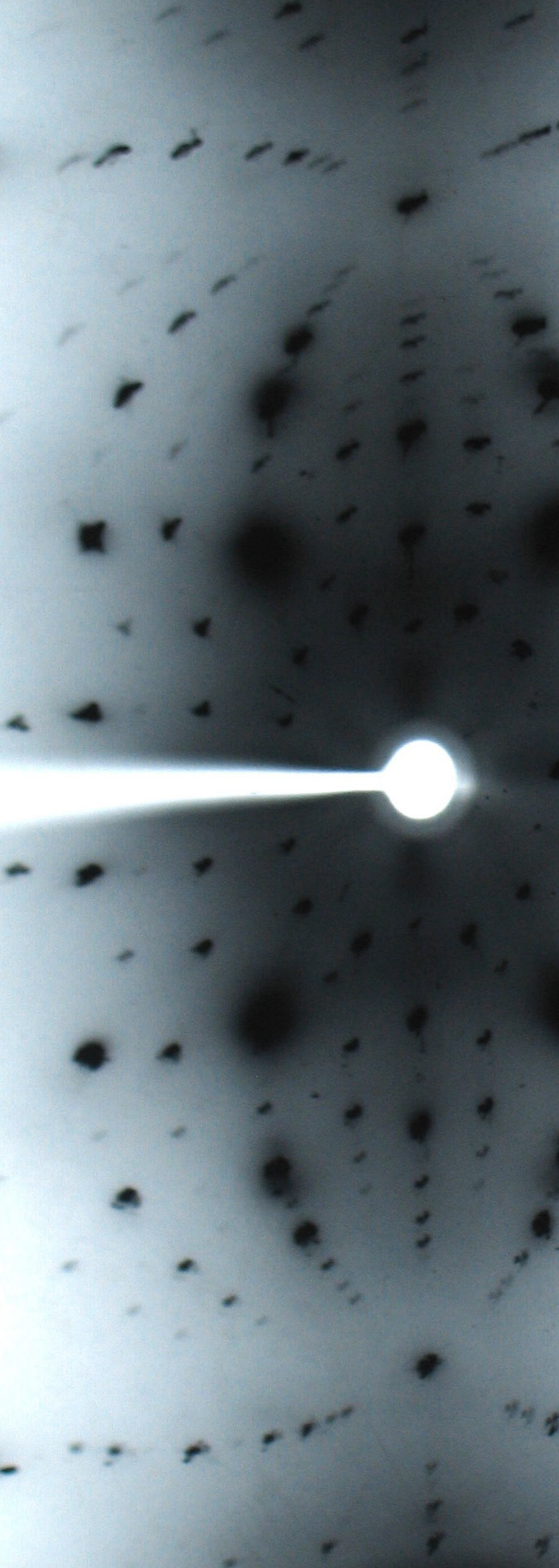

4th Meeting X-ray techniques in investigations of the objects of cultural heritage. Celebrating the 100th anniversary of crystal X-ray diffraction

Krakow, 17–19 May 2012

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Secretary of the Meeting
Alicja Rafalska-Łasocha

Programme

May 17, Thursday

8.00 – 10.00 Registration

Morning session

10.00 – 10.15 ***Opening*** – Krzysztof Stopka – the director of Collegium Maius, Grażyna Stochel – Dean of the Faculty of Chemistry, Jagiellonian University

Chair: Wiesław Łasocha

10.15 – 11.00 ***From Röntgen and Laue to modern crystallography –*** Henk Schenk, University of Amsterdam, The Netherlands

11.00 – 11.30 Coffee break

11.30 – 12.15 ***Science for the cultural heritage: the contribution of X-ray and neutron diffraction*** – Gilberto Artioli, Dipartimento di Geoscienze, Universita di Padova, Italy

12.15 – 12.45 ***Application of μ-XRD for identification of the substrate in red lake pigment*** – Justyna Olszewska-Świetlik[1], Bożena Szmelter-Fausek[1], Marta Grzesiak[2], Alicja Rafalska-Łasocha[3], [1] Faculty of Fine Arts, Nicolaus Copernicus University in Toruń, Poland; [2] The Jerzy Haber Institute of Catalysis and Surface Chemistry Polish Academy of Sciences; [3]Faculty of Chemistry Jagiellonian University, Krakow, Poland

12.45 – 13.15 ***Neutron imaging techniques as alternative tools for non-destructive testing of cultural heritage objects – compared to traditional X-ray imaging*** – Eberhard H. Lehmann, Steven Petermans, David Mannes, Jan Hovind, Paul Scherrer Institut, Villigen PSI, Switzerland

Afternoon session

Chair: Roman Kozłowski

14.00 – 14.30 ***Application of X-ray techniques in conservation studios of the National Museum in Krakow*** – Janusz Czop, National Museum, Krakow, Poland

14.30 – 15.00 ***X-ray based methods for analysis of Henryk Siemiradzki "self-portrait"*** – Dominika Sarkowicz, Anna Klisińska-Kopacz, National Museum, Krakow, Poland

15.30 – 16.00 ***Non-invasive investigations of paintings, metal artifacts and manuscripts using X-ray fluorescence analysis (XRF)*** – Manfred Schreiner, Institute of Science and Technology in Arts, Academy of Fine Arts, Vienna, Austria

15.30 – 16.00 Coffee break & Poster session

17.00 – 19.00 Visit to Collegium Maius – the Jagiellonian University Museum

May 18, Friday

Morning session

Chair: Wiesław Łasocha

9.00 – 9.30 ***Interference of art and science in the von Laue hall*** – Thomas Wroblewski, Hamburger Synchrotron-strahlungslabor (HASYLAB) at Deutsches elektronen-Synchrotron (DESY), Hamburg, Germany

9.30 – 10.00 ***Scanning macro-XRF: How large scale elemental distribution images give insight in compositional changes during the creation of historical paintings and later restoration treatments*** – Matthias Alfeld[1], Koen Janssens[1], Joris Dik[2], [1]Universiteit Antwerpen, Department of Chemistry, Center for Micro- and Trace Analysis, Belgium; [2]Delft University of Technology, Department of Materials Science, Delft, The Netherlands

10.00 – 10.30 Coffee break

10.30 – 11.00 ***Confocal micro X-ray analyses on Renaissance paintings* of the Louvre Museum** – Ina Reiche, Katharina Müller, Estelle Itié, Myriam Eveno, Bruno Mottin, Michel Menu, Centre de Recherche et de Restauration des Musées de France, France

11.00 – 11.30 ***Non destructive X-ray Fluorescence analysis of pigments in a 16^th^ century panel painting*** – Anabelle Križnar[1], Kilian Laclavetine[1], Maria del Valme Muñoz[2], Miguel Ángel Respaldiza[1] and Mercedes Vega[2]; [1]University of Seville, Centro Nacional de Aceleradores; [2]Fine Arts Museum of Seville, Spain

11.30 – 12.00 ***PIXE analysis of silver coins from ancient historical times*** – Erazm Dutkiewicz, Artur Sroka, The Henryk Niewodniczański Institute of Nuclear Physics, Polish Academy of Sciences, Krakow, Poland

12.00 – 12.30 ***Application of energy dispersive X-ray microanalysis for examination of historic glasses***, Małgorzata Walczak, Zofia Kaszowska, Paweł Karaszkiewicz; Jan Matejko Academy of Fine Arts in Krakow, Faculty of Conservation and Restoration of Works of Art, Poland

12.30 – 13.00 ***Studies of a set of early modern portraits of the University of Krakow professors in the collection of Collegium Maius. The meaning of X-ray investigation in the research***, Anna Jasińska, Jolanta Pollesch, Beata Skalmierska, Jagiellonian University Museum, Krakow, Poland

Afternoon session

Chair: Grażyna Korpal

14.00 – 14.30 ***The computed tomography of the 15th century Madonna with a Child from Książnice Wielkie as an example of non-invasive method of a masterpiece examination*** – Dagny Dobrowolska, Private practice in the sculpture conservation, Krakow, Poland

14.30 – 15.00 ***Application of X-ray Dual Source Computed Tomography (DSCT) in investigations of the objects of Cultural Heritage*** – Anna Mikołajska[1], Małgorzata Urbańczyk Zawadzka[2], Robert Paweł Banyś[2]; [1]Jan Matejko Academy of Fine Arts in Krakow, Faculty of Conservation and Restoration of Works of Art; [2]Center for Diagnosis Prevention and Telemedicine, John Paul II Hospital, Krakow, Poland

15.00 – 15.30 ***Szczerbiec (the jagged sword) – the coronation sword of the kings of Poland*** – Marcin Biborski[1], Mateusz Biborski[2], Janusz Stępiński[3], Grzegorz Żabiński[4]; [1]Institut of Archeology of the Jagiellonian University, [2]Museum of Jagiellonian University, Krakow, [3]AGH University of Science and Technology, Krakow, [4]Academy of Jan Długosz, Częstochowa, Poland

15.30 – 16.00 Coffee break

16.00 – 16.30 ***2D Micro-diffraction using PIXcel3D*** – Szymon Stolarek, Panalytical B.V. Branch in Poland, Warszawa, Poland

16.30 – 16.50 ***Miniflex – new brother of Rigaku X-ray diffraction family*** – Andrzej Węsek, Testchem, Pszów, Poland

16.50 – 17.00 Closing remarks

19.30 Conference dinner

May 19, Saturday

10.00 – 13.00 Visit to the Wawel Royal Castle

13.00 – 16.00 Visit to the Wieliczka Salt Mine

Poster session: May, 17, Thursday, 16.00 – 17.00

P.1. ***Conservation of the painting The Sermon of John the Baptist by Pieter II Brueghel the Younger from the Cracow collection of the National Museum*** – Katarzyna Novljaković, Anna Grochowska-Angelus, Małgorzata Chmielewska, Urszula Węgrzynowicz, National Museum, Krakow, Poland

P.2. ***X-ray powder micro-diffraction investigations of selected pigments in Seventeenth-Century Paintings*** – Alicja

Rafalska-Łasocha[1], Marta Grzesiak[2], Justyna Olszewska-Świetlik[3], Bożena Szmelter-Fausek[3], Wiesław Łasocha[1,2]; [1]Faculty of Chemistry Jagiellonian University, Krakow; [2]The Jerzy Haber Institute of Catalysis and Surface Chemistry Polish Academy of Sciences, Krakow; [3]Nicolaus Copernicus University, Faculty of Fine Arts, Toruń, Poland

P.3. ***Application of XRF technique for provenance study of historical bronze aquamanile*** – Iwona Żmuda-Trzebiatowska[1], Anna Fietkiewicz[2], Gerard Śliwiński[2] ; [1]The Szewalski Institute, Polish Academy of Sciences, Photophysics Department Gdańsk; [2]National Museum, Gdańsk, Poland

P.4. ***XRPD Investigations of the ancient coins and other metal objects*** – Marcin Kozieł[1], Marcin Oszajca[1], Alicja Rafalska-Łasocha[1], Elżbieta Nosek[2], Wiesław Łasocha[1,3]; [1]Faculty of Chemistry Jagiellonian University, Krakow;[2] Independent conservator, Krakow; [3]The Jerzy Haber Institute of Catalysis and Surface Chemistry Polish Academy of Sciences, Krakow, Poland

P.5. ***Micro-PIXE and micro-Computer Tomography investigation on elements of a porcelain vase*** – Jakub Bielecki[1], Sebastian Bożek[1,2], Magdalena Chmielińska[3], Erazm Dutkiewicz[1], Zofia Kaszowska[3], Wojciech M. Kwiatek[1], Janusz Lekki[1], Anna Mikołajska[3], and Zbigniew Stachura[1], [1] the Henryk Niewodniczański Institute of Nuclear Physics, Polish Academy of Sciences, Department of Applied Spectroscopy, Krakow; [2]The Jagiellonian University Medical College, Department of Pharmacokinetics and Physical Pharmacy, Krakow; [3]Jan Matejko Academy of Fine Arts in Krakow, Faculty of Conservation and Restoration of Works of Art, Krakow, Poland

P.6. ***Tin and tinned dress accessories of medieval inhabitans from Wrocław. Preliminary results of X-ray fluorescence*** – Beata Miazga, University of Wrocław, Institute of Archeology, Poland

P.7. ***Possibilities of the SEM/EDS technique application for identification of tannins and other compounds in historical leathers*** – Tomasz Kozielec, Grzegorz Trykowski, Nicolaus Copernicus University, Department of Paper and Leather Conservation, Toruń, Poland

P.8. ***X-ray powder diffraction in investigations of yellow earth pigments*** – Alicja Rafalska-Łasocha[1], Marta Grzesiak[2], Ewa Doleżyńska-Sewerniak[3], Wiesław Łasocha[1,2]; [1]Faculty of Chemistry Jagiellonian University, Krakow; [2]The Jerzy Haber Institute of Catalysis and Surface Chemistry PAS, Krakow; [3]Casimir the Great

ABSTRACTS
ORAL
PRESENTATIONS

FROM RŐNTGEN AND LAUE TO MODERN CRYSTALLOGRAPHY

Henk Schenk
Past president of the International Union of Crystallography HIMS, FWNI, University of Amsterdam, 1090 GD Amsterdam, The Netherlands

In 1912, Max von Laue designed an experiment that started modern Crystallography. Till that year, it was not known whether the X-rays, discovered in 1895 by Wilhelm Röntgen, consisted of particles or electromagnetic radiation. After a discussion with Peter Paul Ewald, von Laue got the idea that if X-rays were electromagnetic and the wavelength was of the same order of magnitude as distances between the atoms in crystals, the crystal could act as a three-dimensional grating and should show diffraction spots on a photographic plate. Von Laue asked Friedrich and Knipping to carry out the experiment and that revealed diffraction spots indeed. This proved the electromagnetic nature of X-rays, but, even more important, showed that crystals act as a three-dimensional grating for X-rays. X-ray diffraction (XRD) was born and X-ray crystallography could start to be a new branch in science revealing the 3-D world of atoms and molecules.
Shortly after his finding, William and Lawrence Bragg solved the first structure from XRD data, rock salt.
The lecture will deal with start of the X-ray century till the state-of-the-art of today, with special attention to the state of knowledge in 1962. In that year, the lecturer started X-ray crystallography with a full year course at the laboratory of Carolina MacGillavry at the Amsterdam University. In those years, also computers were beginning to be available and they facilitated the spectacular growth of the subject, both in speed and in methods to solve the so-called phase problem.
Today, probably more than one million crystal structures have been solved of which 600.000 of small molecules are collected in the Cambridge Structural Database and 80.000 of large bio-molecules in the Protein Database. Other databases are specialized in powder diffraction data, inorganic structures and others.

SCIENCE FOR THE CULTURAL HERITAGE: THE CONTRIBUTION OF X-RAY AND NEUTRON DIFFRACTION

Gilberto Artioli
Dipartimento di Geoscienze, Università di Padova, Italy

Diffraction techniques are fundamental tools for the characterization of materials of different nature, including those relevant for the cultural heritage. The aims of the investigations include the issues of diagnostics and conservation of art works, and analysis and interpretation of archaeological artifacts related to human past.
The various methods and techniques of single-crystal and powder diffraction applied to cultural heritage materials will be briefly described with reference to specific examples, in order to extract information on **phase identification and quantification** of crystalline compounds (Fig. 1) and complex polyphasic mixtures (stone artefacts, binders, pigments), **texture** (metals), and **kinetics** of the reaction processes (degradation).
Further insights can be derived from **combined studies** involving simultaneous diffraction, imaging and/or spectroscopy experiments. For example, combined micro-mapping of the elemental and phase composition proves to be very important in understanding the nature and assessing the degradation state of materials.

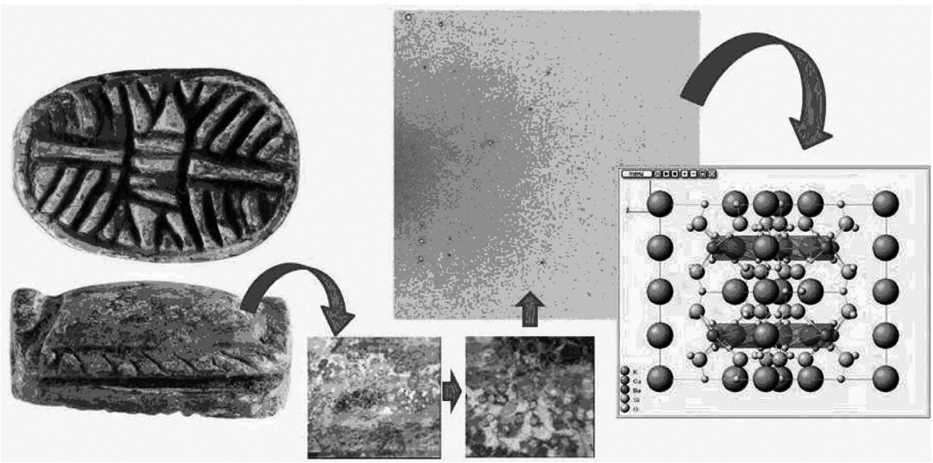

Fig. 1. Example of application of single-crystal X-ray diffraction: identification and structure analysis of a milarite-type high temperature phase crystallized at the core-glaze interface of a glazed-steatite scarab. Combined with chemical analyses and electron microscopy data, the identification of the compound yields interesting detailed information on the manufacturing process.

APPLICATION OF µ-XRD FOR IDENTIFICATION OF THE SUBSTRATE IN RED LAKE PIGMENT

Justyna Olszewska-Świetlik[1], Bożena Szmelter-Fausek[1] Marta Grzesiak[2], Alicja Rafalska-Łasocha[3]
[1]Faculty of Fine Arts, Nicolaus Copernicus University, Toruń, Poland [2]The Jerzy Haber Institute of Catalysis and Surface Chemistry Polish Academy of Sciences, Krakow, [3]Faculty of Chemistry, Jagiellonian University, Krakow, Poland

Identification of artistic materials is very important in research on works of art. Investigations of lake pigments and methods of obtaining them allow to establish the technology and technique of a painting workshop. The main difficulty in analysis of lake pigments as well as their substrates is small size of samples.
The dyes might be met in various works of art like illuminated manuscripts, paintings on wood and on canvas, textiles. The lake pigments most commonly found in works of art are those prepared from red, yellow and blue dyestuffs.
There are documentary sources and pigment recipes which give the information on ways of obtaining red lake pigments.
In European old master paintings, different red organic dyes were favoured depending on the region. The source of a lake pigment was a plant or animal material. Research on the type of a dye and on the method of obtaining it provides important information related to the painting workshop.
The chemical nature and historical aspects of red dyestuffs have been well examined, therefore analysis of the substrates of lake pigments was the aim of performed investigations. The main goal was to verify if the X-ray powder diffraction can characterize the inorganic substrate in the red lake layer.
The µ-XRD measurements were carried out with the use of an X'PERT PRO MPD diffractometer. The analysis was performed on a cross section. Paint with ground samples were taken from the edge of damaged area of a painting and mounted in a synthetic resin.
µ-XRD method was found suitable for characterization of the substrate in red lake layer on the cross-section. It allows to gain knowledge not only on the substrate but also on the source from which the dyes were obtained.

NEUTRON IMAGING TECHNIQUES AS ALTERNATIVE TOOLS FOR NON-DESTRUCTIVE TESTING OF CULTURAL HERITAGE OBJECTS – COMPARED TO RADITIONAL X-RAY IMAGING

Eberhard H. Lehmann, Steven Petermans, David Mannes, Jan Hovind
Paul Scherrer Institut, Villigen PSI, Switzerland

Objects from cultural heritage have to be investigated mainly non-destructively or even non-invasively. In this respect, X-ray radiography has been used as a valuable and effective method since decades [1]. As a macroscopic method, X-ray imaging provides a good overview about the material distribution in depth of the objects given by individual contrasts of the involved substances.
In our contribution, we want to introduce neutron imaging as an alternative tool for non-invasive material studies in respect to different classes of cultural heritage objects. Its power is given by the alternative attenuation behaviour of neutrons in comparison to X-rays, where most of the heavy metals get transparent but organic materials show a high contrast due to the neutron scattering at hydrogen.
Due to the efficiency of modern neutron imaging devices, a single transmission neutron image can be obtained in a few seconds, when the often discussed neutron activation can be ignored. Even neutron tomography, which enables the study of full 3-dimensional volume of objects, has no activation risk if materials like Co or Ag are not present in the sample. In some cases, neutron imaging is the only method to get the needed information either given by the transmission ability of neutrons or the high contrast from light elements which are not visible with X-rays. For other objects, X-ray and neutron imaging can be applied simultaneously and synergetic information can be derived about the inner structures.
With two installations for neutron imaging and another facility for X-rays, the Paul Scherrer Institute, Switzerland is well equipped to perform dedicated investigations of suitable museums objects.
A short introduction into the available techniques will be given during the talk.
In our presentation, we will report about several successful studies covering a famous lead-sealed violinist sculpture from Spain, a stony relic from Fribourg, Buddha sculptures from the 15th century [2], Roman bronze objects [3], casting attempts from the experimental archaeometry and the study of block excavation from a Swiss region. The results should encourage potential users of neutron imaging to perform trials at our beam lines and to understand the potential for future dedicated studies.

References:
[1] J. Lang, A. Middleton, Radiography of Cultural Material, 2nd edition, Elsevier, 2005

[2] E.H. Lehmann, S. Hartmann, M.O. Speidel, Investigation of the content of ancient Tibetan metallic Buddha statues by means of neutron imaging methods, Archaeometry 52, 2010/3, 416-428
[3] Deschler-Erb E, Lehmann EH, Pernet L, et al. The complementary use of neutrons and X-rays for the non-destructive investigation of archaeological objects from Swiss collections, Archaeometry 46, 2004/4, 647-661

APPLICATION OF X-RAY TECHNIQUES IN THE CONSERVATION STUDIOS OF THE NATIONAL MUSEUM IN KRAKOW

Janusz Czop
National Museum, Krakow, Poland

X-ray techniques are standard non-invasive methods used in museums to study the structure and technology of objects, evaluate the state of preservation, gain information on previous conservation treatments or analyse changes in the original author's intention. However, only recently these methods have become readily available and widely used at the Laboratory of Analysis and Non-destructive Investigations of Heritage Objects (LANBOZ) of the National Museum in Krakow (MNK).
In 2008, the Museum purchased an ArtTAX 400 portable Energy Dispersive X-ray Fluorescence spectrometer. It allows for on-site, non-destructive, simultaneous multi-element analysis from sodium to uranium to map the element distribution and to analyse the provenance of materials. The measurement procedure is completely non-invasive and non-contact. It can be performed directly on an object located in the laboratory,

exhibition space or storage room. X-ray fluorescence spectrometry is now a standard technique widely used at the Museum. Recently, this method was used to measure the elemental composition of Early Piast coins, which are primarily constituted of silver and copper. Furthermore, calibration methods are currently being developed for quantitative determination of these metals at the surface of the coins.

The Museum also purchased a digital radiography system (DIX-Ray) with a highly-sensitive flat panel detector. This portable system has the possibility of recording on site X-ray radiographs, which provide a comprehensive documentation invaluable in giving a broader context to the technical and art-historical analysis of museum objects. The radiographs can be digitally processed to improve the visualization of the objects' features. Recently, the Museum carried out a digital comparison of X-rays of *Lady with an Ermine* by Leonardo da Vinci taken before and after travels to other museums to determine the state of preservation of the painting. A digital comparison of radiographs obtained at MNK and those made at other institutions in 1945, 1952 and 1994 is further planned.

In addition to performing technological research based on analyses of radiographs, LANBOZ has also used digital radiography for monitoring wood impregnation and evaluating conservation treatments. This fundamental research program funded by the Ministry of Science and Higher Education within a project *Heritage wooden objects impregnated with polymers: structural changes, risk assessment, preservation strategy* has provided innovative results on a global scale. The digital radiography system enables tracing the impregnation of the wood by consolidating agents, by producing the differential images.

Another innovative application at MNK is the development of energy dispersive X-ray radiography technique in collaboration with the University of Science and Technology in Krakow. The method was developed for the *Lady with an Ermine* painting in an attempt to reveal the original background currently covered with a later repainting and has been so far tested on a painting replicating the technological structure of Leonardo's artwork. The method allows minimizing the dominant lead signal from the X-ray image with the aim of determining the surface distribution maps of copper and iron in the object's paint layer.

X-RAY BASED METHODS FOR ANALYSIS OF HENRYK SIEMIRADZKI *„SELF-PORTRAIT”*

Dominika Sarkowicz, Anna Klisińska-Kopacz
National Museum, Krakow, Poland

The integrity of works of art is the main premise that should guide the professional work of conservators. In particular, there is an increasing need for non-destructive investigations. X-ray based methods are powerful and well accepted for analysis in the cultural heritage field. Non-destructive X-ray analytical methods include X-ray radiography and X-ray fluorescence (XRF). The structure of the whole object, which is characteristic of artist's creative process is revealed by X-ray radiography, while the XRF analysis allows a quick and precise detection and identification of the inorganic elements that compose the pigments.

Henryk Siemiradzki (1843-1902) was a Polish painter known for his Greco-Roman and biblical scenes. He also painted many studies and sketches, including landscapes, as well as a few portraits. However, there are only two oil Siemiradzki's self-portraits known and both are possessed by the National Museum in Krakow. The subject of performed research was the earlier self-portrait, unfinished, painted in the 1870s. Both methods, X-ray radiography and X-ray fluorescence were used for analysis of the painting to collect information on Siemiradzki's painting technique.

X-ray radiography was performed using digital X-ray radiography (DX) with a CARESTREAM DRX-1 System Detector (a gadolinium scintillator, 2560 x 3072 pixel, resolution 14 bit, pixel pitch: 0.139 mm, image size 35 x 43 cm). Elemental composition of the object was found using an ArtTAX® µXRF spectrometer (Bruker AXS Microanalysis, Germany) equipped with a Rhodium X-ray tube (50kV, 500µA, 50 keV, in air, 300s).

X-ray radiography allowed to discover two additional compositions under the *Self-portrait* paint layer. An architectural composition was detected directly under the layer. Another discovered painting was a portrait of a woman, located under the architectural composition. XRF measurements shown the presence of elements indicating the pigments from all of the painting layers, such as lead white ($(Pb(CO_3)_2\ Pb(OH)_2)$), zinc white (ZnO), vermillion (HgS), iron natural pigments (Fe_2O_3) with mineral admixtures, iron black (Fe_3O_4), Naples yellow ($Pb_3(SbO_4)_2$), cobalt blue ($CoAl_2O_4$), chrome yellow ($PbCrO_4$), cadmium yellow ($CdS + BaSO_4$), Prussian blue $KFe[Fe(CN)_6]$.

The performed analysis allowed to understand deeply the craftsmanship and technology used by Henryk Siemiradzki. It also proved the painting to be an advanced sketch, a preparatory phase of the definite self-portrait. Probably to the one ordered by the Uffizi Gallery in Florence.

2D ELEMENTAL MAPPING WITH MOBILE XRF EQUIPMENT: APPLICATIONS TO PAINTING LAYER IMAGING

David Strivay, Francois Philippe Hocquet, Helena Calvo del Castillo, Cecile Oger

Institut de Physique Nucléaire, Atomique et de Spectroscopie & Centre Européen d'Archéométrie, University of Liège, Belgium

Imaging techniques are now commonly used in cultural heritage object analysis. The study of works of art requires usually these techniques to be non-destructive and non-invasive, and frequently to be adapted in order to perform analysis in situ.
Ever since our laboratory developed portable EDXRF and UV-Vis-NIR coupled spectrometers, specially designed for fieldwork studies, all the three techniques have been applied simultaneously. The EDXRF results obtained, though satisfactory, may still be improved by elaborating 2D elemental maps.
Recent developments on a new positioning system have now allowed to perform this 2D elemental mappings with our equipment, the control of which is entirely carried out through a laptop computer running a homemade software written for this purpose.
The positioning is achieved by means of a CCD camera embedded in the system and controlled via a WiFi connection. The acquisition system of the data, which is made through a homemade MCA, being also managed via the software mentioned above, goes through an Ethernet connection. We will present here the new developments of the system as well as an example of in situ 2D elemental mapping.

INTERFERENCE OF ART AND SCIENCE IN THE VON LAUE HALL

Thomas Wroblewski
Hamburger Synchrotronstrahlungslabor at Deutsches elektronen – Synchrotron, Hamburg, Germany

In the Max von Laue year 2012, the PETRA III experimental hall at DESY, Hamburg will be devoted to the discoverer of X-ray interferences. PETRA III is the world's most brilliant storage ring based X-ray source. Fourteen undulators deliver light to 30 experimental stations profiting from the unique properties of PETRA III. Due to its high energy of 6 GeV, hard X-rays of high brilliance in the 100 keV range can be generated, allowing non-destructive high-resolution investigations of bulky samples either by radiographic techniques like tomography or using X-ray diffraction. These techniques may be complemented by X-ray spectroscopic methods like fluorescence analysis and X-ray absorption spectroscopy. Besides these more or less conventional methods, further techniques based on the unique properties of the radiation from PETRA III like high collimation and coherence may be applied in the investigation of cultural heritage objects. Phase contrast techniques seem to be promising candidates. The von Laue hall could become a place where X-ray interferences enlighten art even without the use of crystals.

NON-INVASIVE INVESTIGATIONS OF PAINTINGS, METAL ARTIFACTS AND MANUSCRIPTS USING X-RAY FLUORESCENCE ANALYSIS (XRF)

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Email: m.schreine@akbild.ac.at

In the last decades a particular collaboration between art and natural sciences has been developed and a co-operation between archaeology, art history and conservation-restoration on one side and physics, chemistry and biology on the other side seems fairly well established. Within that collaboration material analysis is of increasing importance as the booming development of analytical methods has brought a great number of new instrumental micro-analytical techniques with non-sampling (without taking original sample material) and in-situ applicability to an artifact. X-ray fluorescence analysis (XRF) plays a unique role in that co-operation: It can be carried out in air, in most cases the analysis is non-destructive or even non-invasive, which means that no changes or alterations occur before, during or after the investigation at an object, and the miniaturization in the field of electronics yielded x-ray tubes as well as x-ray detectors of a few kilograms and below. Therefore, devices can be easily transported to an archaeological site or into museums, libraries and galleries for analytical investigations.

In the presentation a transportable X-ray fluorescence (XRF) analyzer will be discussed, which was developed and assembled at the Academy of Fine Arts in Vienna, in order to permit in-situ examinations in museums, libraries and even at archaeological excavation sites. The system is based on energy dispersive XRF using an Oxford XTF5011 50W-Rhodium x-ray tube, a Röntec XFlash 1000 silicon drift-chamber detector and two lasers for positioning [1]. Additionally, results obtained by a hand-held instrument, Spectro-xSort will be presented and the advantages and disadvantages of such systems in comparison to lab-instruments will be discussed.

SCANNING MACRO-XRF: HOW LARGE SCALE ELEMENTAL DISTRIBUTION IMAGES GIVS INSIGHT IN COMPOSITIONAL CHANGES DURING THE CREATION OF HISTORICAL PAINTINGS AND LATER RESTORATION TREATMENT

Matthias Alfeld[1], Koen Janssens[1], Joris Dik[2]
[1] University of Antwerp, Department of Chemistry, Belgium
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Oil paintings take a central position in the cultural heritage of the western world, as they allow to communicate complicated concepts and emotions in the same medium. Their creation is commonly a sequential process in that layers of oil paint are applied in succession. On the primed ground layer of a prepared canvas, a sketch of the intended composition is made. Based on this sketch, the tone of the different areas is set by applying the *underpainting*, before the painting is executed in multiple layers on top of that. During the whole process, the artists continuously re-evaluates his work and changes its composition if necessary by over painting parts of it with highly opaque oil paint. Sometimes, even whole paintings were completely over painted with a different composition to re-use the expensive canvas, if the painting was considered an artistic or commercial failure. So a visualization of the sub-surface paint layers could give valuable information on the artist's original intention, his modus operandi and also (seldom) to rediscover "lost" masterworks.
Further, in the course of time many paintings get damaged due to improper handling or storage conditions and need to be restored, which is commonly done by applying new paint layers, often with a different chemical composition than the original paint, on the damaged spots. For such a conservation treatment, the original paint left is often not removed but simply over painted. As any original paint needs to be preserved, the differentiation between infillings and original paint is of great importance in the planning of any further conservation treatment of a painting.
Since visual inspection is insufficient to study covered paint layers and chemical composition, scientific methods need to be used. Conventionally X-ray radiography (XRR) and IR-Reflectography (IRR) are employed, but these methods based on the reflection and absorption of electromagnetic radiation yield only information about a limited set of pigments.
To complement these methods, in recent years, scanning macro-XRF (MA-XRF) was introduced as a new method for the study of historical paintings that acquires elemental distribution images, which allow to study a much wider range of pigments. Due to the penetrative nature of X-rays not only elements on the surface layers but also from deeper, covered layers contribute to the acquired distribution images

that thus allow to visualize these layers and to identify and localize infillings.
In the investigation of historical paintings, speed is of crucial importance as valuable pieces can seldom be removed from the galleries or museums they are exhibited in for an extended period of time. Since early macro-XRF scanners required dwell times of several seconds per pixel, few results beyond mock-up and proof-of-principle studies were published.
To accelerate data acquisition in XRF, either an intense primary radiation source and/or a high solid angle covered by the detector(s) recording the fluorescence radiation is needed, so that the first experiments were done at synchrotron radiation sources that provide intense monochromatic radiation. In the course of our research the synchrotron scanners were improved from a simple set-up featuring a single detector to more advanced ones, making use of multiple SD detectors in parallel or pixel detectors, such as the Maia detector developed by the Commonwealth Scientific and Industrial Research Organisation (CSIRO, Australia) and the Brookhaven National Laboratory (BNL, USA), that allows to record fluorescence radiation from a high solid angle, while being able to process high intensity fluorescence radiation.
Yet, synchrotron measurements necessitate the transport of the painting, exposing it to the risk of damage from handling and changed climatic conditions and asking in general for high logistical effort. So mobile instruments based on X-ray tube sources were developed, that allowed for *in-situ* investigations in the normal storage place of a painting, albeit at a slower pace than at synchrotron sources. From a simple one tube one detector set-up with limited motor range (10x10 cm^2), advanced versions with four SD detectors positioned around the X-ray tube were developed that allow to scan a surface of 60x60 cm^2 with a lateral resolution of 1 mm in just under 20 hours (dwell time less than 0.2 s per pixel), while showing the main components in noise free images.
Based on the experiences with the in house built scanners the Bruker M6 Jetstream, a dedicated macro-XRF scanner based on the Bruker M4 Tornado, was developed, consisting of a single X-ray tube with a polycapillary optic and a single detector mounted on an 80x60 cm motor stage. The instrument combines a variable beam size (150 to 950 µm) with capabilities for fast scanning (several 10s of milliseconds) and high user friendliness.
We will illustrate the capabilities of our instruments and how the elemental distribution images acquired by them allowed to attribute paintings to Rembrandt van Rijn and Vincent van Gogh. Further, we will show what large (several square meter) elemental distribution images reveal about the painting technique of Caravaggio and demonstrate the value of MA-XRF investigations for conservators in case studies on paintings by Rembrandt van Rijn and Jan Vermeer.

CONFOCAL MICRO X-RAY ANALYSES ON RENAISSANCE PAINTINGS OF THE LOUVRE MUSEUM

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One of the cumulating points of painting art is the High Renaissance. Painters such as Leonardo da Vinci further perfected many aspects of pictorial art that still need to be understand completely. They painted by superimposing different thin paint layers to obtain faithful optical impressions and wished visual effects. Therefore, Renaissance paintings represent a challenging case study when investigating painting techniques. In this work, we applied compact tridimensional micro X-ray fluorescence analysis (3D micro-XRF, LouX3D) to study non-destructively Renaissance paintings of Leonardo da Vinci and the series of the Famous Men, the Louvre collection, and compared its performances with measurements of cross sections.
It could be shown that the mean information depth of the laboratory set-up is about 80 – 100 µm depending on the paint layer sequence and its chemical composition. Elements between Ca and Zr can be characterized with a probing volume ranging from 58 down to 34 µm. Moving the sample through the analytical probing volume with a step size of 10 µm enabled to discriminate paint layers of about 15 to 20 µm thickness. While studying the original portraits using the LouX^{3D} set-up in comparison to cross section samples, it could be shown that similar information can be gained by means of depth profiling. The profiling is however limited in depth depending on the chemistry of the paint layers and according to the performances of the set-up. Care has to be taken when the same element is present in adjacent layers and when the layer composition is highly heterogeneous. However, the elemental sensitivity of the LouX^{3D} set-up is enhanced in comparison to SEM-EDX analyses, which in turn provide a better spatial resolution. Then, paint sequences on the original paintings including overpaints could be evidenced non-destructively using the 3D micro-XRF measurements and highlight the great potential of the method in combination with other techniques for completely non-invasive painting analyses.

NON-DESTRUCTIVE X-RAY ANALYSIS OF PIGMENTS IN A 16TH CENTURY PANEL PAINTING

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The Museum of Fine Arts of Seville is the second most important gallery in Spain. Besides paintings authored by several world known artists like Murillo or Zurbaran, its complex collection holds also several interesting panels from the 15th and 16th centuries, painted by anonymous Spanish masters of which still little is known. One of these is the panel painting *San Telmo* (16th century) which forms part of the permanent exposition of the Museum. It was restored several years ago. Now, more information on materials applied was of interest. The research work was carried out directly in the exhibition room, so only non-destructive analytical methods were applied. First, the painting was studied with UV light to observe later interventions, next the possible preparatory drawing was searched with IR reflectography using a Xenicx InGaAs cámara, and last X-Ray fluorescence was applied for material analysis. The XRF equipment used was a portable device with an X-ray tube of 30 kV, an anode of W and a SDD detector with energy resolution of 140 eV. The UV image of the painting showed clearly later interventions, which helped us at the selection of points to be analysed by XRF, in order to really detect original pigments. The XRF analysis revealed that painter's palette was common for the 16th century, and similar to those found in other panels from the same period and similar style in the Museum's collection: lead white (identified by Pb peaks), yellow and red ochres (Fe), lead-tin yellow (Pb, Sn), vermilion (Hg), a copper based green pigment (Cu), azurite (Cu), and an organic black, probably bone black (Ca). Also several retouches made on the bases of Zn white were confirmed. The presence of Ca and Pb in every analysed spot shows that there must be a Ca based preparation, probably gypsum, with a lead based imprimatura. Low Hg peaks, present in almost every spectra, show a possibility of a thin layer of vermilion applied under painting layers, but this can only be confirmed by cross-sections. The interesting revelation were IR images which discovered not only a very precise and decisive artist's brushstroke, observed for the modelling of Saint's head or vestments, but also a change in the form of the boat that the Saint holds in his left hand: today it is much smaller than it was originally planned (*pentimenti*).

PIXE ANALYSIS OF SILVER COINS FROM ANCIENT HISTORICAL TIMES

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The PIXE (Proton Induced X-Ray Emission) method is a powerful and non-destructive tool for analysis of various objects. PIXE analysis of four ancient coins was performed in the Institute of Nuclear Physics in Kraków to determine silver amount in their coinage metal. The coins originated from different times and different places in Europe. Two of them come from the 4th century (Roman Empire), one from the year 1782 (Kingdom of Prussia), and one from the year 1840 (Kingdom of Poland controlled by the Russian Empire). The analysis revealed that only the latter coin has the proper composition of the alloy. The other three contain less silver than could be expected from the historical information about the coinage. The missing silver has been substituted by tin because it lightens the colour of the copper-based alloy used in all cases. The information obtained from the chemical analysis also showed that the alloys of 3 coins do not fulfil the required standards.

APPLICATION OF ENERGY DISPERSIVE X-RAY MICROANALYSIS FOR EXAMINATION OF HISTORIC GLASSES

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Scanning electron microscopy combined with energy dispersive X-ray spectrometry (SEM/EDX) is widely used in the field of cultural heritage. It permits the semi-quantitative elemental analysis and so the identification of materials used by the artist. In particular, SEM/EDX is very useful in glass investigation since it enables detection of light elements like: Na, Mg, Si and Al which are the main components of the glass network. Also the information about colouring elements could be obtained.

Here, we present a discussion on the advantages and limitations of SEM/EDX in a few examples: the analysis of samples from 19th century Chinese porcelain vase, the analysis of medieval stained glass windows from Grodziec, Poland and the investigation of laser cleaning effects on colourless stained glass windows originating from St. Catherine church, Krakow, Poland.

STUDIES OF A SET OF EARLY MODERN PORTRAITS OF THE UNIVERSITY OF KRAKOW PROFESSORS IN THE COLLECTION OF COLLEGIUM MAIUS THE MEANING OF X-RAY INVESTIGATION IN THE RESEARCH

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From 2005 to 2007, a research team of the Jagiellonian University Museum worked on an interdisciplinary project involving studies of a set of 35 early modern portraits of professors of the University of Krakow[1] – paintings that had never been studied before. This was carried out by an art historian, a historian, by conservators, physicists and chemists, the works – financed by The Getty Foundation – made it possible to study the paintings from the historical, as well as conservation and technological perspective.

Painted on canvas, the portraits were studied primarily by means of x-ray imaging, which was one of the fundamental and extremely important ways of reading the technique, technology and history of painting renovation. A traditional form of a well taken and developed image, a soft-focus image on a film, reveals many details and permits accurate interpretation.

The 35 paintings were studied using the same parameters, which made it possible to objectively compare and interpret the materials used, the subsequent superimposed layers and the transformations related to renovation and then conservation of the objects over a period of almost 400 years. The following features were compared: the canvases used, their deformation and the way of attaching them to the stretchers, as well as evenness of ground layers – the accuracy of canvas sizing.

The images were used for the initial interpretation of paint layers, ways of building the portraits and changes introduced by the authors. It was possible to read the effect of various binders in the paint layers, which after drying resulted in a diverse mesh of cracks, unrelated to the condition in which the objects have been preserved or to the passage of time.

The paintings were a perfect example of the transformations related to subsequent renovations, which often involved painting over the form of the elements of the works, introducing new signs or moving the existing signs in connection with format changes.

Apart from changes related to the paint layers, the paintings have undergone technical processes, such as reinforcement of painting supports, filling of losses, and use of new stretchers. While comparing the film images, it was possible to systematise the materials used by comparing their absorption. Fragments

[1] Early modern portraits of the University of Krakow professors in the collection of Collegium Maius, Anna Jasińska, ed. Cracow 2010

of additional canvas pieces, known as patches, and the signs covered between the original canvass and the lining canvass became readable.

The x-ray images, taken using a classic method but processed by means of modern computer techniques, made it possible to visually bring out individual elements with greater accuracy, such as one portrait painted over another. It was also possible to read the percentage of the oldest depiction preserved. Interpretation of the paintings based on x-ray images is very important but it could not have been accomplished without other tests and without knowing the technology of the objects. It also helped to make the decisions concerning the planned conservation and develop the related scope of activities.

X-ray imaging is an auxiliary investigation which offers an opportunity to study a work of art in its different aspects, as signalled above. In the case of two 17th century portraits, showing Wojciech Dąbrowski and Łukasz Piotrowski, Jagiellonian University professors, a film image analysis was used as an auxiliary investigation while identifying the authorship of the portrait attributed to Jan Tricius. When compared to colour images of the same fragments of the paint layer, images of selected frames of the films made it possible to observe the artist's proficiency at the oil painting technique. They permitted capturing the author's individual characteristics left in the brushwork, his expert way of guiding the brush, the brush pressure, all of which show the professionalism and reflect the temperament of the artist. At the next stage, the results of image analysis were combined with the results of other studies. The two paintings were compared to each other.

Chemical, physical and technological tests are particularly valuable when they provide information that first and foremost facilitates dating the object, when they make it possible to follow the history of the changes to the object, and when they deliver a wide range of information which, when considered together with archival information and knowledge of an art historian, may allow researchers to attribute an object to a particular technique or even a particular artist.

Those are the fundamental problems the resolution of which brings the greatest satisfaction; however, in the course of work one encounter also other, perhaps less spectacular, information which makes it possible to better study the object, thus giving rise to further search.

THE COMPUTED TOMOGRAPHY OF THE 15TH CENTURY MADONNA WITH A CHILD FROM KSIĄŻNICE WIELKIE AS AN EXAMPLE OF NON-INVASIVE METHOD OF EXAMINATION OF A MASTERPIECE

Dagny Dobrowolska
Private practice in the sculpture conservation, Krakow, Poland

The goal of the presentation is to show scientific possibilities connected with using computed tomography as a non-invasive method of examination of a piece of art. Results of such examination of the 15th century wooden sculpture of Madonna with Child from Książnice Wielkie are discussed.
A wooden sculpture of Madonna with Child (2nd and 3rd quarter of 15th century) has been partly transformed several times. All the transformations may be divided to at least three stages. The first one took place probably in the Baroque period, the second one was a renovation of the sculpture that took place in 1808, finally, probably at the very beginning of the 20th century (the last stage), two wooden crowns were put on Madonna's and Child's heads. Exact changes that were made – especially in the 19th century – were impossible to establish. In particular, differences between the original sculpture and the later insertions were obvious in many parts, but in some areas it was difficult to say which part was original and which part was added later. To examine this problem properly, it was decided to use the computed tomography.
The results of the examination made it possible to define the precise size of each of the 19th century wooden insertions, their present position inside the figure and their proportion to the preserved, original Gothic part of the sculpture. They also gave a lot of new information about the technology of the Gothic sculpture as well as the technology of the restoration that took place in 1808. The cross – sections also showed how well the particular parts of the sculpture are preserved – including the deterioration caused by insects, the remains of paint and gesso layers, etc.
The computed tomography of the 15th Madonna with Child from Książnice Wielkie shows that this type of examination can be effectively used in the contemporary art restoration. The examination is totally non-invasive and as such, it is safe and it cannot damage a piece of art. Moreover, the examination parameters can be precisely chosen and adjusted to a given object, which enables to examine the object in a very exact way and in many aspects. Technology, state of preservation, and possible transformations can be identified. The tomography may be used for a wide variety of materials like wood, metal, stone, etc. Moreover, a 3D view of an object, created on the basis of tomography, gives additional possibilities for the documentation of state of preservation of a piece of art.

APPLICATION OF X-RAY DUAL SOURCE COMPUTED TOMOGRAPHY (DSCT) IN INVESTIGATION OF THE OBJECTS OF CULTURAL HERITAGE

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For over a century, X-ray radiation has played an important role in the area of the conservation and restoration of cultural heritage objects. X-ray techniques are amongst the most fundamental and helpful methods used in the investigation of art works.

This paper reviews the application of X-ray Dual Source Computed Tomography (DSCT) in the investigations of wooden paintings and sculptures.

The idea behind Dual Source CT is quite simple: it is merely using two X-ray sources (at two different kV levels simultaneously) and two detectors at the same time. Using such combination, it is possible to get double temporal resolution, double speed, and twice the power, while lowering dose even further. The result are two spiral data sets acquired in a single scan providing diverse information. Dual Source CT provides images of exceptional quality and is an amazing tool not only in medicine but also in the area of cultural heritage.

As a result, 2D and 3D images, permitting the full volume inspection of an object, were taken in totally non-destructive way. The morphological and physical information about the inner structure of the investigated wooden objects were obtained, revealing changes related to previous restorations, as well as ageing effects.

SZCZERBIEC (THE JAGGED SWORD) – THE CORONATION SWORD OF THE KINGS OF POLAND

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The paper presents results of the newest research of Szczerbiec – the Polish coronation sword. Technological examinations revealed that the blade was manufactured of bloomer steel, with C contents of c. 0.3-0.6 %. The blade was thermally treated (quenched and tempered) in its part bellow the hilt. The X-ray revealed no pattern welding or composite structures. All this testifies of the authenticity of the sword as a genuine medieval weapon and not (as sometimes suggested by scholars) a 19th c. replica. The pommel and the crosspiece were made of silver and then coveted with nielloed gold plates.
A combination of typological , stylistic and epigraphic data suggest a date of c. 1250 for the sword. As suggested by the lavishness of ornament and inscriptions on the all-metal hilt, as well as by the rectangular cross-section of the grip, the Szczerbiec may have been influenced by swords of the Mediterranean (esp. Iberian) cultural sphere. The swords of Sancho IV of castle an Leon, of Santa Casilda, of Friedrich II von Hohenstaufen, of the Comtes de Dreux and a sword from the Museo Arqueologico Nacional in Madrid seem to be especially relevant analogies. Of particular significance is a Hebrew or Hebrew-Latin inscription on the crosspiece, which fits into a tradition of Hebrew- inspired *voces magicae*. The first owner of the sword was, in all probability, Bolesław Pobożny (*the Pious*), Duke of Great Poland (died 1279). For Duke Bolesław, the sword was his *gladius iustitiae* and a protective talisman. The weapon was then inherited (probably trough marriage to his daughter Jadwiga c. 1239) by Duke Władysław Łokietek (the Short or the Ell-High) the future King of Poland. It was, in all probability, Duke Władysław who first use the sword as a coronation insignia.

2D MICRO-DIFFRACTION USING PIXcel^{3D}

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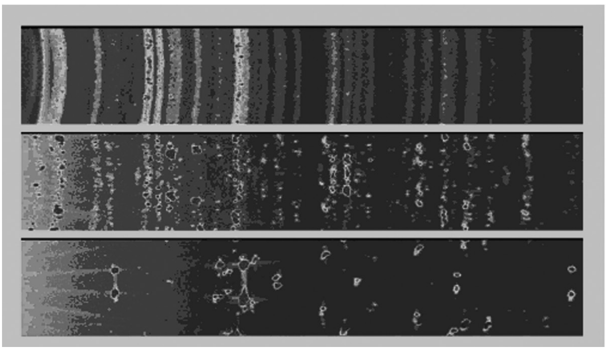
The unique capabilities of the PIXcel^{3D} detector allow micro-diffraction experiments with 2D detection capability on multi-purpose diffractometers, without the need to switch to other detectors for conventional powder diffraction measurements or high resolution applications. Using 2D detection system in micro-diffraction experiments, the diffracted beams appear as arc, known as Debye rings. The rings provide the user with an instant window to the microstructure of the sample giving hints about the homogeneity of the sample (mapping experiment), the grain size and orientation distributions. Additionally, 2D micro-diffraction gives information about the phase composition in sub-mm^2 spots in solid objects. In a polycrystalline sample, only those grains that have the right orientation to the incident beam will meet Bragg's law and generate a diffracted beam.

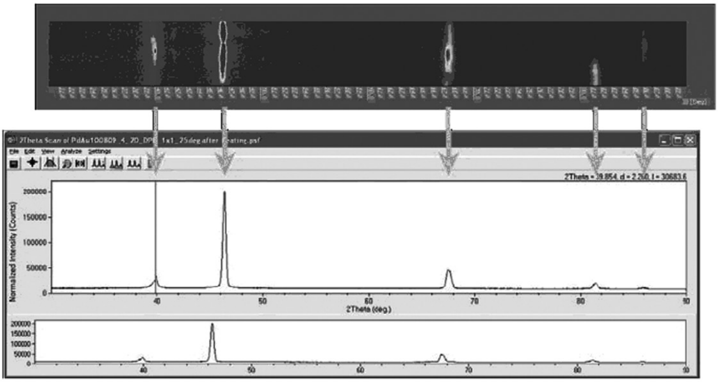
In micro-diffraction experiments, these diffracted beams originate form a small spot an fan out around the incident beam direction forming a cone with half angle 2theta. The 2D detector intercepts the cone of diffracted rays. An arc is formed on the detector. This arc is called a Debye ring.

Even if there are very few crystals diffracting and isolated spots are observed the spots due to diffraction by the same set of planes will be positioned on the ring. Depending on the crystallite size, the ring can be anything between smooth and spotty, with a single crystal being the most extreme case.

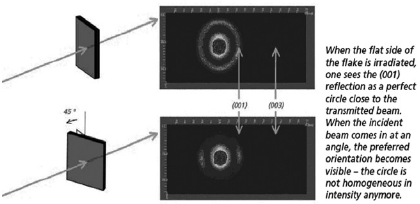


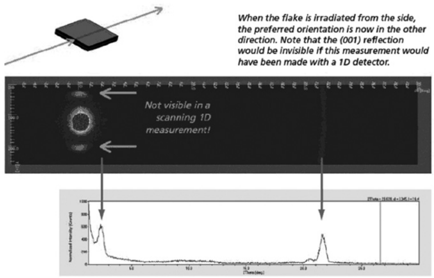
2D micro-diffraction images obtained on the Empyrean showing Debye rings characteristic of sugar grains. From top to bottom: fine, medium and coarse grained samples.

In scanning measurements, the information from the 2D frames is summed up as a function of 2theta to provide a 2theta strip file. By integrating along the arcs in the strip file, one can obtain a conventional one-dimensional measurement, which can be used for further analysis, e.g. in HighScore (Plus).



Transmission and reflection measurements near the primary beam.





The PIXcel^{3D} detector allows diffractionists to switch from conventional 1D to scanning and static 2D measurements without having to switch to a different detector or instrument. In the 2D mode, one can freely optimize between resolution and field of view simply by varying the detector-to-sample distance. Thanks to the solid-state detection technology, these changes can be made without the need for calibration by the user. The very high dynamical range of the detector allows measurements near the direct beam without the need of a direct beam stop.

MINIFLEX – NEW BROTHER OF RIGAKU X-RAY DIFFRACTION FAMILY

Andrzej Węsek
Testchem, Pszów, Poland

Among wide variety of Rigaku X-ray diffractometers MiniFlex is the smallest one. Even if small in size, its analytical performance is comparable with that of bigger instruments.

Because of growing interest in that class of instruments, Rigaku has recently introduced MiniFlex 600, a new bench-top X-ray diffractometer with medium power X-ray tube and high analytical performance. The quality of results is shown on analysis of a prehistorical ceramic sample measured on both MiniFlex 300 and the new MiniFlex 600 diffractometers.

ABSTRACTS

POSTERS

CONSERVATION OF THE PAINTING THE SERMON OF JOHN THE BAPTIST BY PIETER II BRUEGHEL THE YOUNGER FROM THE CRACOW COLLECTION OF THE NATIONAL MUSEUM

Katarzyna Novljaković, Anna Grochowska-Angelus, Małgorzata Chmielewska, Urszula Węgrzynowicz
National Museum, Krakow, Poland

The painting Sermon of John the Baptist is attributed to Pieter II Brueghel the Younger by Klaus Ertz, the researcher of Brueghel's œuvre. In Polish art collections, is one of two works by this painter – Winter Landscape with Skaters and Bird Trap belonging to the National Museum in Wrocław.
Our painting is created in mixed mediums on oak panel composed of three horizontal pieces and an established example of Flemish workshop of the 17 th century, dated by art historians from 1601-1604. Pieter II Brueghel the Younger, was the oldest son of the Pieter Bruegel the Older, known as a follower and copy maker of his very known father, created in the second half of 16th century. The original of Sermon of John the Baptist, painted by Bruegel the Older in 1566, is displayed at the Szépművészeti Múseum in Budapest, and was copied many times by son and his prolific workshop.
In 2009-2011 a team of conservators had contributed general restoration of the masterpiece, accompanied by scientific examination and analysis made by the National Museum Laboratory LAMBOZ and the Conservatory Chemistry Institute of the Faculty of Conservation and Restoration of Art Works the Academy of Fine Arts in Cracow. Our observations and the results of XRF, sample sections and photography of luminescence under UV rays and infrared, helps to identify the technological structure of this painting. Very complex work of Conservatory treatment included revealing from all old conservation on the surface of the painting, local consolidation of the paint layer, largely ground fillings, retouches and reconstructions. Details of the original painting by Peter Bruegel the Older help to make the reconstructions more precise.

X-RAY POWDER MICRO-DIFFRACTION INVESTIGATIONS OF SELECTED PIGMENTS IN SEVENTEENTH – CENTURY PAINTINGS

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The study was focused on identification of the components of paint layers in Anton Möller (1563/65–1611), Isaak van den Blocke (before 1589–1628) and Hermann Han (1580–1627/28) paintings. These three artists lived in Gdańsk (Poland) and are best known for biblical themes and allegorical compositions. The examined samples were taken from two Möller's paintings: 'Tribute Money', 1601 from the Gdańsk History Museum; 'Foundation Table', 1607 from St Mary's Church in Gdańsk; and two paintings attributed to his circle: 'Allegory of Wealth', circa 1600 from the National Museum in Gdańsk and 'Allegory of Wealth' and 'Model of the world', circa 1600 from the National Museum in Poznań. We have also examined one sample from Blocke's painting "Servilius and Appius" (1608–1609) from the Main Town Hall in Gdańsk. Samples from Han's three paintings:: 'Assumption of Mary', 1618 from the Cathedral in Pelplin; 'Coronation of Mary', painted after 1624 and 'Prussian attack on the monastery in Oliwa', circa 1613 from the Cathedral in Oliwa were studied with the use of X-ray powder micro-diffraction technique.
All the measurements were performed on cross sections. Due to the fragile nature of panel paintings, very small samples of paint layers and ground layers were taken from the paintings and mounted in a synthetic resin. XRPD micro-diffraction measurements were performed on the cross-sections. White, yellow, red, blue and green paint layers were examined.
The XRPD measurements were carried out on an X'PERT PRO MPD diffractometer, Cu Kα radiation, 40kV and 30 mA, a graphite monochromator and a PIXCEL PSD detector. The apparatus was equipped with a collimator (output beam diameter 0.1 mm) which allowed to perform the micro-diffraction measurements. Phase analysis and pigment identification was done with the use of the PDF4+ database. The measurement set-up and obtained results (identified pigments, fillers and different types of lead white) will be presented in the poster.

APPLICATION OF XRF TECHNIQUE FOR PROVENANCE STUDY OF HISTORICAL BRONZE AQUAMANILE

Iwona Żmuda-Trzebiatowska[1], Anna Fietkiewicz[2] and Gerard Śliwiński[1]
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In frames of an authenticity and provenance study of historical bronze aquamanile from the collection of the National Museum in Gdansk, the need of analysis of the chemical composition appeared due to several areas on the object surface, characterized by a colour different from that observed on the prevailing part of the surface. Fulfilling conservator's requirements which allowed a non-destructive approach only, the X-ray fluorescence spectroscopy (XRF) was applied. From the measurements performed on different locations on the object's surface, a series of spectra was collected. The qualitative analysis performed by means of the PCA statistical technique revealed two different elemental compositions of the bronze in dependence on the spot position analyzed on the surface. Next, the quantitative XRF analysis of main elements of the historic alloy was carried out using reference samples of bronze alloys. The alloy composition of Cu, Zn, Sn and Pb of the artifact original material was estimated at 85%, 18%, 3,2% and 1,5%, respectively, while the differently coloured part contained 62,5% Cu, 29,4% Zn, 0,5% Sn and 0,004% Pb. From this difference in the chemical composition and also microscopic observations, serious past conservation intervention and repair of local damages of the aquamanile surface were concluded. In order to improve the confidence level of the analysis, complementary measurements were performed on the invisible object part by means of the micro-destructive laser-induced breakdown spectroscopy technique (LIBS). The concentrations of 11,27 % Pb, 8.25 % Sn and 17 % Zn relative to Cu was obtained from spectral intensities of lines Pb (I) 405,78 nm, Sn(I) 326,24 nm, Zn(I) 481,04 nm, and Cu (I) 521,80 nm and were in good agreement with the XRF data.

XRPD INVESTIGATIONS OF THE ANCIENT COINS AND OTHER METAL OBJECTS

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The aim of this study was examination of the suitability of various modern physico-chemical methods such as Scanning Electron Microscopy (SEM), X-ray Fluorescence (XRF), X-ray Powder Diffractometry (XRPD), Proton Induced X-ray Emission (PIXE) or Laser Ablation Inductively Coupled Mass Spectrometry (LA ICP-MS) in determination of the chemical composition and origin of metal alloys. In our research, we have tried to develop a set of characteristics (based on chemical composition, density, and special features of the surface), indicating the authenticity of the ancient coins.
Apart from archeology and numismatics, this research is important in testing the instrumental analytical methods for specific uses and samples, such as precious metals. The undertaken research is also useful in the study of metal corrosion, in the study of destruction and wear of coins being in circulation, and in the knowledge about the manufacturing processes. It can also be applied to detect forgery and counterfeiting of metal objects.
We have investigated authentic Roman coins, Russian coins (1914) and a few coins of questionable authenticity. It was found that both XRPD and density determination methods can be used as a fast and non-destructive way of initial studies. Final reliable conclusions can be drawn only on the basis of the full range of scientific methods, supported by a deep knowledge of archeology.
We have also studied lead seals (named lead bullas) from Grody Czerwieńskie archaeological site. The goal of this study was the determination of chemical composition of an unusual red-brown layer which was found on numerous seals. The results of the undertaken investigations will be presented in the poster.

MICRO- PIXE AND MICRO- COMPUTER TOMOGRAPHY INVESTOGATION ON ELEMENTS OF A PORCELAIN VASE

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The micro-PIXE and micro-Computer Tomography (CT) methods are powerful tools for non-destructive analysis of various rare objects, e.g. works of art. In this study, both methods were used to investigate the elemental composition of a painted porcelain vase from the Łańcut Castle Museum in Poland. The vase had been broken accidentally into almost 90 bigger and smaller pieces, and its reconstruction started in 2007. As it was impossible to fit a few small elements (a few mm) into their original places, they have been stored separately. This apparent misfortune in reconstruction allowed to perform the analysis of trace elements present in pigments used for the vase painting, which is often a clue to the artwork history. The available porcelain samples were scanned with the 2 MeV proton micro beam from the Van de Graaff accelerator and with the X-ray beam from the Hamamatsu tube. The PIXE method revealed the presence of various amounts of Al, Si, K, Ca, Ti, Mn, Fe, Cu, Sn, and Pb in red, green, blue, yellow, black and white-grey paints.
A series of PIXE scans allowed to produce maps of the elemental composition of the painted surface and the depth cross-sections. The micro-CT revealed inhomogeneous density of the porcelain body and internal details. All these data correlate well with the independent historical information about the object (China, 19th century).

TIN AND TINNED DRESS ACCESSORIES OF MEDIEVAL INHABITANS FROM WROCŁAW: PRELEMINARY RESULTS OF X-RAY FLUORESCENCE

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Most of what we know about medieval clothing elements and jewellery comes from portraits, pictures in manuscripts and artifacts from the museum collections. But this knowledge was focused rather on the precious accessories (and jewellery) that adorned knights and nobles. It was, nevertheless, important for all social groups to look "*fashionable*" and to wear jewellery. To women and men of the Middle Ages dress elements were not only the indication of social status, but they were about the religious context too. The jewellery motifs were often devotional: crosses, pilgrims badges (often worn by men on the hat). Women displayed pendants, rings, belts and brooches, which were often wedding gifts [1]. Clothing accessories were made of very different metals: noble metals (gold, silver) for wealthy men and cheaper metals for others. The raw materials which were used to produce the latter were bronze, tin, lead and/or lead-tin alloy [2]. The imitation of noble jewellery is very interesting and will be the subject of this project. Several artifacts after their conservation were selected for detailed studies: temple rings, pendants, beads, rings, brooches, buttons, belts, spurs, buckles, knives, which were found during archaeological excavations in Wrocław (especially in Nowy Targ square in 2010–2011). The research project is focused on the determination of chemical composition of the artifacts. The next step will be to identify similarities and differences between alloys and to determine the origin of the workshop and tin raw materials (in Lower Silesia, they used tin from local sources, as well as imported from Bohemia).

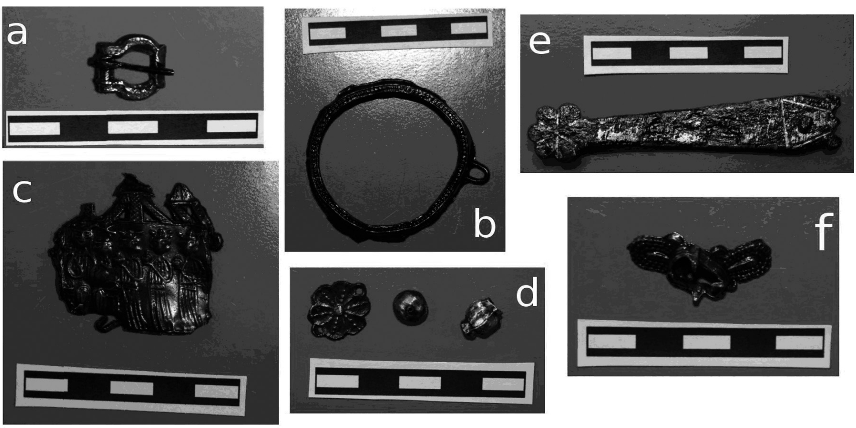

Fig.1 Medieval artifacts from Wrocław (Nowy Targ Sq.): a. buckle, b. frame, c. badge, d. mounts, bead, e. strip end, f. part of jewellery.

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POSSIBILITIES OF THE SEM/EDS TECHNIQUE APPLICATION FOR IDENTIFICATION OF TANNINS AND OTHER COMPOUNDS IN HISTORICAL LEATHER

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Different methods of animal skins processing into leathers were invented through the ages. A variety of leathers obtained from archeological excavations, exhumations, library and archival materials, museums (etc.) are the objects of conservation-restoration treatments. Identification of types of tannins is a basic information for conservators' before treatments start, as well as for planning storage conditions and display possibilities after treatments in the future. Many kinds of tannins have been used in different cultures of the world since the earliest times to the present. Objects made of untanned (i.e. processed without tannins) leathers occur also in collections and among finds. The SEM/EDS technique is in common use for identification of elements in samples from different historical objects. The authors presents possibilities of application of the scientific equipment for examination of structure, chemical composition, and elements distribution in samples on selected leather examples. Not only tannin identification will be presented, but an issue of additional compounds present in leather samples will be emphasized. Tin and tinned dress accessories of medieval inhabitants from Wrocław. Preliminary results of X-ray fluorescence.

X-RAY POWDER DIFFRACTOMETRY IN INVESTIGATIONS OF YELLOW EARTH PIGMENTS

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Among numerous X-ray techniques (radiography, SEM/EDX, XRF), X-ray powder diffractometry (XRPD) is increasingly popular in investigations of works of art. XRPD is used for identification of pigments (mineral or synthetic), ceramics, construction materials, alloys and their corrosion products. Each crystalline material gives a characteristic diffraction pattern. XRPD allows to identify various substances, even in the mixtures of several compounds, which is often the case in the study of samples from historical objects.

The most important advantage of XRPD techniques is their ability of differentiation and identification of compounds which have similar elemental composition but different crystal structures.

It is believed that natural colour of majority of earth pigments comes from iron. When Fe is not presented in earth pigments, they are usually white. If iron is present in the oxide form, the colour of the pigment is yellow, but when iron is incorporated in the crystal lattice of clay minerals (seladonite, glauconite, smectite), the earth pigment may be green. The composition of natural earth pigments depends on the composition of the rocks from which they have been received. This information may be helpful in determination of the origin of an art object.

The aim of the presented study was to define the composition of several yellow earth pigments in order to prepare a small data base for further identification of this pigment in paintings. In the study, seven samples dated to the late 17th to the second half of 19th century were investigated.

The XRPD measurements were carried out at an X'PERT PRO MPD diffractometer, CuKα radiation, 40kV and 30 mA, a graphite monochromator and a PIXCEL PSD detector. The phase analysis was performed with the use of the PDF4+ database. The results will be presented in the poster.

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