Radiation Physics and Chemistry 137 (2017) 238–242

Contents lists available at ScienceDirect

Radiation Physics and Chemistry

journal homepage: www.elsevier.com/locate/radphyschem

# Confocal X-ray fluorescence spectrometer for in-situ analyses of paintings

Tomáš Trojek [a,*], Radek Prokeš [a], Radka Šefců [b], Hana Bilavčíková [b], Tomáš Čechák [a]

[a] Czech Technical University in Prague, Department of Dosimetry and Application of Ionizing Radiation, Břehová 7, 115 19, Prague, Czech Republic
[b] National Gallery in Prague, Staroměstské nám. 12, 110 00, Prague, Czech Republic

## HIGHLIGHTS

- Confocal XRF spectrometer for in-situ measurements of painting was constructed.
- Preparation of a sample plate of historical paint layers was described.
- Movable collimating optics enables us to change quickly the confocal volume.

## ARTICLE INFO

*Article history:*
Received 31 October 2015
Received in revised form
17 February 2016
Accepted 26 February 2016
Available online 2 March 2016

*Keywords:*
X-ray fluorescence
Microanalysis
Confocal setup
Pigment
Painting

## ABSTRACT

This paper describes the properties of the newly constructed device for confocal X-ray fluorescence analysis that was tested with a sample plate consisting of 19 combinations of two single pigment layers. The preparation of this experimental wooden board with layered systems was based on knowledge of the panel painting techniques of Bohemian collections from the National Gallery in Prague dating back to the 14th and 15th centuries. The design of the confocal setup allows its transport and the in-situ measurement of paintings in depositories or even in exhibition areas. The advantages of our confocal setup with movable collimating optics are also described.



## 1. Introduction

X-ray fluorescence analysis is a non-destructive analytical technique that is frequently used for investigation of paintings because it swiftly provides us with information on the elemental composition of a selected part of the painting. This data may refer to the use of a certain pigment or a group of pigments. Such measurements can be performed with handheld XRF analysers. These instruments are great with regard to simplicity and flexibility but they are analysing quite a large area of the investigated object. This disadvantage is usually acceptable for wall paintings or large paintings in general. Tiny details must be surveyed with laboratory instruments for XRF or micro-XRF analysis (Valadas et al., 2011).

The identification of pigment is essential for the work of conservators and in addition the authorship of the painting can be inferred (Šefců et al., 2015; Chlumská et al., 2014). Lots of historical pigments were used for many centuries, and therefore the identification of certain pigments does not usually attribute the piece of work to the author. However, the structure of layers can be distinctive for an author or his workshop (Šefců et al., 2015). The standard procedure for determination of paint layers includes taking micro-samples and their probing with optical and electron microscopy (Eastaugh et al., 2008). Since the taking of micro-samples is destructive it is not acceptable in many cases. The non-destructive analysis of mediaeval paint layers requires identification of rather heavier elements, e.g. iron, copper, lead and mercury, which are distributed in paint layers up to several tens of micrometres thick. We therefore decided to construct a portable spectrometer for confocal XRF analysis.

Confocal X-ray fluorescence analysis is a promising analytical technique intended for non-destructive investigation of objects with a layered structure. Its principle consists in detection of characteristic X-rays from a small confocal volume that is defined by the overlap of the foci of the focusing and the collimating optics located at the source and the detector side of a confocal XRF setup. Experimental setups for the confocal micro-XRF analysis can be distinguished by the source of exciting radiation. For excitation it

* Corresponding author.
*E-mail address:* tomas.trojek@fjfi.cvut.cz (T. Trojek).

http://dx.doi.org/10.1016/j.radphyschem.2016.02.031

is possible to use a synchrotron beam (Janssens et al., 2004; Woll et al., 2006) and confocal XRF analysis has also been realized under laboratory conditions using X-ray tubes as a source of exciting radiation (Kanngießer et al., 2005; Lin et al., 2008). The first archaeometric applications of confocal XRF analysis were published by Kanngießer et al., 2003.

## 2. Confocal X-ray fluorescence device

The new transportable XRF system with a confocal setup was designed and constructed in the Department of Dosimetry and Application of Ionizing Radiation at the Czech Technical University in Prague. The main requirement on its properties is the ability to analyse paint layers in-situ, i.e. good depth resolution is required and the dimensions and the mass of the XRF device must make its transport feasible.

The basis of the new XRF spectrometer is a robust metallic board on which the XRF components are mounted, see Fig. 1. The main XRF components include an X-ray source, a spectrometric detector and two X-ray lenses. The board is movable along a horizontal plane thanks to two durable screw-bolts with stepping motors. While the shifting in one direction (perpendicular to the surface of the painting) is necessary for depth profiling, the second one can be used for a surface line scan, for instance.

The X-ray source radiation is an air-cooled X-ray tube with a Mo target coupled to a focusing polycapillary lens (X-Beam® Superflux PF) manufactured by the XOS company. The maximum power of the X-ray tube is 50 W (50 kV, 1 mA). The beam diameter (FWHM) is lower than 15 µm for the Mo-Kα line (17.4 keV) in the focal spot that is located approximately 4 mm from the end cap of the focusing optics. The secondary lens is a polycapillary collimating X-ray optics also produced by XOS. The fluorescence radiation penetrating through this lens is then detected with a FAST SDD™ detector manufactured by the Amptek company. The detector has an active area of 25 mm$^2$ and a thickness of 0.5 mm. The thickness of the Be entrance window is 12.7 µm. While the X-ray tube and its focusing optics are stationary, the collimating lens, which is located in front of the entrance window of the detector, is attached to a motorised x–y–z stage. The stage, specifically an MP63-DC assembled by the Steinmeyer FMD company, is PC controlled and it enables movement of the optics along all three axes with a step size below 1 µm. This opens up the possibility of seeking the confocal configuration easily or the optics can be removed from the detector and the confocal XRF setup is immediately switched to the micro-XRF mode. The advantage is that the position of the x–y–z stage is preserved even when the stage or the computer is switched off.

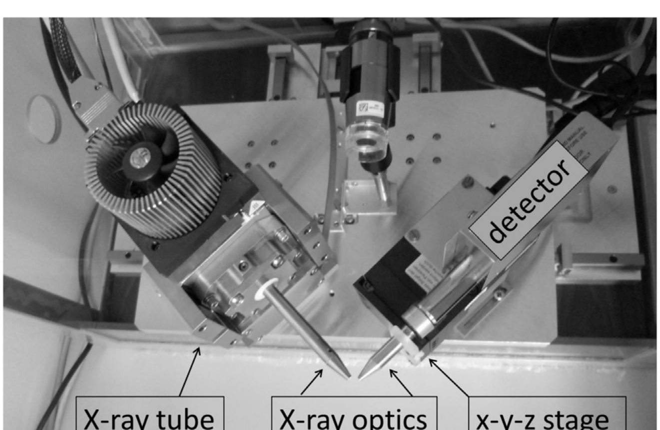


**Fig. 1.** Detailed view of the metallic board with the main components of the confocal XRF device.

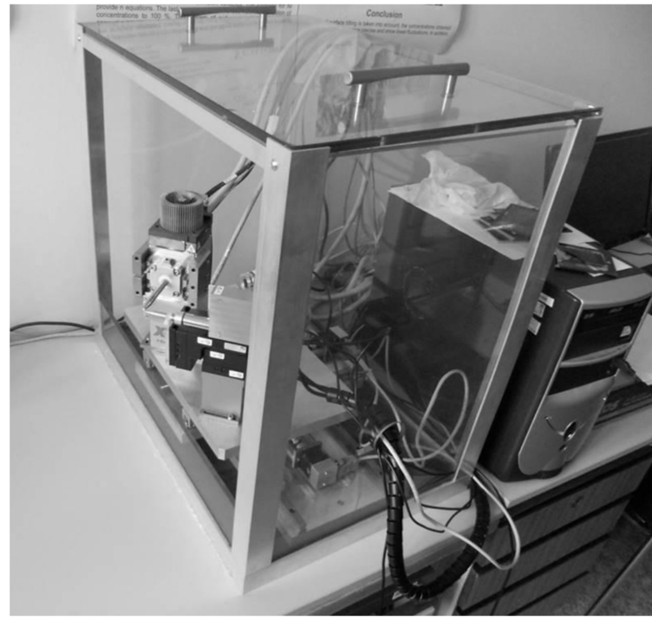

**Fig. 2.** The whole confocal XRF device; the X-ray source and other main components may be placed in the glass box.

The fluorescence and scattered X-rays represent a potential radiation risk to the operator of this spectrometer. The dose rates are around hundreds of µSv/h in the areas ordinarily accessible to workers. Therefore, the whole metallic table with all main components of the confocal XRF system can be put into a glass box with dimensions 60 cmx60 cmx40 cm, see Fig. 2. Only the computer and the HV power supply of the X-ray tube are out of this box. Apart from the front side of the box, through which the table comes out of the box, all other sides of the box and the cover are made of 1 cm thick sheets of glass. The thickness of the glass reduces the dose rate to the value of a natural background.

When other tests of this new confocal XRF device have finished, it will be transported to the National Gallery in Prague where it will be used as a table-top system or it will be placed on a running lifting stage (maximum load of 350 kg and a lift of up to 1.6 m), which will enable us to analyse large paintings in the exhibition rooms of an art gallery. Further work will be focused on techniques of data evaluation that are necessary for correct interpretation of the X-ray spectra acquired within the depth profiling. The full description of matrix effects and geometrical effects for polychromatic X-ray photons in confocal geometry is a demanding task (Wrobel et al., 2014).

## 3. Sample plate of paint layers

The properties of this new confocal XRF setup were tested with samples of paint layers. The preparation of experimental wooden boards with layered systems was based on knowledge of the panel painting techniques of Bohemian collections from the National Gallery in Prague dating back to the 14th and 15th centuries (Slánský, 1953; Chlumská et al., 2012; Royt and Pokorný, 2013). We clearly defined a system with multiple layers, which were simplified to monochrome layers always containing one type of pigment. Therefore, we were able clearly and representatively to evaluate the experimental measurements. The thicknesses of individual layers were several tens of micrometres, an average from 5 to 100 µm. The multilayer system was applied to a 25 × 25 cm wooden board. The board was divided into 20 individual segments