LABORATORY: CNR-ISPC - XRAYLab

NAME OF THE INSTRUMENT

X-ray diffractometer developed within the XRAYLab

GENERAL DESCRIPTION:

Different mineralogical phases can be identified via X-Ray Diffraction (XRD). Differently from element-specific analytical methods (such is the broadly used XRF), XRD provides information on the nature and structure of chemical compounds (even if present in complex mixtures).

Given the crystallinity found in different types of antics materials, XRD is particularly suited for investigation of many archeological and historical-artistic samples. Its application is especially fruitful in the identification of the nature of pigments and their degradation products in paintings realized on any support (wood panels, papyrus paper, frescoes, murals) as well as the study of corrosion and degradation surface layers.

The mobile XRD system developed within the XRAYlab of ISPC is applied on polycristalline sample and employs a parallel beam. This set up has the great advantage, compare to traditional XRD systems, of keeping fixed in position both the sample and the Xray source, while the Bragg angular scan is obtained by moving the detector alone. In this fashion, accurate diffraction patterns are collected since there are less opportunities to introduce angular shifts and the angular resolution improves.

This is a not destructive method and there is no need for any sample preparation.

Practical guide for the choice of XRD method of XRAYLAB at CNR-ISPC

Materials: polycrystalline samples

Optimal applications: paint pigments on any medium, surface degradations and corrosions

Sample positioning: vertical

Type of analysis: point by point, not-destructive, in-situ (also on scaffolding).

Measuring times: in the order of 30 minutes per point, for the main phases

Probe size: ~600 micron

Measuring distance from the sample: around 15mm

X-ray source characteristics and parameters: Fe anode, 50kV and 0,6 mA (30W power)

Measuring distance from the sample: 10 cm

Angular resolution of diffraction pattern: ~0.15 deg

Quantitative analysis: possible for homogeneous mixtures in flat geometry

Additional method available: simultaneous acquisition of XRD pattern and XRF spectrum in the same sample spot.

TECHNICAL DESCRIPTION:

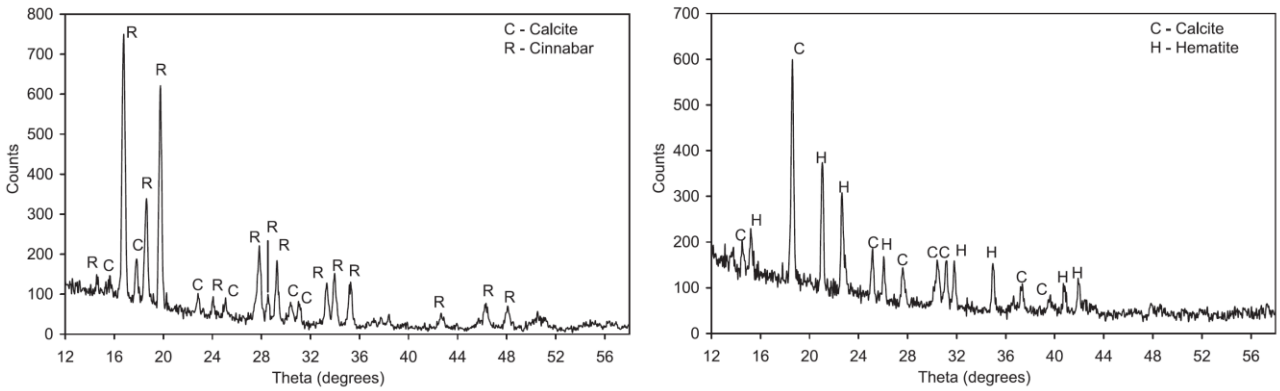
The XRD system developed within the XRAYlab of ISPC is a modular mobile system optimized for in-situ operation. The system is composed of a measurement head equipped with a 30W Fe target microfocus X-ray source coupled with a polycapillary optics for parallel beam with spot on the investigated sample of about 600 micron.

A Si-PIN detector is used for the collection of the diffraction pattern emerging from the samples. A

digital filter acts on the DXP (Digital X-ray Processor) of the processor to select the wavelength (ie the energy) of which visualize the diffraction - typically K-alpha line of the tube anode material. During each measurement it is possible to acquire simultaneously both the XRD pattern and the XRF spectrum for the same measurement point.

The X-source and the detector operate on a mechanical goniometer of theta-theta geometry, with radius of 15 cm. The parallel X-ray beam is kept at a fixed angle of ~10 deg along the goniometer (ie the source tube is not moved), while the detector automatically scans angular position between 12 and 70 deg, from the opposite end of the goniometer. The sample is positioned outside the goniometer rail, at its geometrical center (ie at distance of ~15 cm from all points of the rail). Precise alignment of the sample is gained through a two-lasers pointing system.

During measurement, the whole diffractometer head is held by a tripod that keeps the system in position relatively to the investigated sample area. The sample needs to be held vertically on either a support or hang on a wall. Each XRD point measurement takes approximatively 30 minutes.



Analytical results of the XRD technique in the study of pigment mixtures on frescoes from the Roman era.

The XRD system in use during the analysis of the Last Supper by the Flemish painter Luis de Morales and a bust representing the Hellenistic goddess Demeter.

FURTHER INFORMATION:

- F.P. Romano, et al., The new version of the portable XRD system of the LANDIS laboratory, Il Nuovo Cimento 121 (2005) 881-885.
- G. Gatto Rotondo, et al., Non-destructive characterization of fifty various species of pigments of archaeological and artistic interest by using the portable X-ray diffraction system of the LANDIS laboratory of Catania (Italy), Microchemical Journal 96 (2010) 252–258.
- F.P. Romano et al., The compositional and mineralogical analysis of fired pigments in Nasca pottery from Cahuachi (Peru) by the combined use of the portable PIXE-alpha and portable XRD techniques, Microchemical Journal 99 (2011) 449–453.
- L. Pappalardo, et al., The complementary use of PIXE-alpha and XRD non-destructive portable systems for the quantitative analysis of painted surfaces, X-Ray Spectrometry, 37 (2008) 370–375.

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