
3rd Meeting X-ray techniques in investigations
of the objects of cultural heritage
Around Rembrandt and his workshop
Krakow (Poland) 13-16 May 2010

Jagiellonian University

in Krakow

Institute of Catalysis and

Surface Chemistry PAS

International Union of Crystallography, Crystallography in Art and Cultural Heritage Commission

3rd MEETING

X-RAY TECHNIQUES IN INVESTIGATIONS

OF THE OBJECTS OF CULTURAL HERITAGE

AROUND REMBRANDT AND HIS WORKSHOP

Krakow, 13-16 May, 2010

The aim of the Meeting is to promote the development and use of X-ray techniques in order to extract information from the objects of cultural heritage. It is also a forum to bring together scientists, whose major expertise is in a high-tech field, and museum professionals (conservators and curators) whose particular responsibility is the organization and preservation of collections, and to spread the newest results in the area of scientific investigations of art objects. First day of the meeting will be focused on the investigations of Rembrandt paintings.

Organizers:

Faculty of Chemistry Jagiellonian University
30-060 Krakow, ul. Ingardena 3, POLAND
www.chemia.uj.edu.pl

Jagiellonian University Museum
31-010 Krakow, ul. Jagiellońska 15, POLAND
www.maius.uj.edu.pl

Institute of Catalysis and Surface Chemistry PAS
30-239 Krakow, ul. Niezapominajek 8, POLAND
http://atom.ik-pan.krakow.pl

International Union of Crystallography, Crystallography in Art and Cultural Heritage Commission
http://www.iucr.org/iucr/commissions/ccach

International Scientific Committee:

Prof. Henk Schenk, Amsterdam University, the Netherlands,

Dr Eric Dooryhee, Centre National de la Recherche Scientifique, France,

Dr Thomas Wroblewski, Deutsches Elektronen-Synchrotron, Germany.

Organizing Committee:

Prof. Grażyna Stochel

Prof. Małgorzata Witko

Prof. Roman Kozłowski

Anna Jasińska

Prof. Stanisław Waltoś

Prof. Wiesław Łasocha

Alicja Rafalska-Łasocha

Jolanta Pollesch

Secretary of the Meeting:

Alicja Rafalska-Łasocha

Sponsors:

PANalytical B.V. Branch Office Poland

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Programme

<u>13 May 2010, Thursday</u>

14.00 - 17.00

Registration: Collegium Maius – Jagiellonian University Museum, Jagiellońska str. No. 15, Krakow.

17.00 - 17.15

Opening of the Meeting

17.15 - 18.15

"Climate change effects on Europe's cultural heritage: challenges and possibilities" Roman Kozłowski, Polish Academy of Sciences, Krakow, Poland

18.30 - 21.00 **Get together party in the Cellars of Collegium Maius**

<u>14 May 2010, Friday</u>

Around Rembrandt and his workshop

Chair: Prof. Roman Kozłowski, Polish Academy of Sciences

9.30 - 10.30

"Neutron-Activation-Autoradiography of Paintings by Rembrandt at the Berlin Picture Gallery" Claudia Laurenze-Landsberg, Gemäldegalerie der Staatlichen Museen, Berlin, Germany

10.30 – 11.30

″**Two of Rembrandt's paintings: 'Girl in a picture frame' and 'Scholar at his writing table' from the Warsaw Royal Castle Collection – history, examination and conservation"** Joanna Czernichowska, Regina Dmowska, Anna Nowicka, Royal Castle in Warsaw, Poland

11.30 - 12.00 **Coffee break**

12.00 - 13.00

”**Rembrandt's 'Landscape with Good Samaritan' from the Czartoryski Collection – observation and technical information after restoration”** Anna Grochowska-Angelus, Katarzyna Novljakovic, Dorota Dec, Maria Rogóż, Krakow National Museum, Poland

13.00 - 14.00

Poster Session and Visit to the Conservation Workshop in Collegium Maius

14.00 - 15.00 **Lunch**

Chair: Prof. Zbigniew Sojka, Faculty of Chemistry Jagiellonian University

15.00 – 16.00

”**Scientific analyses in determining original appearance. Studio and Rembrandt, *Saul and David,* c. 1660, oil on canvas (H: 131 x L: 164 cm)”** Petria Noble, Royal Picture Gallery Mauritshuis, The Hague, The Netherlands

16.00 – 16.45

“Is portrait of 'Young Man' from the Cracow Royal Castle Collection by Jan Lievens?” Joanna Winiewicz-Wolska, Ewa Wiłkojć, Wawel Royal Castle, Krakow, Poland

16.45 - 17.30

“Portrait of Joost van den Vondel’ by Philips Koninck from the Jagiellonian University Museum Collection. Attribution and identification” Anna Jasińska, M. Krąpiec, Jolanta Pollesch, Beata Skalmierska, Jagiellonian University Museum, Krakow, Poland

17.30 - 17. 45 **Coffee break**

17.45 - 19.00

Visit to the Collegium Maius Collection

15 May 2010, Saturday

X-ray techniques in investigations of art objects

Chair: Prof. Henk Schenk, Amsterdam University, the Netherlands,

9.00 - 10.00
”**Use of X-ray powder micro-diffraction for identification of local sources of painting materials”** Petr Bezdička, Silvie Švarcová, David Hradil, Janka Hradilová, Academic Materials Research Laboratory of Painted Artworks (ALMA) Institute of Inorganic Chemistry of the ASCR, Husinec-Rez, Czech Republic

10.00 - 10.45
”**Portable digital X-ray radiography system for studies of historical objects**, Piotr Frączek, Joanna Sobczyk, Łukasz Bratasz, Janusz Czop, National Museum in Krakow, Poland

10.45 - 11.15 **Coffee break**

11.15 - 12.00

“X-RAY POWDER DIFFRACTOMETRY FOR STUDIES OF HISTORICAL OBJECTS - A NEW EQUIPMENT AT FACULTY OF CHEMISTRY JAGIELLONIAN UNIVERSITY”

M. Oszajca, M. Grzesiak, K. Podulka, A. Rafalska-Lasocha, W. Lasocha, *Faculty of Chemistry Jagiellonian University, Krakow, Poland*

12.00 - 13.00

Sponsors’ presentations

13.00 - 14.30 **Lunch**

Chair: Prof. Wieslaw Lasocha, Faculty of Chemistry, Jagiellonian University, Krakow, Poland

14.30 - 15.15

“Recovering Erased Scripts from Palimpsests: First results from X-Ray flourescence element mapping experiments” L. Glaser, D. Deckers , G. Falkenberg , C. Mackert, C. Brockmann and D. Harlfinger, *Deutsches Elektronen-Synchrotron DESY, Hamburg, Germany*

15.15 - 16.15 **Closing lecture**

“Rare Silverpoint Drawings by Rembrandt in the Focus of SR-XRF” Ina Reiche, Silke Merchel, Martin Radtke, Heinrich Riesemeier, Holm Bevers, *Centre de Recherche et de Restauration des Musées de France - CNRS UMR 171, PARIS*

16.15 -16.30 **Coffee break**

16.30 - 18.00 Science Festival in Krakow

19.00 - 23.00
Meeting Dinner in the cellars of Collegium Maius

<u>16 May 2010, Sunday</u>

Visit to the historic Salt Mine in Wieliczka

Poster presentations:

1. **REMBRANDT IN LABORATORY. FACTORS OF DETERIORATION OF OIL PAINTINGS CONTAINING LEAD WHITE.**
Dawid Popławski, Maria Poksińska, *Nicolaus Copernikus University, Torun, Poland*

2. **DIGITAL PROCESSING OF X-RAY RADIOGRAMS FOR CONSERVATION PURPOSES**, **Joanna Sobczyk, Piotr Frączek, Jakub S. Prauzner-Bechcicki, Łukasz Bratasz, Janusz Czop,** *National Museum In Krakow, Poland*

3. **MULTI-METALLIC ARTEFACTS FROM WROCŁAW -EDXRF STUDIES, Beata Miazga**, *University of Wrocław, Institute of Archaeology, Poland*

4. **APPLICATION OF X-RAY METHODS FOR PIGMENTS IDENTIFICATION IN COLLECTION OF RAFAŁ HADZIEWICZ PAINTINGS, A. KLISIŃSKA-KOPACZ, A. RAFALSKA-ŁASOCHA, E. ZYGIER and J. CZOP**, *National Museum in Krakow, Poland,*

5. **X-RAY BASED ANALYSIS OF THE METAL THREADS IN HISTORICAL TEXTILES,**
A.Klisińska-Kopacz, M. Włodarczak, A. Prokopowicz and Ł. Bratasz, *National Museum in Krakow, Krakow, Poland*

6. **WITKACY'S PASTELS ANALYSIS FOR SAFE ANOXIA STORAGE, A. Klisińska-Kopacz, J. Sobczyk, J. Thomas, Piotr Frączek, D. Godyń, Ł. Bratasz, J. Bagniuk, T. Łojewski,** *National Museum in Krakow, Krakow, Poland*

7. **XRD and SEM/EDX INVESTIGATIONS OF THE CORROSION PRODUCTS OF METAL THREADS FROM THE TAPESTRY OF SIGISMUND AUGUSTUS**, **Alicja Rafalska-Łasocha, Jerzy Holc, Anna Bielańska, Wiesław Łasocha** , *Faculty of Chemistry Jagiellonian University, Krakow, Poland*

8. **XRD INVESTIGATIONS OF COMMERCIALLY AVAILABLE PRUSSIAN BLUE PIGMENTS AND OIL PAINTS, Alicja Rafalska-Łasocha, Katarzyna Podulka, Roman Dziembaj, Wiesław Łasocha,** *Faculty of Chemistry Jagiellonian University, Krakow, Poland*

9. **SYNCHROTRON X-RAY POWDER MICRO-DIFFRACTION and MICRO-FLUORESCENCE FOR IDENTIFICATION OF GREEN PIGMENTS IN the GOTHIC ALTAR PAINTINGS FROM MALOPOLSKA REGION, Alicja Rafalska-Łasocha, Marta Grzesiak, Zofia Kaszowska, Thomas Wróblewski, Dariusz Zając, Andre Rothkirch, Wiesław Łasocha,** *Faculty of Chemistry Jagiellonian University, Krakow, Poland*

10. **CONSERVATIONAL ANALYSIS OF THE SIGISMUND BELL***,* **Marcin Biborski[1] and Andrzej Biborski***, Institute of Archeology, Faculty of History, Jagiellonian University, Krakow, Poland*

11. **AN INTERESTING DILEMMA – A SMALL LANDSCAPE SIGNED REMBRANDT F. 1627. A DEPOSIT IN THE JAGIELLONIAN UNIVERSITY MUSEUM COLLECTION**, **Anna Jasińska, Jolanta Pollesch, Beata Skalmierska**, *Jagiellonian University Museum, Krakow, Poland*

A B S T R A C T S

CLIMATE CHANGE EFFECTS ON EUROPE'S CULTURAL HERITAGE: CHALLENGES AND POSSIBILITIES

Roman Kozłowski

Institute of Catalysis and Surface Chemistry Polish Academy of Sciences, Krakow, Poland.

The climate parameters in the environment of cultural heritage objects can have a profound effect on changes occurring in the historic materials and consequently on their preservation. Objects expand and contract as the temperature or humidity change, are mechanically damaged by the melting and freezing of water, or dissolution and crystallisation of salts in response to impacts of the outdoor environment. The rates of some important chemical reactions, such as the degradation of cellulose in paper and textiles, increase with rising temperature. Temperature and time of wetness influence the activity of fungi and insects responsible for the biodeterioration of organic materials.

Climate change is now widely recognized as the major environmental problem facing the globe. Addressing climate change is also central to the work of the European Union which has launched research initiatives aiming at the assessment of damage potential of climate change on European cultural heritage, as well as the development of possible mitigation strategies. The Noah's Ark project, implemented between 2004- 2007, focused on the impact of different climate parameters on broadly defined heritage and sought to improve the protection strategies of heritage materials, structures and infrastructures. In November 2009, the "Climate for Culture" project was initiated. It will last five years and will develop high resolution climate evolution scenarios and couple them with whole building simulation models to identify the most urgent risks for various European regions.

The presentation will identify different climate parameters that pose risk to cultural heritage, as well as provide information on the vulnerabilities of the European regions to climate change based on the climate change scenarios. The impact of climate on outdoor and indoor heritage materials will be discussed. Historic wooden structures and decorated wooden objects will be selected as an example of vulnerable historic objects both outdoors due to the biological attack by wood destroying fungi and indoors due to physical damage caused by variations mainly in ambient relative humidity.

Principles of mitigation and adaptation will be discussed. The cultural heritage sector shares responsibility for the environment, and it will need to reduce the impact on the environment, primarily using energy more efficiently. Measures that are necessary to adapt museums and historic buildings to a changing climate will be analysed. The capacity of the built heritage to adapt to climate change should be maximised by changes in both the management of cultural heritage as well as physical changes.

AROUND REMBRANDT AND HIS WORKSHOP

NEUTRON-AUTORADIOGRAPHY OF PAINTINGS BY REMBRANDT IN THE GEMÄLDEGALERIE BERLIN

Claudia Laurenze-Landsberg[1], C.O. Fischer[2]

[1]Staatliche Museen Gemäldegalerie, Berlin, Germany,

[2]Helmholtz-Zentrum für Materialien und Energie, Berlin, Germany.

The research using the Neutron-Autoradiography method is in collaboration with the Helmholtzzentrum für Materialien und Energie, Berlin, formerly called the Hahn-Meitner-Institute, and the Gemäldegalerie of the Staatliche Museen Berlin. The Gemäldegalerie Berlin is the only institute world wide, which systematically employs the method of Neutron-Activation-Autoradiography to analyse paintings. Today we have investigated about 70 mainly 17th century works.

The paintings to be investigated are scanned by means of neutron activation. The isotopes arising during this process have specific half-lives and emit gamma and beta energies. For a period of up to six weeks after activation x-ray films are placed on the painting exposing them to the radiation. Hereby, paint layers, which vary in colour, can be separated on different films and supply valuable additional data to x-radiographs.

Deeper paint layers are made visible. In this way, it is possible to gain insight into the work process and the artistic approach of the painter. A hidden composition might reveal another artist’s influence on the painter, which might be important for the dating. The elucidation of the brushstroke can be read as the handwriting of the artist responsible for the painting. Examples for these various findings by using non-destructive autoradiography will be shown.

Furthermore the development of Rembrandt’s painting technique will be demonstrated. With the assistance of autoradiography it is now possible to be witness to Rembrandt‘s rejection of the contemporary practice of under modelling the whole composition in brown paint. This form of preliminary draft was only adhered to in the earliest painting we investigated. A year later he designs the composition on the panel, the umbra is already thinner and only partly but still flatly applied. Finally the flat design is replaced completely by a linear, evolving sketch which is broadly laid out. Although initially executed in umbra Rembrandt later preferred boneblack for these compositional sketches. This change might be related to his adoption of darker grounds. The distribution of the pigment smalt in the later works is also highly significant and attests to Rembrandt‘s understanding of the characteristic features of materials. While in his early works Rembrandt employed smalt as a coloured pigment only, he later used the special qualities of this pigment by exploiting its optical effects.

J.Kelch (GG), C.-O.Fischer (HMI), C.Laurenze (GG), W.Leuther (HMI), G.Pieh (GG), K.Slusallek (RF), Bilder im Blickpunkt: Der Mann mit dem Goldhelm. Eine Dokumentation der Gemäldegalerie in Zusammenarbeit mit dem Rhatgenforschungslabor und dem Hahn-Meitner-Institut Berlin, 1986

C.-O.Fischer, Claudia.Laurenze.Landsberg, et al., Neues zur Neutronen-Aktivierungs-Autoradiographie, Tizians „Mädchen mit Fruchtschale“ und die Verwendung von Neapelgelb, Restauro 6/99 pp 426

Claudia Laurenze-Landsberg, *Neutron activation autoradiography of paintings by Rembrandt at the Berlin Picture Gallery*, Conservation Science 2002, papers from the conference held in Edinburgh, Scotland 22 – 24 May 2002 pp 254;

Claudia Laurenze-Landsberg, "Neutron-Autoradiography of Two Paintings by Jan Vermeer in the Gemäldegalerie Berlin", Wolfgang Lefèvre, Inside the Camera Obscura – Optics and Art under the Spell of the Projected Image, Reprint 333, pp 211

Katja Kleinert und Claudia Laurenze-Landsberg, Auf der Suche nach einer optimalen Bildlösung. Zur Bildgenese von Rembrandts Gemälde „Simson und Delila", Rembrandt – Wissenschaft auf der Suche, Beiträge des internationalen Symposiums Berlin, 04. – 05.11.2006, Jahrbuch der Berliner Museen, Band 51, 2009, pp 147

TWO REMBRANDT PAINTINGS: GIRL IN A PICTURE FRAME and SCHOLAR AT HIS WRITING DESK FROM THE ROYAL CASTLE COLLECTION IN WARSAW –HISTORY, EXAMINATION AND CONSERVATION

Joanna Czernichowska[1], Regina Dmowska[2], Anna Nowicka[1]

[1] *Academy of Fine Arts, Warsaw, Poland,*

[2] *Royal Castle in Warsaw, Warsaw, Poland.*

In 1994, Warsaw Royal Castle was honored with a gift from the Lancoronsky Family; a priceless art collection with two panel paintings – formerly in the XVII c. royal collection – attributed to Rembrandt van Rijn (1606-1660): *Girl in a picture frame,* Inv. no. ZKW/3906, *Scholar at his desk,* Inv. no. ZKW 3905, both: oil on panel, c.105.5 x 76.5 cm., signed: *Rembrandt f/1641.*

In 2004-2006 both panels were examined, treated and reattributed by Prof. Ernst van de Wetering, head of the Rembrandt Research Project. The comprehensive examination programme concerning the technology of the paintings and the artist's work technique was carried out by laboratories in Warsaw and Krakow.

Both panels are single pieces of poplar, vertical grain, back bevelled along four sides, very little wraped; thicknesses vary: 20-16 mm (*Girl*), 14-19 mm (*Scholar),* trimmed to create *pendants.* Face surface not very smooth, back – stained; some numbers, seals and traces of framing. Panels sized and covered with ground layers: *Girl*- two layers: chalk in fat medium and lead white with chalk in fat medium too, smooth finished; *Scholar*- one layer: lead white in fat medium; light ochre imprimatura on both, no drawings or preliminary sketches prior to the present *tronies.*

The original paint layer pigments – examined by a cross-section and microscope analysis (also seen by K. Groen, co. RRP) – identified as: lead white mixed with chalk, red and yellow ochre, red lake, green earth, bone black, bituminous brown; also lead-tin yellow, smalt and malachite in the male portrait and cinnabar in the female portrait. The binding medium, typical for Rembrandt's works, was examined (using analytical instrumental methods: SEM-EDX, GC-MS, FT-IR): linseed and walnut oil – walnut oil has been found in both paintings. The paint layers in major parts of both paintings were in a good condition with the exception of the red dress of the *Girl* portrait – here the paint layer had developed a prominent raised craquelure, some of which was poorly attached to the panel, some had created tents, and some had flaked off; in this part some pentimenti were visible through the red colours. Surface coated with heavy layers of varnish, badly discoloured and surface grime – very disturbing, partially blanched, with poor saturation in dark areas; also some small losses, changed retouching, a few dents and scratches.

X-ray and IR examination as well as the cleaning process revealed the existence of an initial composition – a sketch for the portrait of another woman executed with a few black brushstrokes – which shows up in the background around the girl's head and chest (on the present portrait) as a result of the growing transparency of the upper paint layers. Most likely the appearance of this first sketch was the reason for the later overpainting, restoration and damage. *Scholar* signature – solid, done in wet paint, *Girl* – most likely added later, partially damaged.

Two varnish layers with old retouching and some overpainting were removed from both panels. In the case of the *Girl* this process was combined with local consolidation of the red dress cupping and cleavage area. Large scale overpainting of the upper background with the added column, frame, extremely heavy dress (in the most part) were removed as well as some smaller retouches on the hat, face, ears and right earring, hair, necklaces, belt and some on the hands – some overpainting had to be left due to damaging the original or the impossibility of distinguishing or removing it. Aquarelle, then Movilith AYAB in ethanol and Gamblin retouching was done over the dammar varnish.

Afterwards they were exhibited with great success at the most important Rembrandt Year, 2006, two venue exhibition *Rembrandt. Quest of a genius* in the Rembrandthuis (Amsterdam) and Gëmaldegalerie (Berlin).

REMBRANDT'S LANDSCAPE WITH GOOD SAMARITAN FROM CZARTORYSKI MUSEUM - OBSERVATION AND TECHNICAL INFORMATION AFTER RESTORATION

Anna Grochowska-Angelus[1], Katarzyna Novljakovic[1], Dorota Dec[1], Maria Rogóż[2],

[1]Krakow National Museum, Krakow, Poland,

[2]Academy of Fine Arts, Krakow, Poland.

Painting collections of Western Art in Poland are privileged to include the Rembrandt masterpiece *Landscape with Good Samaritan* owned by Cracow's Prince Czartoryski Museum. In Rembrandt Year, 2006, this distinguished painting was exhibited at the Museum Stedelijk De Lakenhal, Leiden, with other landscapes of the Master that have survived to this day, along with prints and designs by him.

The Czartoryski painting opened the show, having been a main attraction for many years behind the "Iron Curtain" during the communist era. The painter, Jean-Pierre Norblin de la Gourdaine, brought the painting from France in 1774, and has remained in the hands of the Czartoryski family since

1813. Throughout the last 200 years of the dramatic history of Poland, the painting was moved many times but also with great care and respect so that it remains in good condition despite the instability.

In 2003, the painting underwent treatment by our Conservatory to rediscover and recognize the work done by the Rembrandt Research Project in its A Corpus of Rembrandt Paintings. After removing two layers of varnish, including one layer two hundred years old, the painting’s original surface was examined in great detail using this ingenious technique. This was also possible because of the painting’s excellent condition. One of the discoveries made under the microscope was to find that the signature had been executed in wet paint.

Among one of the Great Masters of Painting and Prints is a famous technique that is widely known. This new method helps to dispel the “glazing-myth” by Rembrandt with resin varnish or mediums most characteristic of his work. The mistake of seeing translucent paint is not a result of this glazing, but the result of very fluid paint with a lot of oil. This makes certain browns and black colours semi-transparent.

Also the light parts of impasto details were created in wet paint. The significance of the oil medium used was the way it had been prepared which shows the extent of knowledge in the workshop at that time. Four hundred years had not substantially affected the layer of paint, with almost imperceptible craquelure, and only very soft cracks.

The "Golden Age" of Dutch painting has been taken to new heights with these technologies. In 2007 the French company Lumière Technology completed photographic examinations of *Lady with Ermine* with a multispectral camera, as well as other important paintings owned by the Czartoryski Foundation, including *Landscape with Good Samaritan*. Thanks to these photographs we have full documentation of the results after restoration.

SCIENTIFIC ANALYSES IN DETERMINING ORIGINAL APPEARANCE. STUDIO AND REMBRANDT, *SAUL AND DAVID,* C. 1660, OIL ON CANVAS (H: 131 X L: 164 CM)

Petria Noble

Royal Picture Gallery Mauritshuis, The Hague, The Netherlands.

Dating from around 1660 this controversial picture in the Mauritshuis (inv. 621) depicting the figures of Saul and David portrayed against a dark background, was considered for a long time to be one of Rembrandt's most important late paintings. At some point in the past the two figures were cut apart and reassembled, at the same time replacing a large piece of missing canvas with a modern insert. For a long time the true condition of the painting has been unclear. Recent investigation of the picture using a range of technologies involving the use of X-rays: high resolution scanning of the X-ray and analyses of the paint layers with light microscopy, X-ray fluorescence (XRF) and SEM coupled with energy-dispersive X-ray microanalyses (SEM-EDX) provides important new information about the painting's condition and original appearance.

Detailed examination and manual thread counts of the X-ray assembly (24 films) makes clear that the painting is comprised of ten separate pieces of canvas and that the four pieces of linen on which Saul and David are painted are identical. Furthermore, the later additions of narrow strips at the upper, lower and right edges are clearly visible. High resolution scanning of the X-ray (600 dpi) and a novel computer-assisted thread counting software developed by Rick Johnson (Cornell University), also makes thread counts of the narrow strips possible. As a result we now know for instance, that one of the added strips at the lower edge originates from the lost segments of the painting from either above or below the figure of David. Discontinuity of weave faults in the linen support also makes it possible to prove that a section of canvas is missing along the vertical notched join between the two figures. This is significant, since in the Paris auction catalogue of 1830, when the picture first appeared, it was listed as 16 cm wider that it is now. Other features in the X-ray, such as deep cusping along three edges, strainer bar marks and the presence of (part) of the original right tacking edge, make it possible to deduce the picture’s original format.

Unfortunately little or nothing is known about the restoration history of the picture. It can be deduced, however, from comparison of measurements cited in the auction catalogues that the painting was fashioned into its current state between 1830 and 1869. The painting was last restored in Berlin in 1900 by the German restorer, Alois Hauser. In Dutch newspaper articles that appeared at the time it was stated that Hauser gave the insert its present dark tone. Microscopic examination of the paint surface and analyses of the paint involving light microscopy, hand held XRF and SEM-EDX, in collaboration with Conservation Scientist Annelies van Loon, have demonstrated the presence of overpaint in much of the background. Characterization of the pigment composition and paint layer build-up of several paint cross-sections from the background makes it possible to discern the extent of the overpaint, although in some cases, this proved challenging due to the use of similar pigments. The original dark brown paint of the background comprises several layers containing bone black and Kassel earth, as well as red/yellow earth pigments. The folds in the original curtain are applied with a compact red paint containing red lake, red earth and smalt. Notable is the presence of intermediate fluorescing (varnish) layers, suggesting different campaigns of painting. It can be concluded that a curtain is part of the original design, although it is difficult to assess its original appearance and whether other features/ figures in the background may be obscured by the overpaint. It would therefore seem that Hauser not only gave the insert its present dark tone, but to a large extent overpainted the original curtain between the two figures in order to camouflage the disfiguring joins and the abraded condition of the paint.

Through the use of both novel and standard analytical methods, new insights have been made regarding this famous painting. The detailed results of this investigation will be presented and consequences and options for treatment will be discussed.

IS THE PORTAIT OF A YOUNG MAN, IN THE ROYAL WAWEL COLLECTION THE WORK OF JOHN LIEVENS?

Joanna Winiewicz-Wolska, Ewa Wiłkojć

Wawel Royal Castle, Krakow, Poland.

In 1910 at the Amsterdam auction a professor of the history of art, George Mycielski, bought *The Portrait of a Young Man*, considered as the work of a Dutch painter John Lievens. After Mycielski's death, in 1929, *The Portrait* ,together with his complete collection, were donated to the Wawel Royal Collection according to his will. In 1917 Hans Schneider published an article that comprised the analogy with *The Portrait of a Young Man* by Rafael in the Czartoryski Family collection.

The authenticity of Lievens work, the disciple of Rembrandt, has not been undermined yet, although some scientists and researchers have spotted similarities to works of Juriaen Ovens or Govaert Flinck.

The painting represents the so called "international" portrait style, that has been formed under Flemish art, particularly portraits by Anton van Dyck. Thus the author of the painting should be searched among the artists for whom van Dyck works constituted an important source of artistic inspiration and the proper perspective. Those could have perceived *The Portrait* by Rafael both original and copy or graphic form. Van Dyck knew Rafael's work as he saw it during his journey to Italy in 1623 and sketched it.

The stylistic analysis of the Wawel collection *The Portrait* apart from claiming the precise dependence both on Rafael masterpiece as well as van Dyck heritage cannot provide an answer to the dilemma of authorship. The essential notion seems to be reference to technological research, particularly that Lievens works have been methodically analysed due to the last monographic exhibition at the National Gallery in Washington and Rembrandthuis in Amsterdam.

The fact that the Wawel painting was not enlisted in Lievens *oeuvre* presented at the exhibition mentioned above makes it very adequate that it is the masterpiece of Flinck or Ovens. Flinck's portraits based on Flemish tradition constitute a mere margin and the artist did not manage to escape some typisation. They do not bear any traces of van Dyck painting fascination or premises to base the conclusion that he knew Rafael's work. He could get acquainted with its replica while collecting or art dealing. His fascination with van Dyck paintings as well as familiarity with his works find prove in many sketches he based on the Flemish artist's works. Some details, such as clothing folds or background in the Wawel *Portrait* also seem to be to his advantage.

PORTRAIT OF JOOST VAN DEN VONDEL' BY PHILIPS KONINCK FROM THE JAGIELLONIAN UNIVERSITY MUSEUM COLLECTION. ATTRIBUTION AND IDENTIFICATION.

Anna Jasińska[1], Danuta Skowron[1], Marek Krąpiec[2], Maria Ligęza[3], Jan Rutkowski[3], Jolanta Pollesch[1], Paweł Karaszkiewicz[3]

[1]*Jagiellonian University Museum, Krakow, Poland,*

[2]*AGH University of Science and Technology, Krakow, Poland,*

[3]*Academy of Fine Arts, Krakow, Poland.*

The cleaning of the painting "The scholar in his study" provided for an opportunity to reveal the long-forgotten signature of the artist, previously hidden under the dirty varnish. It was already earlier that the painting captured and held a considerable interest of the investigators. But only upon the disclosing of the signature, which was that of Ph. Koninck, the painting became the object of research, Koninck being either the disciple or, if not, then certainly a close friend of Rembrandt.

The objective of the present contribution is the establishment of the authenticity of the painting and its author. To arrive at this goal complex conservators studies of the painting were carried out. The painting became also an object of stylistic analysis as well as manifold examinations. It was viewed in the light of ultraviolet, infrared and sodium rays. The painting was X-rayed, its pigments were analysed by means of spectral emission while its support was exposed to dendrological as well as dendrochronological investigation. In addition, a series of macrophotographs were made.

While stylistically analysing the painting, the investigators drew above all upon the only monograph written about this artist by H. Gerson: "Philips Koninck" (Berlin 1936). Among other materials exploited for the analysis also the recent research by W. Sumowski published in his "Gemälde der Rembrandt Schüler" (London 1990) were used.

The discussed painting may be either the portrait of a specific individual with the accentuated vanitative motif (still life) or it may be an allegorical representation of Vanitas. As regards the potential portrait of a specific individual, one may bring forward a hypothesis that the painting is the portrait, the seventeenth one, of poet Joost van den Yondel who was Koninck's close friend. Both Koninck and the poet stayed in Amsterdam at the time when the painting came to being.

Upon the stylistic analysis we may arrive at a few fundamental conclusions decisive of the authenticity of the painting and its author:

1) considering its theme the painting fits the scope of interests of the artist,

2) there may be found a few significant details: types of faces, the handling of the painted individuals, which allow for the association of this painting with other works of the artist,

3) the composition of the painting, the use of pigments and the manner of applying them complies with the *modus procedendi* of the artist as described in the monograph by H. Gerson,

4) the coloristic scale applied in the painting corresponds, according to what Gerson observed, to the early period of the artist's creativity,

5) the influence of Rembrandt, so typical of the early period of Koninck's output, is easily detectable in the painting,

6) the fact, emphasized by the author of the monograph, that only the early works of the artist, as affected by Rembrandt, were painted on the board. While supported by dendrological and dendrochronological investigation, this fact is additionally determinative of the dating and authenticity of the discussed work,

7) in the light of conservator's examination and stylistic analysis, also the artist's signature, previously unknown to the owner of the painting, seems to confirm the authenticity of the authorship of the picture.

While drawing on the conclusions listed above, it is possible to state that the painting is one of the early works produced by Philips Koninck. The work must have come to being around 1645. Later it sank into oblivion while its signature for a long time remained hidden under the varnish. The recent research made by Piotr Oczko (Opuscula Musealia 2008) confirmed the hypothesis, that portrait is the image of Joost van Vondel.

X-RAY TECHNIQUES IN INVESTIGATIONS OF ART OBJECTS

USE OF X-RAY POWDER MICRO-DIFFRACTION FOR IDENTIFICATION OF LOCAL SOURCES OF PAINTING MATERIALS

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Combined use of flexible and non-destructive methods together with the knowledge of historic painting techniques, the mineralogy and the knowledge of geological and/or geochemical processes may generate a reliable basis to uncover local sources of materials that could help to attribute the provenance, dating or authorship of painted artworks. Among the modern non-destructive (with respect to the sample) micro-analytical methods the laboratory x-ray powder micro-diffraction becomes more and more widely used as an effective tool for such a direct analysis especially of inorganic and mineral phases without any special sample pretreatment.

Clay-based (earthy) pigments represent an interesting group of materials widely used for painting and ground layers. Their elemental composition, similar from one mineral to another, is not sufficient for their reliable identification without the use of another instrumental technique. Direct phase analysis by X-ray diffraction plays unsubstitutable role in the identification of clay minerals structures. Similarly, the identification of specific minerals like vivianite in paint layers together with the knowledge of only few deposits around Europe could also put the light into the attribution of an artwork.

Kaolinite containing ground layer found in wall painting in St. Maria Magdalena Church in Bor (district Karlovy Vary) represents a technological rarity. Calcareous (lime wash) ground layer was usually used for that purpose. Here identified kaolinite comes most probably from local kaolin deposits. Also the presence of accompanying minerals such as fluorite, titanium oxide and phosphates may lead to an idea of the use of material sources which are typical for Western-Bohemian region around Karlovy Vary.

PORTABLE DIGITAL X-RAY RADIOGRAPHY SYSTEM FOR STUDIES OF HISTORICAL OBJECTS

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The use of X-ray radiography for art research, and particularly for paintings has a long history. X-ray photographs of paintings, sculptures or other pieces of art are used as one of the basic techniques in physico-chemical analysis. The image previously caught on light sensitive films is nowadays stored via digital sensors caries a lot of information about the structure of an object, its build-up and state of preservation.

This presentation is about the use of a portable digital X-ray radiography system during research at the Laboratory of Analysis and Non-destructive Examinations of Historical Objects at the National Museum in Krakow.

The system used by the Laboratory is based on a wireless detector in a form of a flat container sized 35 x 43 cm 1,9 cm thick. The set consists also of a radiation source – a lamp with variable voltage and dose, computers used for acquisition and image processing and a server for data storage.

The PORTABLE DIGITAL X-RAY RADIOGRAPHY SYSTEM is used for technological research, to identify the construction of paintings as well as for other tests being part of our own research projects. This presentation show chosen examples of the practical use of radiography for various types of objects from the MNK collections. Its' vital advantages are mobility combined with the wireless data transfer from the detector and size allowing for tests outside the Laboratory, for example in the galleries or storage rooms. This is particularly considerable in a situation when MNK has 10 separate Departments.

“X-RAY POWDER DIFFRACTOMETRY FOR STUDIES OF HISTORICAL OBJECTS - A NEW EQUIPMENT AT FACULTY OF CHEMISTRY JAGIELLONIAN UNIVERSITY”

Marcin Oszajca, Marta Grzesiak, Katarzyna Podulka,

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The powder diffraction technique in studies of historical objects can be applied to the investigations of such as substances as pigments, corrosion products of metals and other crystalline artistic materials.. Unlike techniques such as X-ray fluorescence (XRF) and other methods of chemical analysis that provide information on elemental composition, XRD enables identification and differentiation of materials with similar or even identical chemical compositions. Shell and limestone are chemically the same (calcium carbonate), but the atoms are arranged differently in each of them. It would be difficult to tell these materials apart using elemental analysis. Some techniques, however, such as X-ray diffraction (XRD), provide information on the way atoms are arranged in a given sample.

As other examples, one can mention several pigments; e.g. two types of lead-tin yellow, Pb_2SnO_4 and $PbSnO_3$ polymorphic modifications of $TiO_{2,}$; or different kinds of verdigris. Such information is sometimes of great importance in dating and authentication a work of art, and in studying the origin of historical materials. Moreover, a description of secondary changes in the phase composition enables us to study the signs and causes of damage produced by environmental conditions and is vital to the proper conservation of the object, whether through preventive measures or restorative treatment. X-ray diffraction analysis is particularly useful in the study of museum objects because it requires a very small sample (in micro-diffraction measurements, often much less than the size of a pinhead).

When X-rays are fired at a crystalline sample, a part of them are diffracted by the regular crystal structure. These diffracted X-rays produce a diffraction pattern whose nature depends on the crystal structure of the sample. This pattern can be used as a kind of 'fingerprint' to identify a wide variety of materials. Such an identification can be performed with the use of reference powder diffraction data (PDF Files), which are prepared and distributed by the International Centre for Diffraction Data (Pennsylvania, USA).

X-ray micro-diffraction is used for the study of very small amounts of powder samples, or of small single-crystals. The X-ray beam, with a high intensity in this case, is concentrated on a very small area (often smaller than 50 microns) in order to produce a sufficiently clean signal so that the data may be collected in a reasonable time.

Micro-diffraction can be applied for many diffraction studies where local information of the sample is needed. Such studies include:

- small samples such as paint flakes from ancient masterpieces
- small spots on samples with strong gradients in composition, stress and texture, such as worked pieces of metal or mineralogical samples

The new X'Pert PRO MPD diffractometer at Faculty of Chemistry Jagiellonian University is equipped with a theta/theta goniometer, ceramic Cu X-ray tube, the position sensitive detector PSD PIXCEL, crystal monochromator, programmable incident radiation slit and focusing mirror.

With appropriate additional equipment (special cameras) with the possibility of high-temperature and low-temperature measurements, our diffractometer can be used for research focused on phase analysis, structural surveys and studies of phase transitions in a wide temperature range. The proper set of collimators and zero-background sample holders allows micro-diffraction measurements important in the study works of art.

In conclusion, the purchased equipment creates opportunities for research of small amount of samples, volatile and reactive compounds, enables phase analysis and structural surveys, important for chemists, physicists, archeologists and art conservators.

RECOVERING ERASED SCRIPTS FROM PALIMPSESTS: FIRST RESULTS FROM X-RAY FLOURESCENCE ELEMENT MAPPING EXPERIMENTS

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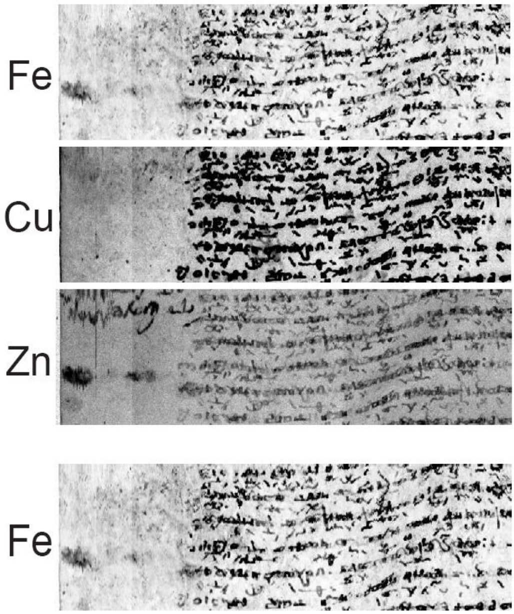
In the Middle Ages, literary texts where frequently copied on parchment before the use of paper prevailed. As the availability of affordable parchment was at times limited, it was not uncommon to reuse parchment from older books to write upon, creating what we call a palimpsest. The original writing was erased by chemical or mechanical means, and the repristined parchment leaves from one or several former books were written upon once more. Today, the erased underlying texts on these palimpsests are often at least as interesting to the scholar as the upper text, and there are even some texts from Classical Antiquity which have only been preserved through such copies.



The inks used where of the iron gall variety, and in those palimpsests created by chemical erasure are often still (or again) partially visible to the naked eye. Various methods have been used to make these better readable. Most recently X-ray fluorescence spectroscopy was used in an experiment similar to the one presented here, to map the text on four pages from the Archimedes palimpsest [1]. X-ray techniques pose no significant danger to a parchment's preservation, as radiation damage is negligible [2].

The project presented here is a collaboration of Teuchos – Zentrum für Handschriften- und Text-forschung (Universität Hamburg, DFG), Uni-versitätsbibliothek Leipzig and HASYLAB showing the possibilities of X-ray fluorescence element mapping to enhance the contrast of upper and lower writing concentrating on the scant impurities of the iron used in the manufacture of the iron gall ink. Element maps as shown in figure 1 for iron, copper and zinc have been recorded for various trace elements in regions of visible and erased writing. While reproduction of part of the text is often possible using the iron contrast only, additional information can be obtained from the contrast maps based on the iron impurities.

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RARE SILVERPOINT DRAWINGS BY REMBRANDT IN THE FOCUS OF SR-XRF

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The silverpoint drawing technique had its cumulating period in the late Middle Ages and the Renaissance. However, some undoubtful Rembrandt drawings were made on prepared vellum by the master with this already obsolete technique at the Golden Age. Among these drawings is the best-known portrait of his wife Saskia, 1633 (KdZ1152, Berlin). It is thus interesting to investigate these drawings. In addition to art historic studies, it was also important to get new insights into the graphical material employed in order to know whether it was different from that used in former periods, which in turn can give information on the genesis and dating of the drawings.

Silverpoint drawings belong to the most valuable treasures of graphical art collections. They are generally very precise drawings of excellent quality. Therefore, only completely non-destructive analytical methods are applicable. Moreover, they need to be very sensitive because of the low quantity of matter present in the strokes (less than some hundreds of $\mu g/cm^2$). Several preliminary tests showed that only Particle Induced X-ray Emission (PIXE) spectroscopy and Synchrotron radiation induced X-ray fluorescence analyses (SR-XRF) fulfil the analytical requirements for the investigations of these drawings meaning that they are sensitive enough, feasible in air and require no sampling.

Synchrotron radiation induced X-ray fluorescence results obtained at the BAM*line*, BESSY II, HZB, Berlin on three Rembrandt silverpoint drawings of the collection of the Kupferstichkabinett Staatliche Museen zu Berlin will be presented (Reiche et al. 2006).

The chosen method will be explained as well as the requirements for studying non-destructively valuable works of art such as these silver point drawings. The main part of the presentation focuses on the meaning of the results and illustrates how SR-XRF analysis can reinforce art historical assumptions on the genesis, the dating of the drawings and their connection. Additional information can be gained from such analytical studies on the conservation state of the drawings.

The results will also be compared to those available on other silverpoint drawings by Van Eyck, Dürer and the Holbein family (Reiche und Roth, 2009, Ketelsen et al. 2005, Reiche et al. 2004).

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POSTERS

REMBRANDT IN LABORATORY.

FACTORS OF DETERIORATION OF OIL PAINTINGS CONTAINING LEAD WHITE

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The ageing processes that take place in the structure of historical objects have not been fully recognized yet. As an example we can give untypical damage of the paint layers observed on the face of some oil paintings containing lead white. A very characteristic effect of "micro-craters" was observed. 100-200 um cavities are spread over the whole surface of the painting and go inside as deep as the ground. This effect was first observed in 1997 by Dutch researchers on "The anatomy lesson of Dr Nicolaes Tulp" by Rembrandt.

The borders of the mysterious holes were surrounded or filled with white fluorescent products of reactions that took place in course of time in the paint layer and the lead white ground. Their formation is connected with the presence of chlorine. The compound formed – $Pb_3Cl_4(OH)_2$ was identified as fiedlerite by means of a mass spectrometer SIMS-TOF and XRD methods (P. Noble, J.Wadum, K. Groen, R.Heeren, K.J. van den Berg, Aspects of 17th century binding medium: Inclusions in Rembrandt's Anatomy Lesson of Dr Nicolaes Tulp, in Art et Chimie, la couleur. Actes du congrés, Paris 2000, s. 126-129).

Independently, unexplained fluorescence of lead compounds was also observed in case of Gothic paintings during restoration works in 1996. XRD diffraction allowed to classify it as laurionite - Pb(OH)Cl (M. Poksińska, Przyczyny szarzenia minii w malowidłach ściennych in Gotyckie malowidła ścienne w kościele św. Jakuba w Toruniu, Toruń 2001, s. 73-83).

The present work reports the many-year experience of the authors in the area of interpretation of this phenomenon of significant danger to historic works of art which appears quite common and affects not only oil paintings. It also concerns other lead pigments. Hitherto research on the chemical nature of these compounds and the role of chlorine in their formation inspired us for laboratory investigation in order to verify this theory.

The authors hope that drawing attention to self-destruction processes of the works of art, in some cases the masterpieces of the world's heritage, and the research results will accelerate actions leading to reduction of these effects and to provision of a restoration program for this type of deterioration.

DIGITAL PROCESSING OF X-RAY RADIOGRAMS FOR CONSERVATION PURPOSES

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Radiography is a very useful tool in the identification, care and understanding of historical objects. Here we would like to report on application of digital processing and analysis of radiographs for conservation purposes. It is shown that the use of a computer may ease or even in some cases enable an interpretation of the x-ray photograph. One may point, at least, two aspects of digital processing of radiographs [1]. Namely, removal of some limitations that appear upon preparation of analogue x-ray radiograms and gain of an additional information about the historical object. Both aspects are analysed further basing on examples.

A typical problem appearing in analogue x-ray photography is film blackening (called *fog*) [2]. The fog emerges mainly due to long film storing periods or too long exposition times. In both cases, taking a digital photograph of the radiograph seems to be a good solution. Human eye adapts to the amount of light that is present at the moment of observation, while a CCD camera sums up the light as long as the shutter is open. Therefore with long exposure times it is possible to read out the dark radiograph. Moreover, use of graphical tools such as a histogram or tone curves may allow further correction of a picture [1].

Additional information may be acquired from the radiograph by making it more legible. It may be achieved by adding particular sets of colours to the picture originally made in a grey scale. The method is based on knowledge of human visual perception. For example presenting an x-ray photograph as a duotone yellow-to-black or white-to-blue picture makes the observer to involve more emotions and causes him to see more details [1].

More advanced digital image processing and computer analysis techniques are also presented [3]. These methods not only make visible details that can not be seen in a normal way due to limits of the human eye, but also upgrade a regular qualitative analysis into quantitative one [4]. Two examples are presented in detail. Firstly, extraction of the painting layer of a part of Matejko's painting *Joan D'Arc* (from the National Museum in Krakow collection) [5] is shown. Secondly, penetration of wood strengthening substances injected into wooden object is analysed in a quantitative manner.

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MULTI-METALLIC ARTEFACTS FROM WROCŁAW -EDXRF STUDIES

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Metallic artefacts from archaeological excavation in Wrocław were analyzed by energy dispersive X-ray fluorescence in order to establish the type of material used. The investigated object (mostly utensils fragments) were made using many different metals, also some artefacts are multi-metal composition. Because of high historic value, the artefacts need to be analyzed with non-destructive or non-invasive methods, preferably before conservation works. There are two main reasons for studying artefacts prior to their conservation or restoration. One of them is the determination of chemical composition. It is necessary to choose the appropriate conservation program (materials and procedures adequate for each metals). Second reason is possibility of appearance of negative effects during conservation (surface contamination or modification by tools and chemicals). The EDXRF is an appropriate research tool for this investigation: universal, fast, inexpensive and the analyses do not cause damages of artefacts. The opportunity of “point-analysis” (area - $1mm^2$) is very useful in the analysis of small parts of objects (e.g. incrustation, decoration, solders, rivets). The main disadvantage of EDXRF is a surface character, but using simple sample preparation it is possible to overcome the limitation of the method.

APPLICATION OF X-RAY METHODS FOR PIGMENTS IDENTIFICATION IN COLLECTION OF RAFAŁ HADZIEWICZ PAINTINGS

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The classical X-ray analytical methods include X-ray fluorescence (XRF) and X-ray diffractometry (XRD). First one gives qualitative identification of chemical elements, while second delivers crystallographic structural analysis. During the last decades both of them have been widely applied in historical object studies. In this work capabilities of these analytical techniques have been used to collect information on the painting technique of Rafał Hadziewicz.

Rafał Hadziewicz was a Polish 19th century painter, author of many religious paintings and portraits. He was the best known for historic compositions, which were often compared to art of the Italian Renaissance and European Baroque.

National Museum in Krakow posses broad collection of that author paintings, which have been conserved last year. Identification of compounds used by Hadziewicz was necessary before conservation treatment and it was performed with X-ray fluorescence analysis (XRF) in combination with X-ray diffraction analysis (XRD). Three women portraits, painted in various life periods of painter were chosen for analysis. The study was focused on the pigments used by the artist in the paint layers. The inorganic components of the ground were also analyzed.

Three paintings were studied: *"Portrait of Stefania Jarońska"* (ca.1850), *"Portrait of young lady"* (1845) and *"Portrait of Julia Hadziewicz"* (1860). Before the cleaning of the objects, micro-samples were taken in order to study the materials used by the painter. For XRD analysis a very small amount of painted materials (four samples, each the size of a few mm^2) were taken from damaged areas, without damaging the picture further. All XRD measurements were carried out with the use of X'PERT PRO MPD diffractometer. CuKα radiation at 40kV and 30mA, a graphite monochromator and scintillation or X'Celerator detectors were used. The divergence of the beam was 0.5° or 1°, zero-background holders were used for samples available in very small amounts. The measurements were performed in the 2Θ range 5-80° with a step size of 0.02°. The obtained diffraction patterns were interpreted with the use of diffractometer software (XMenu: Philips Diffraction Software) and PDF-2 or PDF-4 databases.

Elemental compositions of all objects were found by using an ArtTAX® µXRF spectrometer (Bruker AXS Microanalysis, Germany) equipped with a Rhodium X-ray tube (50kV, 500µA, 50 keV, in air, 300s).

Performed analysis indicated that white lead, naples yellow, vermilion, prussian blue and natural iron oxide pigments were used for all three paintings. Additionally cobalt blue was detected in the case of *“Portrait of Stefania Jarońska”* and *“Portrait of Julia Hadziewicz”*, while chromium green was applied for *“Portrait of Stefania Jarońska”* and *“Portrait of young lady”*. Besides, manganese violet was found in *“Portrait of Julia Hadziewicz”*.

The combined use of XRF and XRD techniques enabled us to identify inorganic components of the painting materials used by Rafał Hadziewicz. Performed analysis allowed to understand deeply the craftsmanship and technology used by this painter.

X-RAY BASED ANALYSIS OF THE METAL THREADS IN HISTORICAL TEXTILES

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Historical textiles were decorated by metal threads, since thousands years. The term metal thread includes every type of thin, like-yarn textile decoration as strips and wires, made of solid metals, metal-coated organic materials or the combination of these with natural or man-made fibers. Metal strips can be made of pure gold, gold alloyed with silver, gilded or gild-silvered copper and gold-like copper alloys. Pure gold, silver or gilded silver were used from early times in manufacture of valuable materials, signifying wealth and social status. Copper, brass or tin were often coated with a thin layer of silver or gold [1, 2].

The knowledge about the composition of the metal threads in old textile objects gives very valuable information about the ancient manufacturing techniques and also about the appropriate treatment for cleaning and conservation. The aim of the study was to determine the composition of the various metal threads of textiles dated to different epochs (between 15^{th} and 19^{th} centuries) and originating from Europe and Asia. Performed study allows to create metal composition database for identification and classification of museum textiles of various provenience.

The investigation of textiles was carried out by analytical non-destructive methods, as XRF and Raman spectroscopy. Elemental compositions were found by using an ArtTAX® µXRF spectrometer (Bruker AXS Microanalysis, Germany) equipped with a Rhodium X-ray tube (50kV, 500µA, 50 keV, in air, 120s). Raman spectra were acquired by using a portable Raman spectrometer, DeltaNu, equipped with a diode 785 nm laser.

Performed analysis indicated that flax and silk textiles were decorated by metal threads. In the case of European textiles the elemental composition of metal threads changed through the ages from gold in 15^{th} century, gold with silver in 17^{th} century and finally gilded copper in 18^{th} century. Similar trend was observed for Asiatic historical objects. Gold and gilded silver metal strips were used in 18th century, while tin strips were found in 19th century Japanese hanging scroll. Beside, additional elements as iron, antimony and nickel were detected.

Non-invasive µXRF technique was found suitable for characterization of the elements in the metal strip decorations. The compositional analysis of the metal threads in historical textiles provided significant information as regards the technology of their manufacture as well as the economical value and consequently the social rank that they represent.

This work was partially supported by a grant “Direct monitoring of strain for protection of historic textiles and paintings on canvas” from Iceland, Liechtenstein and Norway through the EEA Financial Mechanism

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WITKACY'S PASTELS ANALYSIS FOR SAFE ANOXIA STORAGE

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Due to photofading, pastels on coloured-paper supports are some of the most sensitive and unstable museum objects. The National Museum in Krakow possesses a broad collection of this type of light sensitive object, and their preservation as well as accessibility through exhibition are co-concerns. Oxygen-free storage and display have been proposed as a means to achieve both goals. However, before application of anoxic conditions, identification of the pigments used in art object is required.

This work presents the results of studies performed on three Witkacy pastels. Objects created in different years were chosen: "Portrait of Władysław Dunin-Borkowski" (1920), "Portrait of Stefan Szuman" (1929) and "Portrait of Anna Friedrichowa" (1936). Pastels by Witkacy are interesting due to their coloured-paper supports, which are very light sensitive.

Analysis of pigments was performed by X-ray fluorescence spectroscopy (ArtTAX, Bruker), while the paper support colour changes were measured by UV/VIS spectrophotometer (OceanOptics USB 4000). Additionally, the objects were also studied in analytical light (VIS, UV).

Colour measurements indicate that Witkacy used the same paper support in the case of “Portrait of Władysław Dunin-Borkowski” and “Portrait of Stefan Szuman”. Analysis of exposed and unexposed regions of the support indicates that significant colour change has occurred in the past. XRF analysis showed that painter used both inorganic and organic pastels, however only inorganic pigments could be recognize by this method. Chrome yellow was detected in all objects, while vermillion was found in “Portrait of Władysław Dunin-Borkowski” and “Portrait of Stefan Szuman”, and Schelle’s green was present in “Portrait of Stefan Szuman” and “Portrait of Anna Friedrichowa”.

XRD INVESTIGATIONS OF COMMERCIALLY AVAILABLE PRUSSIAN BLUE PIGMENTS AND OIL PAINTS

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Prussian blue is thought to be the earliest of the modern synthetic pigments. Discovered in 1704 by Diesbach in Berlin, it was soon manufactured in France (Paris), England and other countries. It was used as a pigment in oil paintings, printing inks, typewriter ribbons, carbon paper and sometimes also cosmetics. Since the middle of the twentieth century it has been replaced by phthalocyanine blue.

The chemical formula of Prussian blue is $Fe_4[Fe(CN)_6]_3$ xH_2O or $KFe[Fe(CN)_6]$ xH_2O. However, Kremer Pigments Company produces this pigment as an ammonium complex with the formula $NH_4Fe[Fe(CN)_6]$. xH_2O.

The aim of the presented study was to perform the phase analysis of commercially available Prussian blue pigments and oil paints. The obtained results may be helpful for identification of this pigment in the scientific analysis of works of art.

We have investigated Prussian blue powder pigments produced by the Maimeri, Schmincke and Kremer companies. The results showed that the pigment produced by Schmincke was $Fe[Fe(CN)_6]\cdot 4H_2O$ [PDF 74-9174] whereas the powder patterns of the other two samples were similar to each other. We have found no reference data for these patterns in the PDF 4 file, that is why the XRPD data for phase characterization of $NH_4Fe[Fe(CN)_6].H_2O$ (Prussian blue produced by Kremer Company) were elaborated. The compound crystallizes in the cubic system, with lattice parameters a=b=c=10.232(1)Å and Fm3m space group.

In commercially available blue oil paints the pigment Prussian blue, due to its high tinting strength, is mixed with various, usually white, compounds. We performed XRD phase analysis of blue oil paints named Prussian blue produced by

Pollena-Astra, Maimeri, Daler-Rowney, Renesans, Royal Talens, and Phoenix and established their phase compositions.

It was found that only Pollena-Astra produces Prussian blue oil paint which contains $Fe_4[Fe(CN)_6]_3 \cdot 14H_2O$. [PDF 73-0689] without any crystaline fillers. In other paints ferric ferrocyanide hydrate was accompanied by $CaCO_3$ [PDF 83-0578], $(Ba,Pb)SO_4$ [PDF 77-0432] or $Al_2(Si_2O_5)\cdot(OH)_4$ [PDF 89-6538].

We have also investigated a blue powder pigment named Prussian blue (no inw.MNK IV - V 1024/ 1- 16) which belonged to well-known Polish painter Henryk Siemiradzki. It was found that the sample contained $Fe_4[Fe(CN)_6]_3 \cdot 4H_2O$ [PDF 74-9174] and gypsum $CaSO_4\cdot2H_2O$ [PDF 06-0046].

All XRD measurements were carried out with the use of an X'PERT PRO MPD diffractometer. CuKα radiation at 40kV and 30mA, a graphite monochromator and scintillation or X'Celerator detectors were used. The divergence of the beam was 0.5°. The measurements were performed in the 2Θ range 3-80° with a step size of 0.025° or 0.02°. The obtained diffraction patterns were interpreted with the use of diffractometer software (X'Menu: Philips

Diffraction Software) and PDF-2 or PDF-4 databases. Details of the performed studies will be presented during the Meeting.

XRD and SEM/EDX INVESTIGATIONS OF THE CORROSION PRODUCTS OF METAL THREADS FROM TAPESTRY OF SIGISMUND AUGUSTUS

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The Sigismund Augustus collection, partially exhibited in Wawel Castle in Krakow, consists of 136 tapestries, commissioned in the middle of the 16th century from the most prominent workshops in Brussels on the occasion of the king's wedding [1]. The materials used were wool, silk and metal threads for the weft. Some areas of the design (mainly those depicting clothing) contain precious metal threads with a fibrous, usually silk core.

The Wawel collection of tapestries includes 19 pieces with subjects taken from the Book of Genesis. Seven pieces depict *The Story of the First Parents,* eight pieces *The Story of Noah* and four pieces *The Story of the Building of the Tower of Babel.* The tapestries are about 4.8 meters high and up to 8.8 meters wide.

During the conservation work after cleaning *The Building of the Tower of Babel* tapestry it was found that the metal threads were partially corroded. Samples of damaged threads as well as corrosion products (black powder) were investigated with the use of XRD powder diffractometry and SEM techniques.

All XRD measurements were carried out with the use of an X'PERT PRO MPD diffractometer. Cu$K\alpha$ radiation at 40kV and 30mA, a graphite monochromator and scintillation or X'Celerator detectors were used. The divergence of the beam was 0.5°, or 1° for samples available in very small amounts. The measurements were performed in the 2Θ range 4-80° with a step size of 0.02°. The obtained diffraction patterns were interpreted with the use of diffractometer software (X'Menu: Philips Diffraction Software) and PDF-2 or PDF-4 databases.

Scanning electron microscopic studies of the metal threads were carried out by means of a JEOL JSM – 7500F field emission scanning electron microscope.

The obtained results allowed us to establish the elemental composition of the metal threads as alloys of silver and gold, or silver, gold and copper. As one might have expected the main component of the corrosion product in the case of such composition was Ag_2S. The details of the investigations described above will be presented in the poster.

[1] M. Piwocka, *The tapestries of Sigismund Augustus*, Wawel Royal Castle State Art Collections, Krakow 2007

[2] A.M. Hacke, C.M.Carr., A. Brown, D. Howell, Investigation into the nature of metal threads in a Renaissance tapestry and the cleaning of tarnished silver by UV/Ozone (UVO) treatment., JOURNAL OF MATERIALS SCIENCE 38 (2003) 3307 – 3314

SYNCHROTRON X-RAY POWDER MICRO-DIFFRACTION and MICRO-FLUORESCENCE FOR IDENTIFICATION OF GREEN PIGMENTS IN the GOTHIC ALTAR PAINTINGS FROM THE MALOPOLSKA REGION

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The aim of the presented work was to investigate the samples of green paint taken from five Gothic altars from the Malopolska region. The oldest paintings in this group are the altar wings of a triptych from Zator (one of the wings is in the parish church in Zator, the other in the National Museum in Krakow). They were painted in the second half of the 15th century by an unknown master. Holly virgins depicted in the painting represent the end of the early years of Malopolska panel paintings, in which the tradition of international style of the end of the 15th century (soft arrangement of drapery) is present. The painting workshop where the wings from Zator were executed is recognizable by the physiognomy of the depicted characters (swollen eyes and thick necks).

All the investigated samples were collected from unobtrusive areas of the paintings. Each sample was divided into two similar pieces, one of which was then embedded in polyester resin. X-ray diffraction and micro X-ray fluorescence measurements were performed at DORIS III of Synchrotronstrahlungslabor HASYLAB in Deutsches Elektronen-Synchrotron DESY (Hamburg, Germany).

On the basis of optical microscopic study the conservators suspected the presence of verdigris in the samples. Verdigris is a name used to describe a group of copper salts of acetic acid which range in colour from green, via green blue and blue green, to blue. Different kinds of verdigris may be divided into two groups: basic verdigris and neutral verdigris.

Basic verdigris may contain one or a mixture of the following acetates: $[Cu(CH_3COO)_2]_2Cu(OH)_2.5H_2O$ (blue), $Cu(CH_3COO)_2Cu(OH)_2.5H_2O$ (blue), $Cu(CH_3COO)_2[Cu(OH)_2]_2$ (blue) and $Cu(CH_3COO)_2[Cu(OH)_2]_3.2H_2O$ (green). The formula of neutral verdigris is $Cu(CH_3COO)_2.H_2O$ [1]. In powder diffraction data file PDF 4+ one can find 10 entries for copper acetate; however, none is a basic copper acetate.

Results obtained from μXRF analysis show that all green pigments in the investigated samples are copper compounds (high intensities of copper bands) and also indicate the presence of lead and tin.

In our investigation we prepared verdigris in the lab according to a historical recipe, to provide a reliable reference pattern. Comparison of diffraction patterns of prepared verdigris with the patterns of investigated samples shows that most peaks fit verdigris. In the case of the sample from Grywałd there are some peaks which could belong to malachite. Additionally in the sample from Łącko some peaks may indicate that lead tin yellow type I might have been used by the painter.

1. Kühn H. Identyfication of the materials of paintings: *Verdigris and copper resinate*, Studies in Conservation 15 (1970), 12-36

CONSERVATIONAL ANALYSIS OF THE SIGISMUND BELL

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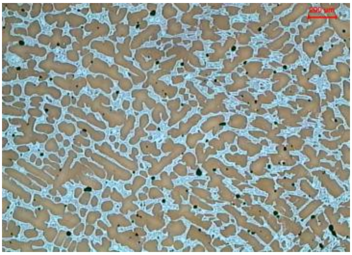
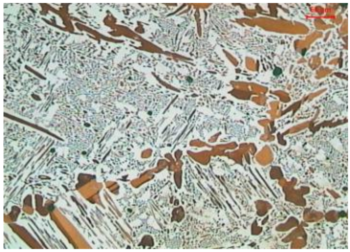
The Sigmund Bell was cast of bronze in 1520 in Krakow by a nurembergian bell-founder Hans Bohem. The height of the bell is equal to 2.58 m, its diameter at the bottom is 2.42 m, and the periphery in the widest place extends to almost 8 m. The thickness of the walls of its body is in the range between 7 and 21 cm. The length of the clapper is 218.5 cm and its weight is 323 kg. The body itself weights about 10 t and the total weight of the whole bell with the clapper and yoke is almost 13 t.

In the year 2000 Sigismund Bell became silent for over three months because of the fracture of its steel clapper. Upon preliminary investigations, it was decided build a new clapper. It has been agreed that the clapper ought to be reproduced by forging technique reconstructing its original shape and weight. In addition thorough examination of the current state of preservation of the bell's body was also performed. It was shown that there are numerous creases and traces of abrasion in the parts where the clapper strikes. Nevertheless, a very massive crusting of metal on the bell's rim was the most alarming feature. Due to this reason, chemical analysis of the alloy composition was carried out by means of X-ray fluorescence spectrometry and metallographic analysis of the metal structure. The results revealed that the Sigismund Bell was cast of bronze, with an average chemical composition of about 80% Cu and 20% Sn. The distribution of both metals is not homogenous in all parts of the bell (see table). In the top parts the content of tin in the range of 13%, whereas in the bottom parts it increases up to 30%.

part\element	Sn	Cu	P	Sb	Fe	Al	Si	other
Upper part	12,84	81,68	0,8085	0,4497	0,2127	0,035	0,179	3,8
Bottom part	29,56	63,37	2,823	1,165	0,636	0,198	0,295	1,96





The results allowed to answer the question of the present state of the bell's body and to unravel the reasons for crusting of its rim. The crusting was caused by higher amount of tin in the lower part of the body, resulting probably form liquation – a phenomenon that can occur during solidification of the cast. Higher amount of tin results in increased hardness of the alloy, giving rise to its enhanced crustiness. In such a case a sharp rim of the bell's body, affected by vibration, will keep crusting until it becomes rounded. The metallographic analyses showed that there are no traces of corrosion, especially the intercrystalline one, showing that the surface of the Sigismund Bell is still well preserved.

AN INTERESTING DILEMMA – A SMALL LANDSCAPE SIGNED *REMBRANDT F. 1627.* A DEPOSIT IN THE JAGIELLONIAN UNIVERSITY MUSEUM COLLECTION

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In 1995 the Jagiellonian University Museum in Cracow received for long-term deposit a painting that depicts a *Landscape with a mill*; oil on oak board; dimensions: 23.2 x 31.5 cm. The painting has a signature on the bottom edge, almost in the centre: *Rembrandt f.1627.* The *Landscape* was bought by its owner at an antiquarian market in Stockholm. At the time of purchase, it had a different frame. The frame had covered the signature, which was discovered only when the frame was changed for the present one. On the face of the painting, there is a clearly visible print of the old frame that overlaps with the signature.

The scene shows a large mill with a granary with a mill wheel and gates in front. When seen in large close-up, the painting reveals a rich diversity of detail which cannot be seen with the naked eye. The work has survived in good condition. It was painted on an oak board cut radially. The reverse side of the board is unbevelled. In the right upper part, a piece of paper with information printed in French is attached and reads that the painting was a part of the Duke of Nemours’s collection, and then was kept in Hombourg (name of the owner illegible). Next to it, there is a notice written in red pen: by Rembrandt. At the bottom, centrally, there is a barely visible text written in pen (?): "Rembrandt était agé de 21 ans quand il fit ce tableau, a l'age de 18 ans il était déja celébre."

A painting with a signature like this obviously aroused concern and interest, the more so because it seemed to be an interesting Dutch period piece already during the initial examination. The decision was taken to investigate it closely. On account of that, a number of examinations were carried out to determine the authenticity of the painting as a Dutch work of art from the 17th century and investigate it in terms of technological and graphological aspects against works by Rembrandt. The examinations were carried out in the Conservation Workroom of the Jagiellonian University

Museum in 1997 and 1998, with the assistance of the Academy of Fine Arts in Cracow and experts from Warsaw, the Hague and Germany.

The painting was examined in sodium light, UV light, and infrared light (the observation under the IM 3 infrastereoscopic microscope revealed a sketch of the composition; particularly compelling is the outline of the perspective sketch) and subjected to an X-ray inspection, dendrological and dendrochronological examinations, an examination of pigments and graphological analyses performed by I.H. Hardy.

The performed examinations prove the authenticity of the painting as a Dutch work of art from the 17th century. None of the technological examinations radically excluded the *Landscape* as a potential early work by Rembrandt. It only remains to conduct a thorough stylistic and comparative analysis, which – based on the results of the completed examinations – should give a final answer to the questions:

- is it a work by the Master?

- is it a work by one of the apprentices from his studio or his followers?

- is it a forgery created later than the signature suggests?

- is it a work by an unknown artist which reflects the so-called *Zeitgeist*?

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