

## LABORATORY: CNR ISPC - XRAYLab

### NAME OF THE INSTRUMENT

MICRO-XRF scanner developed at XRAYLab

### GENERAL DESCRIPTION:

Micro-XRF mapping is a non-destructive analytical technique based on scanning the sample surface using an X-ray beam strongly focused on the micrometric scale that provides images of elemental distributions with high lateral resolution.

The micro-XRF technique is non-destructive, it can be applied in situ and it is indicated for all those applications in which elemental analysis of materials is aimed at studies of provenance, nature of materials and their state of conservation, manufacturing technique or artistic process. For example, the technique can be used in the search for platinum metals (PGM) in native gold archaeological objects or for studying the artist's brushstrokes in a pictorial composition.

Micro-XRF technique is based on irradiation of the sample surface by continuous scanning with speeds of up to 50mm/sec with a primary X-ray beam with dimensions of the order of 10 microns over an area of 50x50cm<sup>2</sup>. Given the size of the scanning area, for the first time the micro-XRF technique can also be applied in situ on macroscopic contexts. The X-ray fluorescence emitted by the atomic species of which the sample is composed is suitably revealed as a function of location by a multi-detector system (up to 6 elements) that allows to operate with great efficiency and, consequently, with residence times per pixel of the order of ms. This feature drastically reduces measurement times. Investigations are videos driven by a high-resolution optical microscope, and images of elemental distributions in studio samples are provided and processed in real time, already during measurement.

### *Synthetic guide for choosing the micro-XRF technique of ISPC-CNR*

Materials: all materials of an inorganic nature

Optimal cases of application: pictorial works, metals, painted or glazed ceramics, mineralogical or petrographic material, etc.

Sample placement: vertical and horizontal

Type of analysis: non-destructive and in-situ

Measuring times: 3 hours to cover an area of 10x10cm<sup>2</sup> with a resolution of 10 microns.

Maximum resolution: 3-5 microns

Characteristics and parameters of the X source: Mo anode, 50Kv and 0.6mA (30W power); Cr anode, 50 kV 0.6mA for specific, high-efficiency measurements of low atomic number elements.

Beam size: in the range of distances between 10-15 microns 3 mm from the sample surface.

Detection system: Multi-detector up to six 50mm<sup>2</sup> SDD elements operating in parallel

Other techniques present in the instrument: MA-XRF, confocal XRF.

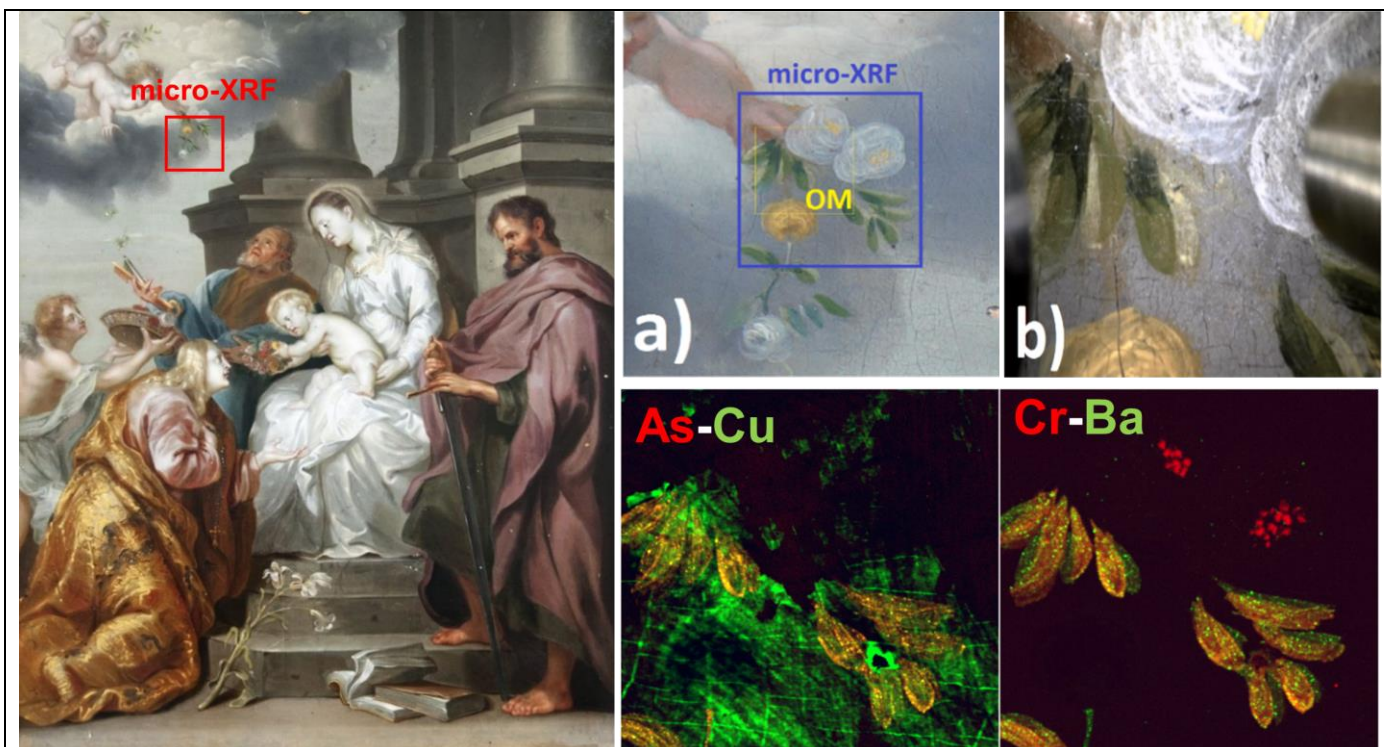
Analytical specifications: Elemental images are provided in real time during measurements with XRF spectra for already deconvoluted pixels (absence of artifacts); the analysis software allows real-time imaging-processing, RGB elemental correlations, correlation scatter plots, PCA statistical analysis, ICA and NMF, spectra analysis on individual regions of interest, maximum spectrum (to identify trace elements or localized inclusions)

Safety: automatic stop in case of exceeding a threshold of approach.

### **TECHNICAL DESCRIPTION:**

The micro-XRF mobile scanner developed at XRAYLab laboratory is a mechatronic system operating in real-time technology. The system consists of a spectrometric measuring head equipped with a microfocus X-ray tube with a 30W Mo anode coupled with a focusing polycapillary lens. A second primary source of X-rays with Cr anode can be alternating with rh's for better acquisition of images of chemical elements with spectroscopic lines in the low energy region. In addition, the combined use of the two sources allows to separate the distributions of the same chemical element present at different stratigraphic depths of the work. X fluorescence induced by primary radiation is revealed in event mode by a multi-detector system that contains up to 6 50mm<sup>2</sup> SDD and energy resolution <130eV at 5.9 keV, operating in parallel. The spectrometric head is mounted on a 3-axis system (XYZ) with a stroke of 50x50x20cm<sup>3</sup> that allows the scanning to be carried out in continuous mode at high speed (up to 50 mm/sec) and with a holding time per pixel up to 1msec.

The system is equipped with a central CPU for the control of measurement operating parameters and darkening systems. The CPU handles: the scan sequence; automatically adjusts the parameters of the X source and detectors to avoid degradation of dead time and energy resolution in the XRF spectra of individual pixels; manages a 750 frames/sec laser interferometer for dynamic measurement distance correction, so as to avoid accidental shocks of the scanner with the work in question; preserve scan coordinates so that analysis resumes from the same location in case of prolonged pauses or system downtime (e.g. night breaks). In addition, the CPU operates dynamic spectra-pixel analysis by applying an accurate fit procedure and providing elemental images in real-time without artifacts. The scanning system is also equipped with software for real-time image analysis through which it is possible to correlate images (RGB), apply logical-mathematical tools, apply statistical analysis (e.g. PCA, ICA and NNMF), generate scatter-plots and export individual XRF spectra by selecting any area (ROI) of the image. The scanner can be assembled in two different spectrometric head configurations allowing to analyze large surfaces that can be placed both vertically and horizontally.



*Micro-XRF guided video analysis conducted on an unallocated copy (oil on panel, 40x30cm) of Anthony van Dyck's painting "Madonna and Child and Saints Rosalia, Peter and Paul". The presence of anachronistic pigments has made it possible to question the authenticity*

#### **FURTHER INFORMATION**

- Romano, F. P., Caliri, C., Nicotra, P., Di Martino, S., Pappalardo, L., Rizzo, F., & Santos, H. C. (2017). Real-time elemental imaging of large dimension paintings with a novel mobile macro X-ray fluorescence (MA-XRF) scanning technique. *Journal of Analytical Atomic Spectrometry*, 32(4), 773-781.
- H. Santos, et al., Identification of forgeries in historical enamels by combining the non-destructive scanning XRF imaging and alpha-PIXE portable techniques, *Microchemical Journal*, 124 (2016) 241–246.

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