration of Spatially Offset Raman Spectroscopy (SORS) and microscopy, resulting in a new technique known as micro-Spatially Offset Raman Spectroscopy (micro-SORS) [16]. This method is particularly noteworthy for its ability to non-invasively collect Raman signals generated from microscale depths underneath the surface. In heritage science, micro-SORS has been successfully applied to the reconstruction of painting stratigraphies [17–19], the study and monitoring of diffusion processes, such as pigment degradation and the penetration of conservation materials [20,21], and the detection of hidden texts or figures in sealed historical documents [22,23].

The technique was quickly adapted to portable instrumentation. The initial implementation involved the simplest variant of micro-SORS, known as the defocusing approach, applied to a commercial portable Raman spectrometer [24]. Only minor modifications were required, such as replacing the standard lens with a microscope objective and adding a microstage to control defocusing distances. A year later, the same commercial spectrometer was adapted to support the more advanced variant of the technique, referred to as full micro-SORS [25]. This required more substantial modifications and optimizations of existing Raman systems: the laser beam had to be decoupled from the Raman collection path using a dichroic beamsplitter and directed into a microscope objective mounted on a motorized stage to control the spatial offset; the Raman collection path was similarly equipped with a microscope objective. In the same year, Vandenabeele et al. [26] developed a custom-built sensor that used bare optical fibres to deliver the laser to the sample and to collect Raman signals from a spatially offset region, transferring the signal to the spectrometer.

Despite these advances, current systems still face several limitations. These include restricted versatility and sensitivity due to their dependence on commercial spectrometer platforms, limited spectral window, absence of a camera for selecting the point measurements, low accuracy in setting spatial offsets and, perhaps most critically, the need for mechanical movements to adjust either spatial offsets or defocusing distances, which inevitably introduce unwanted vibrations during in-situ analysis and delays the measurement.

For these reasons, an advanced in-house portable micro-SORS prototype was developed at the CNR-ISPC Raman Lab [27] a few years after the first basic prototypes, with the goal of creating a tool featuring the following capabilities: spectrometer and CCD performance comparable to benchtop instruments; a lower spectral range than that of most commercial portable spectrometers; an optimal configuration for micro-SORS measurements (utilizing spatial offset instead of defocusing distances); suitable spatial resolution for extracting depth information; presence of a camera for guiding the selection of the measurements points; absence of mechanical movements for setting spatial offsets.

Following the initial promising laboratory tests, validating and optimizing the prototype through in-situ studies was essential. This provided an opportunity to test the instrument in real world scenarios guiding further its optimisation and refinement. The MOLAB platform at the Italian node of E-RIHS-ERIC (European Research Infrastructure for Heritage Science) offered the ideal framework for this purpose. MOLAB provides access to an integrated set of non-invasive and portable instruments for the in-situ investigation of artworks. Research projects are selected based on the quality of proposals, and the infrastructure supports these studies by leveraging the expertise of conservation scientists and advanced portable technologies to conduct non-invasive analyses on-site. This approach enables cross-comparative studies and fosters the use of several methodologies, exploiting the synergy among different methods so that the strengths of each technique are maximized and their limitations mitigated by complementary approaches. Through our participation as a provider in MOLAB, we had the opportunity to test, validate, and refine our prototype while contributing significantly to the understanding and preservation of the studied artworks.

After one year of fieldwork, we are able to demonstrate the advantages of our system compared to conventional instruments. Specifically, here we highlight improvements in four key areas:

1. Non-invasive identification of pigments with diagnostic Raman bands in the lower spectral range.
2. Non-invasive identification of ground layers.
3. Non-invasive reconstruction of painting stratigraphy.
4. Characterization of surface compositional heterogeneity.

A selection of representative measurements carried out on Andrea Solario's paintings at the Museo Poldi Pezzoli (Milan, Italy) and on the Book of the Dead of Kha at the Museo Egizio (Turin, Italy) is presented here.

2. Case Studies

In February 2024, an analytical campaign was launched at the Museo Egizio in Turin (Italy) focusing on the Book of the Dead of the architect Kha (Figure 1a). Kha was responsible for the works in the royal necropolis during the Eighteenth Dynasty, under the reign of Amenhotep III (1390–1353 BC). His tomb (Theban Tomb 8, Deir el-Medina, Luxor) was discovered in 1906 by the Italian Egyptologist Ernesto Schiaparelli and is remarkably well-preserved, containing personal belongings such as tools, furniture, and, among many other objects, this papyrus [28]. Measuring ca. 14 m in length, it is one of the best-preserved New Kingdom papyri ever found. Rich in formulae and spells intended to guide the deceased through the afterlife, it provides unique insights into the funerary culture of the period.

A second analytical campaign in November 2024 was carried out at the Museo Poldi Pezzoli in Milan (Figure 1b), dedicated to the study of Andrea Solario's paintings. Solario, an Italian Renaissance painter and contemporary of Leonardo da Vinci, is renowned for blending Venetian, Milanese, and Flemish influences. His works are characterized by religious themes and, in his late period, elements inspired by Leonardo's sfumato, the delicate transitions between light and shadow. Various advanced techniques were employed to analyse a selection of his paintings, with the aim of investigating his preferred palette and painting methods. 7 paintings have been studied: *Madonna with Child*, *Madonna nursing the Child*, *Rest on the Flight into Egypt*, *Ecce Homo*, *Saint Catherine of Alexandria and Saint John the Baptist*, *Saint John the Baptist*, *Saint Anthony Abbot* [29].

The following sections present selected results from both campaigns, demonstrating the potential of our portable prototype for in-situ heritage analysis.

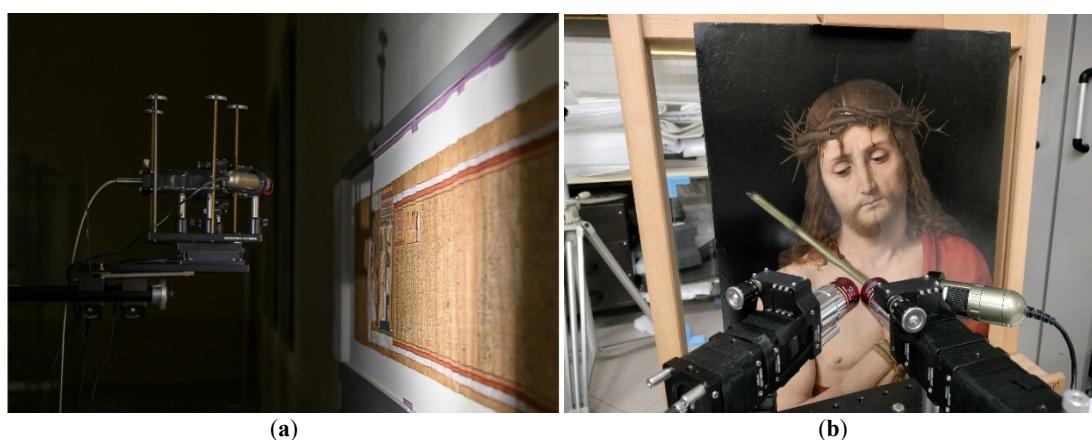


Figure 1. Portable micro SORS set-up implemented during the MOLAB analytical campaign at the Museo Egizio (a) and Museo Poldi Pezzoli (b).

3. In House Micro-SORS Portable Prototype

The portable prototype is described in detail elsewhere [27]. Here, we provide a brief overview.

WP785 spectrometer (Wasatch Photonics), coupled with an iDus DU4011A-BR-DD CCD (Andor) and a wavelength-stabilized 785 nm diode laser from Innovative Photonic Solutions (IPS) are used. Spectral resolution was 8 cm^{-1} . Two long working distance microscope objectives M-PLAN Apo NIR 10 \times (Mitutoyo; NA 0.26–WD 30.5 mm) are used as excitation and collection optics. The laser excitation microscope objective is positioned at an angle of approximately 45° with respect to the sample plane. The laser is delivered to the excitation objective by an optical fibre with a 50 μm core diameter (0.22 NA) collimated by a 12 mm lens (0.25 NA). A digital microscope (DinoLite) is used to observe and select the portion of the sample to be measured, and a micro-stage (Thorlabs M30XY) is positioned under the optical system enabling the sample alignment and focusing operations with accuracy of 8 μm .

The main innovative aspect resides in the utilization of a customized fibre bundle (Armadillo SIA–LV) to collect Raman photons from the sample: the bundle comprises 30 in-line fibres at the micrometre scale; by shining the laser between the 15th and 16th fibre, it is possible to simultaneously collect in-focus and offset spectra. The CCD is split in 30 different portions, so that the final read-out is able to provide a different spectrum for each collection fibre. A LabVIEW program was developed to control and analyse the measurements, taking the full CCD image and slicing it in spectra divided by offset. In this way, the necessity of moving the instrument to set an offset is removed, effectively diminishing vibrations during the measurements. Only movements along the Z-axis of the micrometric stage are required to achieve precise focus alignment between the laser spot and the fibre bundle.

Another novel aspect concerns the spectrometer's spectral range ($100\text{--}3050\text{ cm}^{-1}$). This increased spectral range enables the detection of numerous compounds with distinct Raman signatures in the low-frequency region, such as certain pigments and minerals.

The optical components (microscope objectives, camera, and micrometric stage) can be mounted on a tripod, enabling Y-axis movement, which is essential for selecting multiple measurement positions. Nonetheless, when the artwork can be moved along the Y-axis, placing the optical assembly on a stable table in front of the object is preferable to avoid the minor vibrations introduced by the tripod.

Since the prototype is not a compact system, the ambient light interference can be an issue in many situations. To mitigate this, the optical section is covered with a custom-designed black plastic box; when necessary, an additional black cloth (Thorlabs) is used to further obscure the spectrometer and CCD.

For the experimental data acquired during the campaigns, the excitation power was in a range from 10 to 18 mW at the sample. The integration time was in the range between 15 and 60 s. The spectra were processed using OPUS software (Bruker); a meaningful spectral range was selected, according to the main bands of the compounds, and a baseline correction was performed.

4. Results

4.1. Non-Invasive Identification of Pigments with Diagnostic Bands in the Lower Spectral Region

The characterization of flesh tones in seven paintings by Solario was a key focus of this study, aimed at gaining insights into the artist's palette and painting technique. The main finding provided by the prototype is that Solario's preferred mixture for flesh tones consists of vermilion, lead white, and lead–tin yellow Type I. In historical painting techniques, two yellow pigments based on tin and lead are documented: Type I, a lead stannate (Pb_2SnO_4), and Type II, with a warmer hue, a lead–tin silicate [$\text{Pb}(\text{Sn},\text{SiO}_3)$]. Lead–tin yellow Type I pigment is particularly challenging to identify *in situ* because of the cutoff of portable Raman instruments; in fact, its unique characteristic band, around 129 cm^{-1} , typically coincides with the standard cutoff region of commercially available portable Raman spectrometers. However, the extended spectral window of our instrument enabled the detection of compounds with diagnostic bands at very low wavenumbers, down to 100 cm^{-1} (Figure 2a,b).

It is worth noting that, in this campaign, no other analytical technique has unequivocally confirmed the presence of this pigment. While X-ray fluorescence occasionally detects lead and tin, this alone is insufficient for definitive identification, and in some paintings, the pigment is present at such low concentrations that no other analytical technique can detect it. In contrast, the high spatial resolution of Raman spectroscopy, combined with imaging capabilities, consistently allowed us to target and identify the pigment. Lead–tin yellow Type I was found in five of the seven paintings; thus, its absence in *Rest on the Flight into Egypt* and *Saint John the Baptist* could hold significant art-historical implications.

Interestingly, lead–tin yellow Type I was also identified in other areas beyond the flesh tones (Figure 2c,d): in the yellow hues of St. Catherine's mantle, the light shades of the green staff in *Ecce Homo*, the blond hair of Jesus in *Madonna nursing the Child*, the orange drapery of Mary's mantle in *Madonna with Child*, and the pale green drapery of St. Joseph's robe in *Rest on the Flight into Egypt*. This widespread use highlights the artist's skill in selecting pigments to achieve the desired chromatic effects.

Another significant finding highlighting the prototype's capability to operate in the low spectral range is the detection of titanium dioxide, in the form of anatase, within one of St. Catherine's flesh tones (Figure 3a,b). Anatase exhibits its primary characteristic band at $142\text{--}143\text{ cm}^{-1}$, which falls outside the range accessible to instruments with a limited spectral window. As with lead–tin yellow type I, detecting its other Raman bands is challenging due to their intrinsically low intensity and the high background signal typically encountered in *in-situ* Raman spectra, where ambient light and various interferences compromise spectral quality. The detection of titanium dioxide is particularly significant from a conservation perspective, as this pigment was introduced into art only after 1920 [30], unequivocally indicating that this painted area is not original but a quite recent overpainting. This finding is consistent with the presence of other modern pigments in adjacent areas, such as chrome orange and phthalocyanine green. In Figure 3a the presence of rutile, the other titanium dioxide polymorph, cannot be excluded.

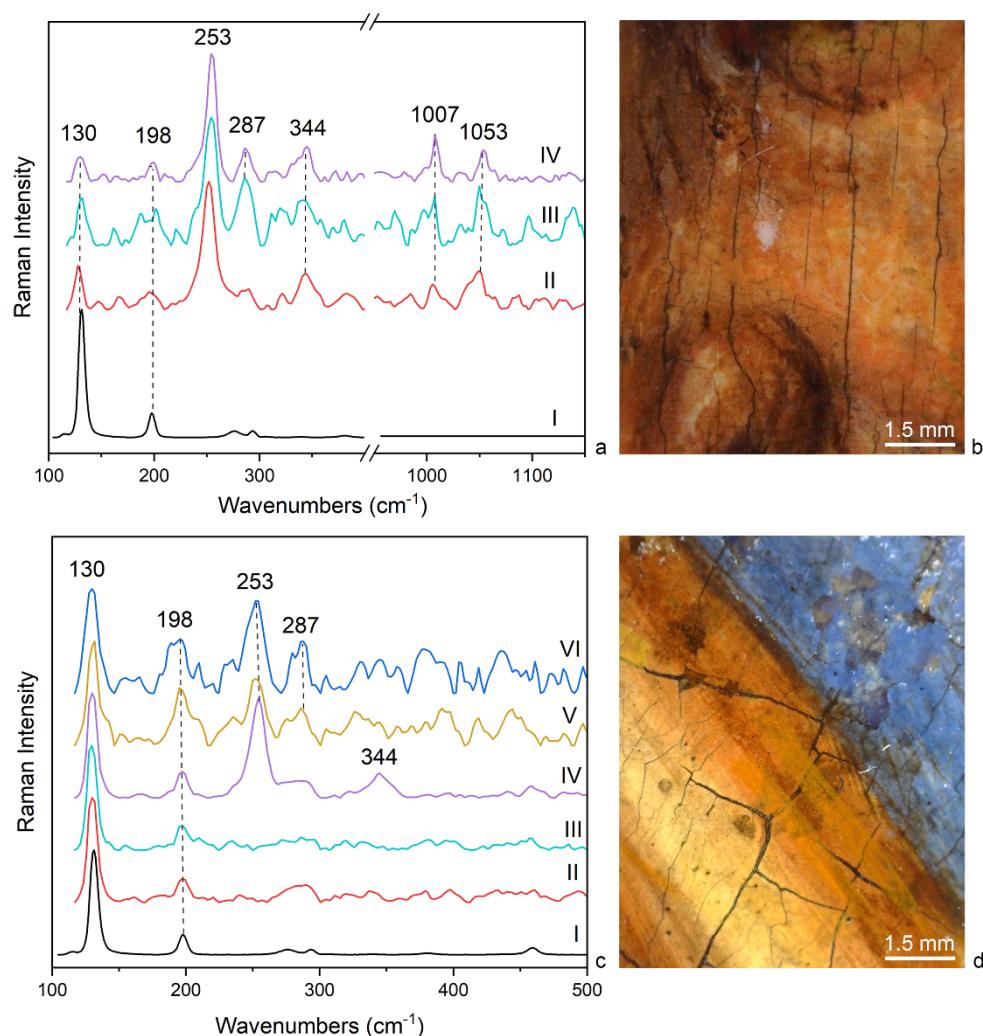


Figure 2. (a) Reference Raman spectrum of lead–tin yellow type I (I), and Raman spectra collected on the flesh tones of *Rest on the Flight into Egypt* (II), *Ecce Homo* (III), and *Saint Anthony Abbot* (IV). Along with lead–tin yellow, the spectra reveal vermillion (253 and 344 cm^{-1}), ochre (287 cm^{-1}), gypsum (1007 cm^{-1} , from the underlying preparation layer), and lead white (1053 cm^{-1}). (b) Detail of the flesh tones in *Saint Anthony Abbot*. (c) Reference Raman spectrum of lead–tin yellow type I (I), and Raman spectra collected on the light green shades of the staff in *Ecce Homo* (II), the drapery of St. Joseph's robe in *Rest on the Flight into Egypt* (III), the yellow hues of St. Catherine's mantle (IV), the drapery of Mary's mantle in *Madonna with Child* (V), and the hair of Jesus in *Madonna Nursing the Child* (VI). (d) Detail of Mary's mantle in *Madonna with Child*. The locations of all the measurements carried out in the Solario's paintings with our prototype are shown in Figure S1.

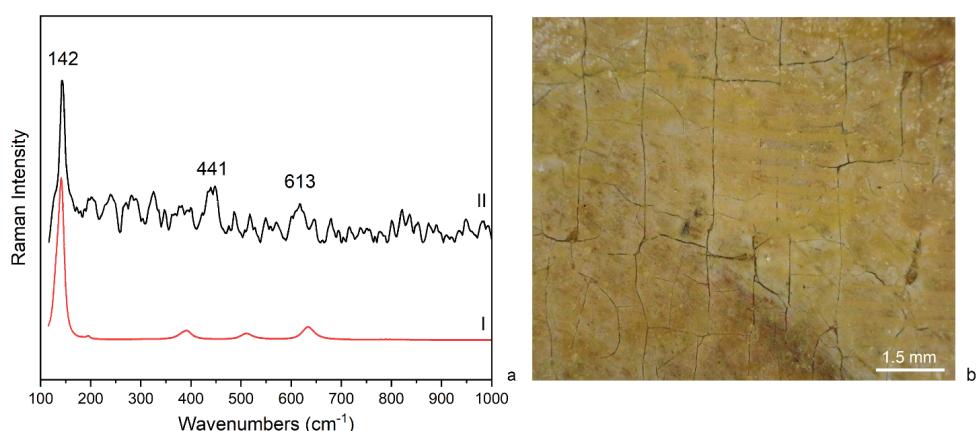


Figure 3. (a) Reference Raman spectrum of anatase (I) compared with Raman spectrum collected on the hand of Saint Catherine (*Saint Catherine of Alexandria*) (II); (b) detail of the retouched hand of the Saint.

4.2. Non-Invasive Identification of Ground Layers

The identification of the ground layer is achievable in a fully non-invasive manner using micro-SORS. In the case of Solario, the prototype successfully identified multiple areas where the emergence of the most characteristic gypsum signature from the subsurface was unequivocal. This is clearly visible in the micro-SORS series (Figure 4): after normalization to the gypsum Raman band, the lead white signal decreases with increasing offset, indicating the presence of a gypsum layer underneath the painting surface. If these compounds were mixed, this trend would not be present, as both would vary in the same way with increasing offset.

The detection of lead white on the surface provided valuable insight into the artist's technique, namely the use of lead white mixed with pigments for the surface layers, and gypsum as the preparation layer underneath.

Interestingly, the gypsum band does not increase with the offset in three paintings: *Saint Catherine of Alexandria*, *Saint John the Baptist*, and *Madonna with Child*. Both *St. Catherine* and *St. John the Baptist* were transferred from their original wooden panel supports to canvas; this intervention may have partially or completely altered the ground layer, as suggested by the micro-SORS data.

In the case of *Madonna with Child*, portable microscope images revealed significant delamination of the painting layer, resulting in evident lacunae. The painting is known for its compromised state of preservation, despite a recent restoration, and was therefore initially attributed to the artist with some uncertainty [29]. The absence or poor presence of a ground layer, as indicated by micro-SORS, may be related to this complex conservation history.

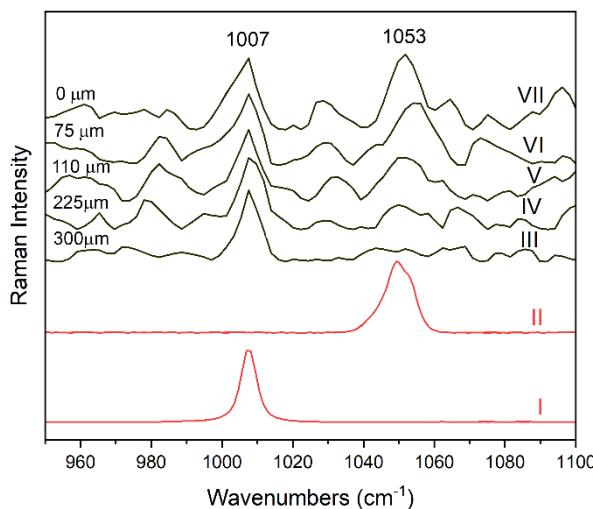


Figure 4. Reference Raman spectra of gypsum (I) and lead white (II), and micro-SORS sequence collected on the flesh tones of *Ecce Homo* (III–VII) that displays the progressive relative decrease of lead white intensity compared to gypsum with the increase of the spatial offset (0–300 μ m). The spectra are normalized to the Raman band of gypsum at 1007 cm^{-1} .

4.3. Non-Invasive Reconstruction of Painting Stratigraphy

The unique contribution of Raman spectroscopy to the study of the Kha papyrus lies in its ability to distinctly identify arsenic sulfide pigments, orpiment (α -As₂S₃) and realgar (As₄S₄), and their primary degradation products, arsenolite (As₂O₃) and pararealgar (As₄S₄), respectively. No other technique, except for X-Ray diffraction, offers comparable sensitivity to these differences, as the compounds share a similar chemical composition but differ in mineralogical structure. In artifacts of this kind, yellow, red, and orange areas are typically painted with either ochres or arsenic sulfide pigments. The advantage of Raman also over X-Ray diffraction is the strong Raman scattering cross section of these compounds, so that their identification is straightforward. Consequently, the initial approach to the papyrus focused on analysing these coloured areas, guided by the results obtained with other techniques, particularly X-ray fluorescence mapping.

The distinctive contribution of our prototype is exemplified by the yellow solar discs placed above the frieze of uraei in the upper section of the papyrus (Figure 5b). Multiple micro-SORS sequences were acquired for each disc, providing a robust statistical basis essential for analysing heterogeneous artworks. In the zero-offset spectrum of the micro-SORS series, equivalent to the one obtained with conventional Raman, signals from both orpiment and realgar are detected (Figure 5a). Without the depth-resolving capability of micro-SORS, it would be impossible to determine whether these pigments are surface mixtures or arranged in different layers. As the offset

distance increases, the realgar signal from the subsurface gradually intensifies, revealing a stratigraphy: the yellow discs, composed of orpiment, were painted over a red layer of realgar.

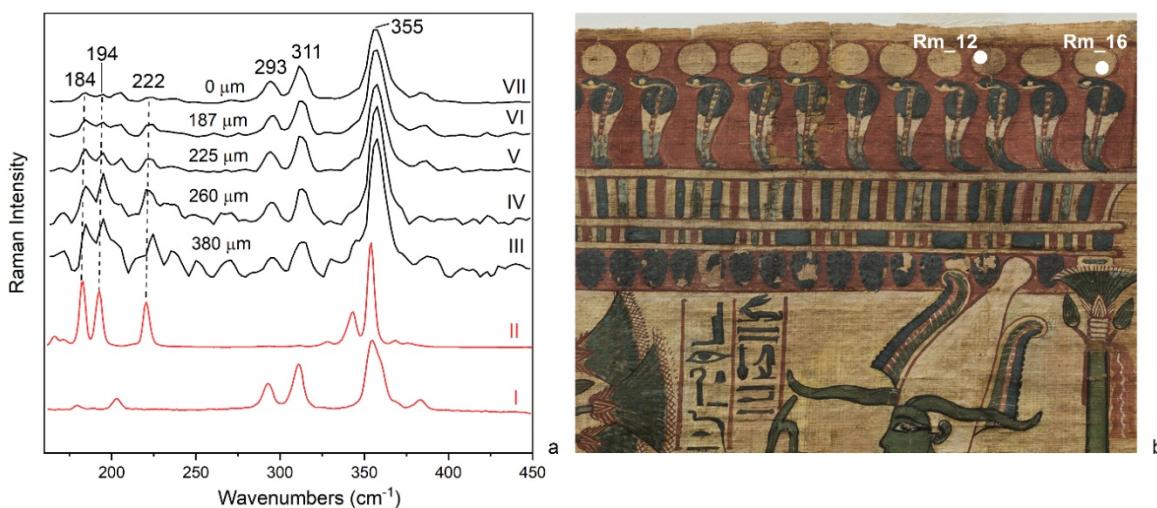


Figure 5. (a) Reference Raman spectra of orpiment (I) and realgar (II), together with the micro-SORS sequence acquired on the papyrus (III–VII) in Rm_12 area. The sequence shows a progressive relative intensity increase of the realgar contribution relative to orpiment with increasing spatial offset (0–380 μm). All spectra are normalized to the Raman band of orpiment at 311 cm^{-1} . (b) Papyrus detail of the frieze with uraei and solar discs, indicating the micro-SORS acquisition areas. The location of all the measurements carried out in the papyrus with our prototype are shown in Figure S2.

4.4. Characterization of Surface Heterogeneity

Interestingly, some of the micro-SORS sequences acquired in the yellow suns showed a different pattern. In addition to the orpiment and realgar sequence, further analytical evidence emerged from some offset spectra (Figure 6): some bands centered at 155, 221, 475 cm^{-1} appeared and disappeared almost suddenly in some of the offset spectra. They did not appear regularly, and they did not follow a gradual increment or decrement with the offset as in the previous case. Excluding the known arsenic sulfide compounds, wider research has been carried out, leading to the unexpected identification of elemental sulphur. Due to the absence of graduality in the emergence and disappearance of the signal, micro-SORS suggests that elemental sulphur is randomly present on the surface of the papyrus, highlighting a strong heterogeneity from the compositional point of view. Moreover, the papyrus exhibits a glittering effect visible to the naked eye, characterized by small, shiny particles; it cannot be ruled out that at least part of this glittering effect results from the crystallization of elemental sulphur on the surface.

It is reported in literature that elemental sulphur originates from the degradation of crystalline orpiment that, when exposed to specific environmental conditions, triggers a mechanism leading to the release of arsenic and sulphur [31]. Therefore, we concluded that the presence of this compound is likely unintentional and resulted from a degradation process.

The extensive in-situ studies also identified some limitations of the new technology. First, the system requires considerable time to align the optical components, specifically, to focus the laser spot on the surface so that it is aligned with the optical fibre bundle, which in turn must be aligned with the spectrometer. This adjustment is necessary not only during the initial setup but also repeatedly throughout the measurements to maintain proper alignment for the entire duration of the analysis. Moreover, the present magnification level is too low to resolve surface layers just a few microns thick, such as those produced by specific degradation mechanisms.

In some cases, the use of a black cover was insufficient to mitigate ambient light interference. To address these issues, the next steps include integrating advanced techniques, such as SERDS (Surface Enhanced Raman Difference Spectroscopy), already tested in the prototype on a trial basis [27] and charge-shifting, previously validated on a benchtop SORS instrument [32]. These enhancements aim to create a single portable device capable of non-invasive subsurface analysis in broad daylight and in the presence of fluorescence.

Additionally, enabling micro-SORS mapping will enable the extension of the analysis to larger areas and allow automated acquisitions. The development of advanced in-field data analysis routines is also among our objectives, significantly improving the speed and reliability of data processing.

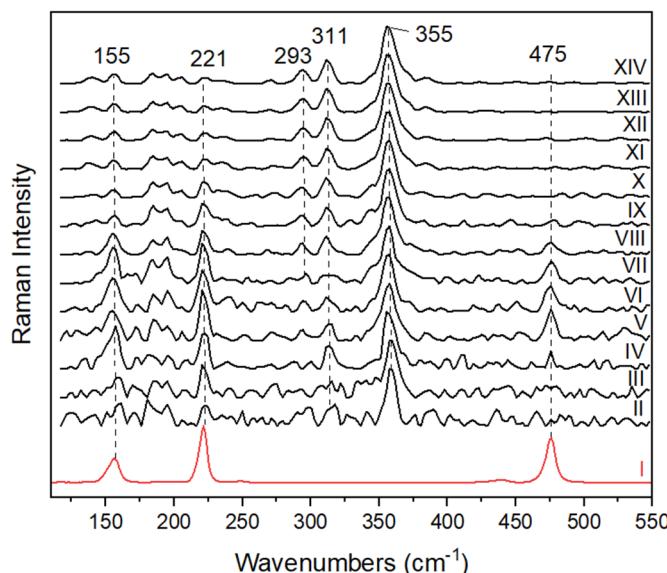


Figure 6. Reference Raman spectrum of elemental sulphur (I) and micro-SORS sequence acquired on a yellow area of the papyrus (II–XIV) in Rm_16 area (see Figure 5b). With increasing spatial offset, the spectra highlight the appearance and disappearance of the sulphur signal (155 , 221 and 475 cm^{-1}) in relation to the characteristic bands of orpiment at 355 , 311 , and 293 cm^{-1} . The spectra are normalized to the 355 cm^{-1} Raman band of orpiment.

5. Conclusions

This study reports the field validation and first in-situ applications of a new portable micro-SORS prototype designed for cultural heritage research. A number of new findings of artistic significance were made using the advanced portable micro-SORS prototype. Field campaigns within the MOLAB access activity, on Andrea Solario's paintings and the Book of the Dead of Kha papyrus, demonstrated the system's ability to non-invasively detect low-wavenumber pigments (typically inaccessible to conventional portable Raman systems), reconstruct complex stratigraphies, and reveal compositional heterogeneities linked to degradation. At the same time, several areas for future improvements have been identified, as the integration of SERDS and automated data-processing modules to mitigate fluorescence and improve efficiency. The prototype represents a major advancement in portable, non-invasive Raman analyses, bridging laboratory micro-SORS performance with the practical needs of Cultural Heritage in-situ scenarios.

Supplementary Materials

The additional data and information can be downloaded at: <https://media.sciltp.com/articles/others/2511191619042211/PS-25100127-SM.pdf>. Figure S1: Locations of micro-SORS measurements performed on Madonna with Child (a), Madonna nursing the Child (b), Rest on the Flight into Egypt (c), Ecce Homo (d), Saint Catherine of Alexandria and Saint John the Baptist (e–f), Saint Anthony Abbot (g) and Saint John the Baptist (h). Photos credit: Museo Poldi Pezzoli, Milano. Figure S2: Locations of micro-SORS measurements performed on the Book of the Dead of Kha. Photo credit: Museo Egizio, Torino.

Author Contributions

A.B.: conceptualization, methodology, data curation, writing—original draft preparation; writing—reviewing and editing; A.L.: methodology, data curation, investigation, writing—original draft preparation, writing—reviewing and editing; C.C. (Chiara Colombo): supervision, writing—reviewing and editing; S.T.: conceptualization, investigation, supervision, writing—reviewing and editing; E.F.: conceptualization, investigation, supervision, writing—reviewing and editing; L.G.: conceptualization, investigation, supervision, writing—reviewing and editing; C.B.: conceptualization, investigation, supervision, writing—reviewing and editing; K.A.: methodology, data curation, investigation, writing—reviewing and editing; M.P.: supervision, writing—reviewing and editing; P.M.: supervision, writing—reviewing and editing; A.S.: data curation, investigation, writing—reviewing and editing; C.M.: conceptualization, supervision, writing—reviewing and editing; C.C. (Claudia Conti): conceptualization, methodology, supervision, data curation, writing—original draft preparation; writing—reviewing and editing. All authors have read and agreed to the published version of the manuscript.

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Institutional Review Board Statement

Not applicable.

Informed Consent Statement

Not applicable.

Data Availability Statement

Data will be made available on request.

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Conflicts of Interest

The authors declare no conflict of interest.

Use of AI and AI-Assisted Technologies

No AI tools were utilized for this paper.

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