

# The Incoatec Microfocus Source $\mu$ S for XRD-Applications

## Microdiffraction using an $\mu$ S

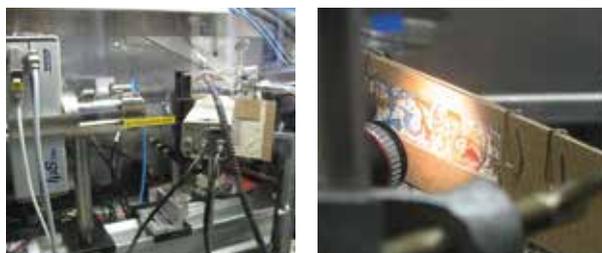


Frederik Vanmeert, University of Antwerp has examined De Heem's painting 'Flowers and Insects', Royal Museum of Fine Arts Antwerp with the Incoatec Microfocus Source  $\mu$ S<sup>High brilliance</sup>.

In recent years the interest in the non-destructive investigation of cultural heritage objects has risen strongly. Besides infrared and optical imaging and spectroscopic methods, X-ray methods like X-ray fluorescence, X-ray diffraction, X-ray radiography and X-ray imaging methods (e. g. tomography) are often used for the analysis of these artifacts [1].

Using for example X-ray fluorescence (XRF) the scrolls of Qumran were investigated [2], fragments could be identified as belonging together or not [3]. Using XRF analysis of paintings generally provides information about the possible presence of elements on the surface of art-objects due to pollution (e.g. sulphur or chlorine), about the elements and pigments used by the artist, about previous restored areas detected by the presence of "modern" elements like titanium or zinc, and identification of fraudulent submission. In combination with X-ray diffraction (XRD) also the crystallographic composition of the used pigments could be characterised. A study about the alteration involved the oxidation of cadmium yellow (CdS) to  $\text{CdSO}_4 \cdot 2\text{H}_2\text{O}$  under the influence of light, oxygen and moisture is an example of the use of this technique in the investigation of paintings [1]. Other X-ray methods are used as well. A painting by Rembrandt van Rijn was analysed using an X-ray absorption imaging technique at ESRF and with XRF revealing an overpainting [4].

In the study shown here a medieaval book painting was investigated using XRF and XRD-techniques simultaneously. While with XRF the elemental composition of the used pigments are analysed, with XRD crystallographic information could be revealed. Using both methods, the chemical composition of the pigments could be analysed. Here a Mo- $\mu$ S with a focusing optics was used. The sample was tilted by 45° relative to the beam direction. The XRD patterns were recorded with a SMART 1000 CCD-detector (Bruker AXS) in transmission geometry, XRF signals were measured with an energy dispersive detector arranged in 90° to the beam direction.



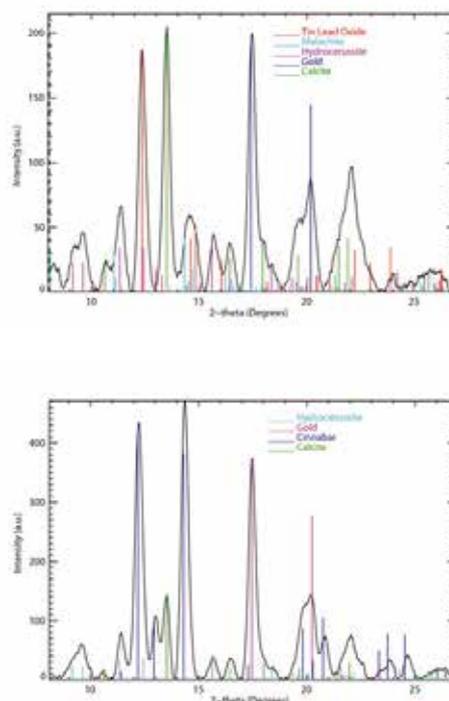
$\mu$ S setup for the measurements in transmission geometry



Book painting

The focal position of the  $\mu$ S was between sample and detector. A sample area of about 130  $\mu$ m x 130  $\mu$ m was illuminated with the X-ray beam. Using this setup, XRD frames were recorded within 30 seconds exposure time, the XRF measurement was done simultaneously. Integrated XRD pattern for green and red regions are shown with peak positions of the used pigments.

In an overnight-scan (approx. 18 hours) an area of several square millimeter and a resolution of 150  $\mu$ m could be investigated.



Diffraction pattern of green (top) and red (below) colored regions

This study shows, that it is possible to obtain high quality results even with a quite simple setup. The  $\mu$ S, the sample and the XRD-detector were just aligned on an optical bench while the XRF-detector was just placed on a shelf in a suitable position.

**Acknowledgement:**

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**References:**

[1] K. Janssens, J. Dik, M. Cotte, J. Susini; Photon-Based Techniques for Nondestructive Subsurface Analysis of Painted Cultural Heritage Artifacts. *Accounts of Chemical Research* 43 (2010) 814–825  
 [2] O.Hahn, T.Wolff, B.Kanngießer, W.Malzer, and I.Mantouvalou; Non-destructive investigation of the scroll material: „4Qcomposition Concerning Divine Providence“ (4Q413). *Dead Sea Discoveries*, Bd. 14, 2007, S. 359-364 .  
 [3] I. Rabin, O. Hahn, T. Wolff, A. Masic, G. Weinberg; On the origin of the ink of the Thanksgiving Scroll (1QHodayot{a}). *Dead Sea Discoveries* 16, 97-106  
 [4] [http://www.esrf.eu/news/general/hidden-rembrandt/index\\_html](http://www.esrf.eu/news/general/hidden-rembrandt/index_html); retrieved 20.08.2015