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CRONO: a fast and reconfigurable macro X-ray fluorescence scanner for in-situ investigations of heritage objects

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CRONO: a fast and reconfigurable macro X-ray fluorescence scanner for in-situ investigations of heritage objects

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Abstract

CRONO is a new portable and easy reconfigurable macro X-ray fluorescence (MA-XRF) scanner based on the energy dispersive X-ray fluorescence technique, which has been specifically designed for in-situ, fast and non-invasive elemental mapping. The main components are fully integrated into the measurement head which includes an X-ray tube, a large area silicon drift detector, a microscope camera, two pointing lasers, a He purging set, and fast acquisition electronics. This very compact head is mounted on motorized stages (with a linear speed up to 45mm/s) that allow the scanning of areas up to 45×60cm^2. Three collimators (0.5mm, 1mm and 2mm diameter) are software selectable to obtain different spot-sizes on the sample. The typical measurement time for the full scanned area ranges from 1h with the 2mm collimator to about 15h with the 0.5mm collimator using a dwell time of 50ms. Technical details and achievable performances of the instrument are presented and discussed along with an example of application which illustrates the value of the developed instrument in the investigation of paintings.

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Introduction

X-ray fluorescence spectroscopy is a long established technique for non invasive in situ elemental analysis of heritage objects [1]. In most of the analytical applications, the compositional heterogeneity of heritage materials poses high challenges especially for paintings and, more in general, for polychrome surfaces. In order to reveal not only the composition but also the distribution of painting materials and thus the artist's working method, the use of chemical imaging techniques at the macro-scale and with high-spatial resolution is extremely valuable. In order to achieve these purposes in the last years the use of XRF for elemental macro scale mapping of paintings at high spatial resolution was rapidly increasing with rising success [2-5]. The technique provides (sub-)surface non-invasive analysis of painted artefacts ([6] and references therein) and a deep insight into painting materials especially when combined with complementary imaging techniques [7-8].

Until recent times, micro- and macro- XRF mapping/imaging analysis was possible only by synchrotron radiation requiring the transport of precious artworks to large scale facilities [9- 11]. Only in the very last years the advancement in instrumental technologies has made it possible to transpose the technique to laboratory and to in-situ applications [2, 3, 12, 13]. However finding a good compromise which takes into account instrument portability, large scans, beam dimension, count rates, and acquisition time is still very challenging [2, 14, 15].

In this paper, we present CRONO, a new commercial reconfigurable MA-XRF scanner implemented by XGLab (Milan, Italy) in collaboration with ISTM-CNR. CRONO is fully portable, it scores state of the art analytical performances, and has been specifically designed for applications in cultural heritage in order to meet the high demanding analytical requirements for use in the mobile laboratory MOLAB [16]. On the basis of the MOLAB decennial experience on non-invasive in-situ investigations of artworks, full portability, easy reconfiguration, and fast acquisitions are requisites indispensable for "in field" studies of heritage materials. In the following, a detailed description of CRONO and of its analytical characteristics is given. Furthermore, as applicative example, results from the analysis of a historical painting are presented and discussed in order to illustrate the value of the instrument in heritage science.

Instrument and technical characteristics

A general view of CRONO in some of the possible configurations is shown in Fig.1. XRF components are fully integrated into a compact ($30\times16\times15 cm^3$) and light (3kg) detection head mounted on a motorised XYZ stage. The excitation source is a highly efficient and compact 10W

X-ray tube generator with Rh anode (tube voltage and current are selectable from 10 to 50kV and form 5 to 200μA, respectively); the X-ray detector element is a large area (active collimated area is 50mm^2) SDD (Silicon Drift Detector) equipped with CUBE preamplifier (a CMOS circuit provided by XGLab [17]). Such X-ray detection module is characterised by state-of-the-art performances. The detection module is read-out by a new digital pulse processor (named DANTE and developed by XGLab) characterised by excellent noise and high count-rate performances, and real-time data transmission capability for on-the-flight data collection [18]. The complete spectra information is saved and made available for the post processing, since the electronic and the software can handle one 4096-bin spectrum every 30ms for an indefinite-long acquisition. The Mn_Kα energy resolution is below 130eV for 100kcps input-rate using 1μs filtering peaking-time. The system can reach a count-rate up to 1Mcps input-rate using 400ns of peaking-time with a resolution <140eV at Mn_Kα. Peak to background ratio is typically greater than 15000.

The system is driven by an integrated controller board and an embedded PC with Ethernet or wireless connection to the external computer. Three X-ray tube pine-hole collimators of 0.5mm, 1 mm and 2mm diameter are software selectable in order to obtain different spot-sizes on the sample. A filter-set (with four software-selectable filters) is also integrated in the instrument head to improve the detection limit in special applications. The design of the spectrometer allows the detection of elements ranging from Na to U with good efficiency even in the region between 1 and 2keV (i.e. Na, Mg, Al, Si, P K-edge emissions) and in the region above 25keV (e.g. Sn, Sb, Ba K-edge emissions). Na and Mg sensitivity can be improved further by purging Helium through a valve integrated into the detection unit.

The XRF head is mounted on motorized stages that allow up to 60×45cm^2 scanning area and 7,5cm focusing axis. The system operates in non-contact mode with about 1 cm of distance from the analysed object. The head is equipped with two lasers used to obtain the optimal working distance and to check for the correct alignment of the scanning plane with respect to the sample surface. The area under analysis can be monitored in real-time by the operator thanks to a microscope camera (with dimming illumination adjustment) and an external camera with a larger field of view, both integrated into the measurement head.

Thanks to a proper design of the mechanics, of the electronics and of the XRF components, the scanning of the sample can be performed with a maximum speed up to 45mm/s. On-the-fly spectra acquisitions with dwell time down to 30ms allow the scanner to perform fast and high-spatial resolution measurements. The typical measurement time ranges from few tens of minutes up to few hours depending on required spatial resolution, scanned area and contrast in the XRF maps. For

example the full scanned area (45×60cm^2) takes about 1h with the 2mm collimator and about 16h with the 0.5mm collimator using a dwell time of 50ms.

The scanner can be mounted on a light trolley (see Fig.1) that enables the tilting of the motorized frame between -90° and +110° with respect to the horizontal plane and height coarse regulation (from 120cm up to 220cm) of the measured area. The overall weight of CRONO (including trolley, motorized frame, and measurement head) is about 60kg, therefore it can be transported completely assembled. Nevertheless, if necessary, the motorized frame and the trolley can be easily dismounted for transportation to difficult sites. This is the case of archaeological sites, scaffoldings or uncomfortable historical buildings for which CRONO can be profitably turned into a portable spot-XRF device by simply detaching the measurement head from the motorized frame whenever mapping is not necessary. The system is completed by an advanced software interface for the complete instrument control, for data acquisition, for data analysis and to save raw data or prepare in different format (including pdf files) final report documentation.

Results and discussion

Analytical performances

CRONO has been purposely designed for the use of pin-hole collimators instead of polycapillary optics in order to make the system more suitable for analytical applications in heritage science. The use of a polycapillary optics in MA-XRF instrumentation presents the advantage to have high excitation photon flux when a small spot size (in the order of 100μm or below) is required. On the other hand, a pin-hole collimator solution shows some remarkable advantages: (i) good sensitivity over all elements even in the low energy region and for transitions in the range up to 35keV (e.g. Sn, Sb, Ba K-edge emissions), (ii) very low divergence of the incident X-ray beam [2, 19] and therefore low sensitivity to irregularities of sample's surface or misalignments that can degrade the spot size uniformity over the sample, affecting the image quality, (iii) no need of expensive optics and high power micro-focus tube with consequent cost effectiveness, (iv) possibility to design a more compact and portable instrument.

The analytical sensitivity of CRONO was assessed on a NIST SRM610 glass standard with 500ppm trace elements in a broad Z-range. Measurements were carried out at the operating conditions of 50kV, 200μA, 1mm collimator, without X-ray filter and no He-purging (considered the reference configuration). Fig.2 reports the spectrum acquired in 1000s with the identification of some reference peaks. Fig.3a shows Kα-line sensitivity in terms of counts per second (cps) of the net area under the Kα-line (processed with fitting algorithms implemented in the instrument software) normalised to the certified concentration expressed in mg/g for the elements from Z=19 (K) to Z=56

(Ba): the measurement shows a good sensitivity (for a compact and portable instrument) both for low-Z and high-Z elements. Considering the 1mm collimator, in the energy region around 3keV, for instance, the sensitivity is above 40cps/(mg/g) for K; while in the energy region above 30keV the sensitivity is above 100cps/(mg/g) for Ba. For elements in the intermediate range of $26 \leq Z \leq 47$ the sensitivity reaches 1000-2000cps/(mg/g). Similar measurements were carried out using the 0.5mm and 2mm collimators apertures available on CRONO. The results, presented in Fig.3a, show the expected trend with the X-Ray flux hitting the sample: the sensitivity increases with larger collimator and decreases with the smaller collimator. In order to validate the analytical sensitivity of the instrument in conditions closer to the typical macro scanning operative ones, similar measurements were performed with different acquisition time (1s, 10s and 100s) and with the 0.5mm collimator. The results are shown in Fig.3b. It is worth noting that, in tests with low acquisition time and poor statistics due to the trace elements concentration (500ppm) of this reference sample, the quality of the spectra fitting algorithm becomes crucial.

Fig.4 shows the limit of detection (LOD) calculated according to the standard definition [4], measured with the same sample and in the same experimental conditions, in particular max tube power (50kV, 200μA), 1mm collimator and 1000s of acquisition. In the energy region around 3keV for instance the LOD is below 40ppm for K; while in the energy region above 30keV the LOD is below 20ppm for Ba. For elements in the more sensitive intermediate range of $26 \leq Z \leq 47$ the LOD is 1-2 ppm.

In order to test the spatial resolution capability of the scanner, the X-ray beam size at the optimum focusing position (1cm from the sample) has been precisely determined with the knife-edge technique performing dense scans of a thin metal sheet. The resulting data have been processed to determine the X and Y dimension of the beam. The 0.5mm, 1mm and 2mm collimators produce a beam size on the sample respectively of 0.85mm, 1.7mm and 3mm FWHM. It has also been verified that the beam has almost circular geometry with about 10% ovalization.

The spatial resolution capabilities of CRONO have been confirmed on a reference standard with several horizontal and vertical copper strips structures (line-pair per mm). The scan has been performed at the maximum tube power settings (50kV and 200μA), horizontal motor speed of 4mm/s and dwell-time of 50ms per spectrum. The pixel dimensions of the reconstructed image are $250 \times 250 \mu m^2$ on the sample and the overall area is $95 \times 65 mm^2$. As shown in Fig.5, the 500μm collimator aperture makes possible to discriminate one line-pair per mm in the lateral dimension (that is 500μm detail discrimination). Further instrument developments are foreseen to reduce the spot size on the sample, especially for applications that require higher spatial resolution. In particular, preliminary studies regarding optimization of geometries of the X-ray collimator show

that a beam size on the sample of the order of 0.65mm with an estimated 40% reduction of the X-ray flux can be implemented in the CRONO system.

Example of application to a historical painting

We benefited from potentials of CRONO for fast acquisitions of elemental distribution images to assist the restorers of the "*Istituto Superiore per la Conservazione ed il Restauro-ISCR*" of Rome during the restoration of a XV Century panel painting representing the *Virgin with the child* by an unknown artist. The painting was damaged during a fire that deformed the wood panel and blackened large part of the surface, as shown in the image of Fig.6. Preliminary point XRF analyses performed by ISCR evidenced the permanence of the original pigments under the blackened layer. Scanning XRF measurements were, therefore, considered necessary for a full characterization of the elemental composition of the surface in order to provide information on the distribution of the original pigments. Particular attention was focused on the recognition of the gilded areas in order to preserve the remains of the gilding under the black layer during restoration.

Fig.6 reports some of the most informative elemental distribution images (Pb_Lα, Hg_Lα, Cu_Kα, Au_Lα Fe_K and Ca_Kα) recorded on a total scanned area of 46×58cm^2 in three different measurements with the following conditions: tube settings 50kV and 200μA, 2mm collimator aperture, scanning speed of 19mm/s, dwell-time per pixel 100ms. Total measurement time was about 2.5h. The elemental XRF maps were obtained by a standard *region of interest* (ROI) analysis since more complex data elaboration is beyond the aim of this paper, being the authors more focused on the presentation of the instrument capabilities. Due to the pronounced distortion of the wood panel, the elemental distribution images present some mismatch between the overlapping scanned areas. Nevertheless, the use of a pin-hole collimator allowed mitigating the artefacts due to the unevenness of the surface and therefore good quality images have been obtained. They are shown in Fig.6 and enable to appreciate significant details of the painting technique. For example the Pb map suggests the use of the white pigment lead white (lead carbonate/hydrate lead carbonate) for the flesh tones, revealing the face expression of the two figures, otherwise completely indiscernible to the human eyes. Lead white has been also used for the drape around the hips of the Child and it clearly appears in the Pb map to be fold and held by the Virgin's left hand from where it falls down. Furthermore, the diffuse distribution of lead - especially in the gilded background around the two saint figures - hints towards the presence of a preparatory white layer (*imprimitura*) containing again lead white. The Hg map reveals the use of cinnabar (HgS) for the darker highlights of the flesh tones of the two faces and of the Child's body. Cinnabar has also been used for the vest of the Virgin together with iron (see Fe map), but only few remains of the pigment

are left and still visible only by MA-XRF. The Cu map clearly shows the use of a copper-based pigment (most probably azurite, a blue basic copper carbonate) for the mantle of the Virgin. Interestingly, in the internal side of the mantle the copper distribution reveals a fine floral decoration which is also visible in the Fe and, with much lower intensity, in the Au maps, but as negative images. In a similar way Fe looks more intense at the edges of the blue mantle around the Virgin's figure. This fact can be ascribed to the use of bole (a red clay rich in iron) above the blue layer as a preparation for the application of gold in order to produce a warmer colour. The Au distribution image also reveals the technical choice of the artist to use gold foils for the background gilding. In fact, especially on the right part of the gilded background, squared profiles with increased intensity are clearly visible and ascribable to the overlapping of the gold leafs at the boundaries. Besides to the gilded areas, iron is also present in the flesh and the hairs of the two figures as well as in the red vest of the Virgin, suggesting the use of ochres (natural clays rich in iron) as brown-red pigments. The same pattern of Fe is followed by the distribution of Ca, as this latter is always present in clays. Furthermore, since Ca is the principal element of the grounds (usually gypsum and glue), it can be used as indicator of the presence of missing paint areas (*lacunas*), here mainly observed in the gilded background.

Conclusions

In this paper the recent development of the portable macro X-ray fluorescence (MA-XRF) scanner CRONO and its complete characterization in terms of detection sensitivity, spectroscopic and spatial resolution performances has been presented. The system has been designed with the aim of fulfilling specific analytical requirements for *in-situ* MA-XRF investigations of polychrome surfaces on heritage objects, i.e.: high sensitivity, good spatial resolution, large scanning areas, high scanning speed, portability, and easy re-configurability. In order to demonstrate the instrument capabilities for applications in the field of cultural heritage, the results form the study of a XV Century panel painting representing the *Virgin with the child* have been presented and discussed. The study was motivated by the presence of a diffuse blackened layer over the original paint produced by a past fire that prevents the visual reading of the painting and inhibits the work of restorers. Among the results provided by the MA-XRF investigation, the good resolution of the Ca, Fe and Au distribution images here presented allowed the restorers to trace in detail the distribution of the original gilding of the background under the black layer. Furthermore, MA-XRF revealed on the blue mantle of the Virgin a gold floral motif which is no longer visible. Nevertheless, the information on its original appearance is still preserved by the Fe and Cu distribution maps.

The successful use of CRONO to investigate an ancient painting as challenging case study not only demonstrates its diagnostic potentials in heritage science but also paves the way for applications in other fields demanding for high analytical performances in elemental analysis, such as industrial processes and material research in general.

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Caption to figures

Fig.1: Crono XRF detection head mounted on the motorized frame and trolley in some of the possible configurations (vertical, horizontal, only the frame on the floor).

Fig.2: Spectrum of NIST SRM610 glass standard with 500ppm trace elements in a broad Z-range. Measurement conditions: 50kV, 200μA, 1mm collimator, 1000s, no filter and air atmosphere. Some reference peaks are identified.

Fig.3: (a) Measured sensitivity in counts per second per (mg/g) concentration for the elements from Z=19 (K) to Z=56 (Ba) and 2mm, 1mm and 0.5mm primary beam collimators. (b) same measurements with 1s, 10s and 100s and the 0.5mm collimator.

Fig.4: Limit of detection (LOD) for the elements from Z=19 (K) to Z=56 (Ba). X-ray tube operated at 50kV and 200μA, 1mm collimator and 1000s of acquisition.

Fig.5: Cu map from a scan of a reference standard with several horizontal and vertical copper strips structures (line-pair per mm). Conditions: X-ray tube settings 50kV and 200uA, no filter, air atmosphere, horizontal motor speed of 4mm/s, acquisition time 50ms per pixel, image reconstruction pixel dimensions 250×250μm^2, overall area 95×65 mm^2.

Fig.6: Far Left: Visible image of the painting "*Virgin with the child*" (XV Century) and evidenced scanned area in white line. Clockwise from middle-top: XRF elemental distribution maps of Pb_Lα, Hg_Lα, Cu_Kα, Ca_Kα, Au_Lα and Fe_Kα.

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Fig.1: Crono XRF detection head mounted on the motorized frame and trolley in some of the possible configurations (vertical, horizontal, only the frame on the floor).
Fig.1
218x168mm (300 x 300 DPI)

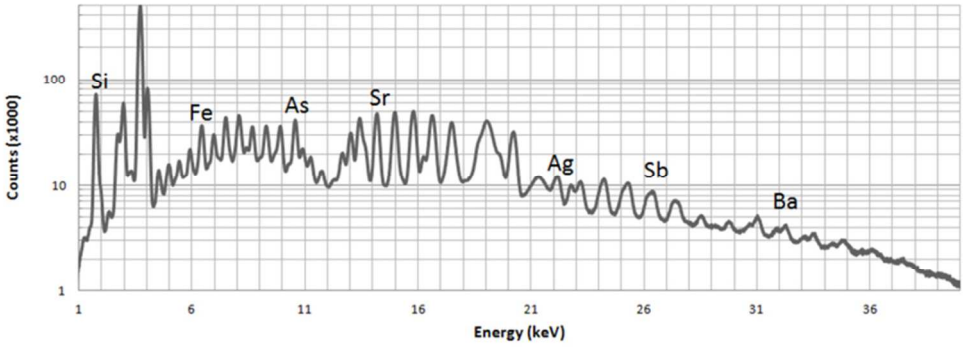

Fig.2: Spectrum of NIST SRM610 glass standard with 500ppm trace elements in a broad Z-range. Measurement conditions: 50kV, 200µA, 1mm collimator, 1000s, no filter and air atmosphere. Some reference peaks are identified.
Fig.2
35x13mm (600 x 600 DPI)

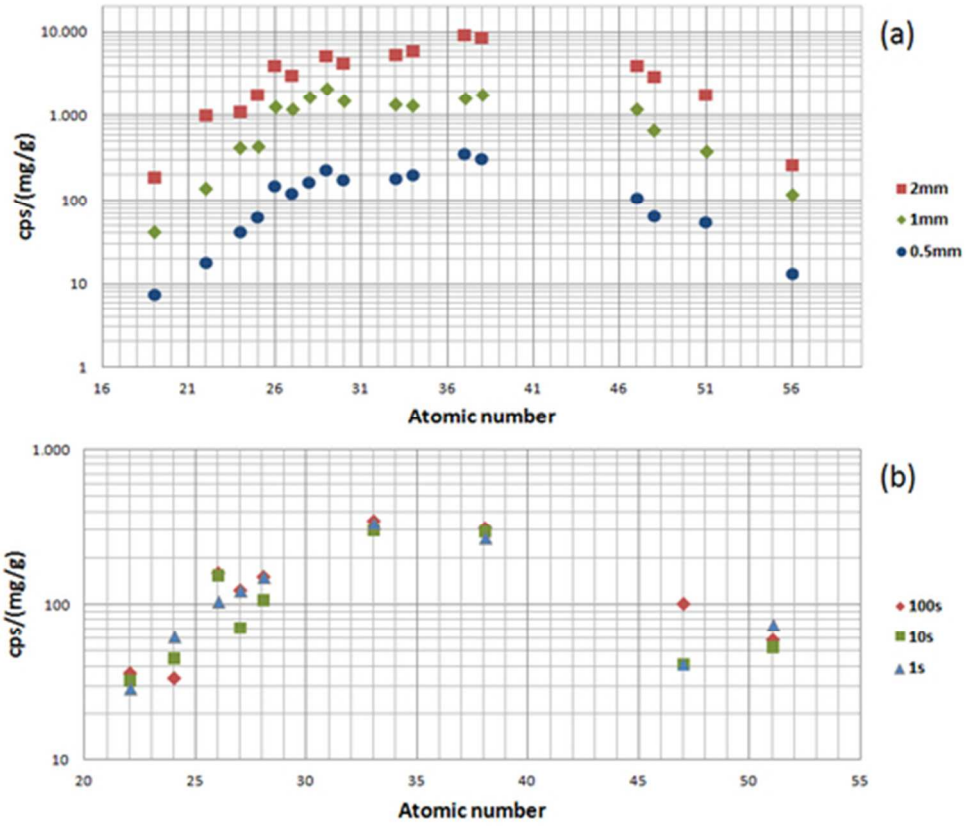

Fig.3: (a) Measured sensitivity in counts per second per (mg/g) concentration for the elements from Z=19 (K) to Z=56 (Ba) and 2mm, 1mm and 0.5mm primary beam collimators. (b) same measurements with 1s, 10s and 100s and the 0.5mm collimator.
Fig.3
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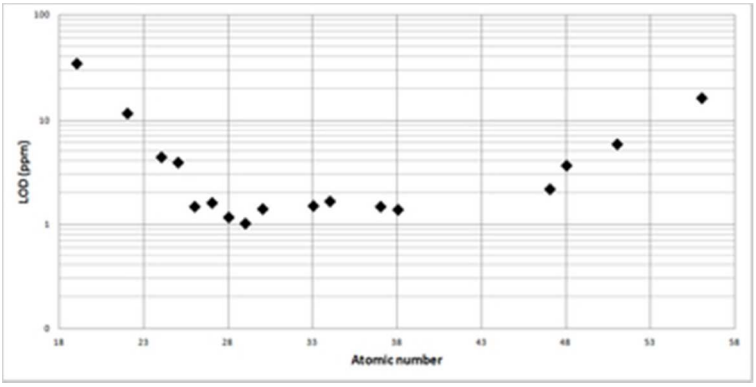

Fig.4: Limit of detection (LOD) for the elements from Z=19 (K) to Z=56 (Ba). X-ray tube operated at 50kV and 200μA, 1mm collimator and 1000s of acquisition.
Fig.4
15x8mm (600 x 600 DPI)

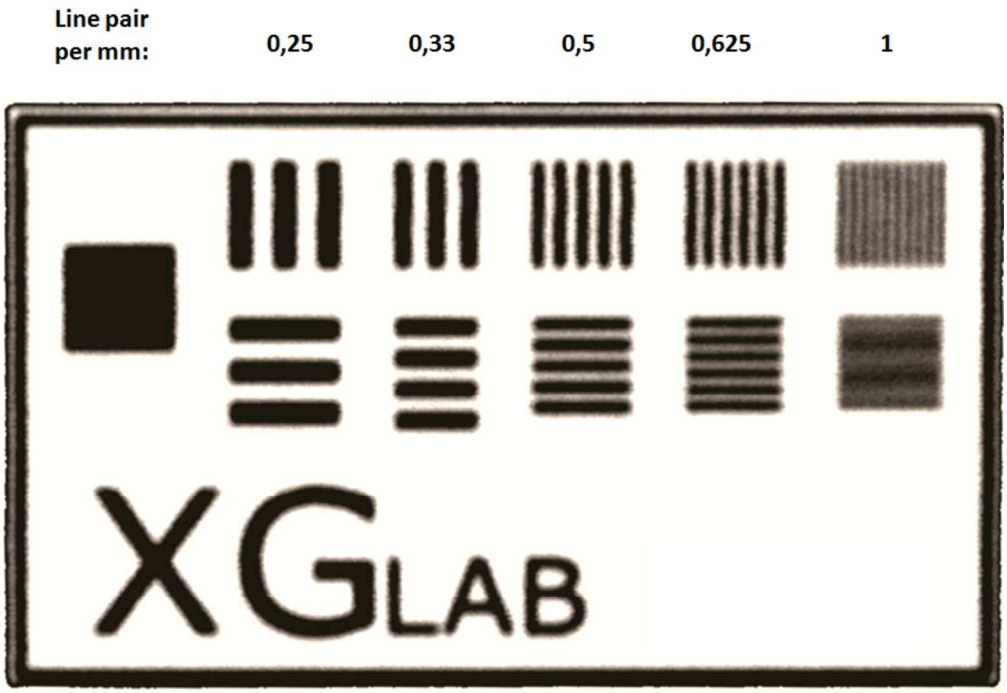

Fig.5: Cu map from a scan of a reference standard with several horizontal and vertical copper strips structures (line-pair per mm). Conditions: X-ray tube settings 50kV and 200uA, no filter, air atmosphere, horizontal motor speed of 4mm/s, acquisition time 50ms per pixel, image reconstruction pixel dimensions 250×250µm2, overall area 95×65 mm2.
Fig.5
169x119mm (300 x 300 DPI)

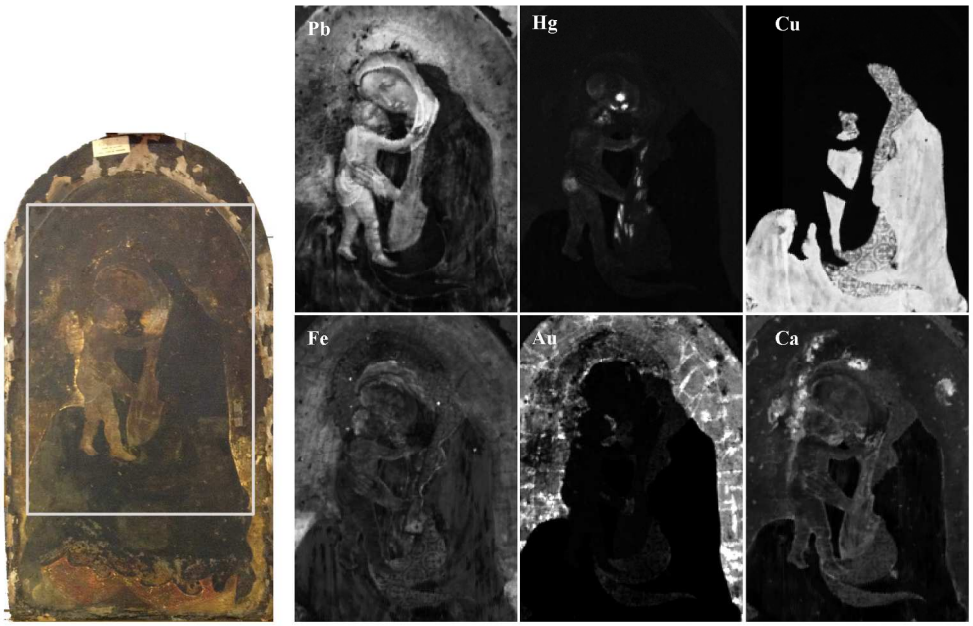

Fig.6: Far Left: Visible image of the painting "Virgin with the child" (XV Century) and evidenced scanned area in white line. Clockwise from middle-top: XRF elemental distribution maps of Pb_La, Hg_La, Cu_Kα, Ca_Kα, Au_La and Fe_Kα.
Fig.6
211x136mm (300 x 300 DPI)