Detail of the pl. 2.

A macro-to-microscale scientific investigation of the *Visitation* panel

Insights into its materiality and the metal oxalate alteration phenomenon

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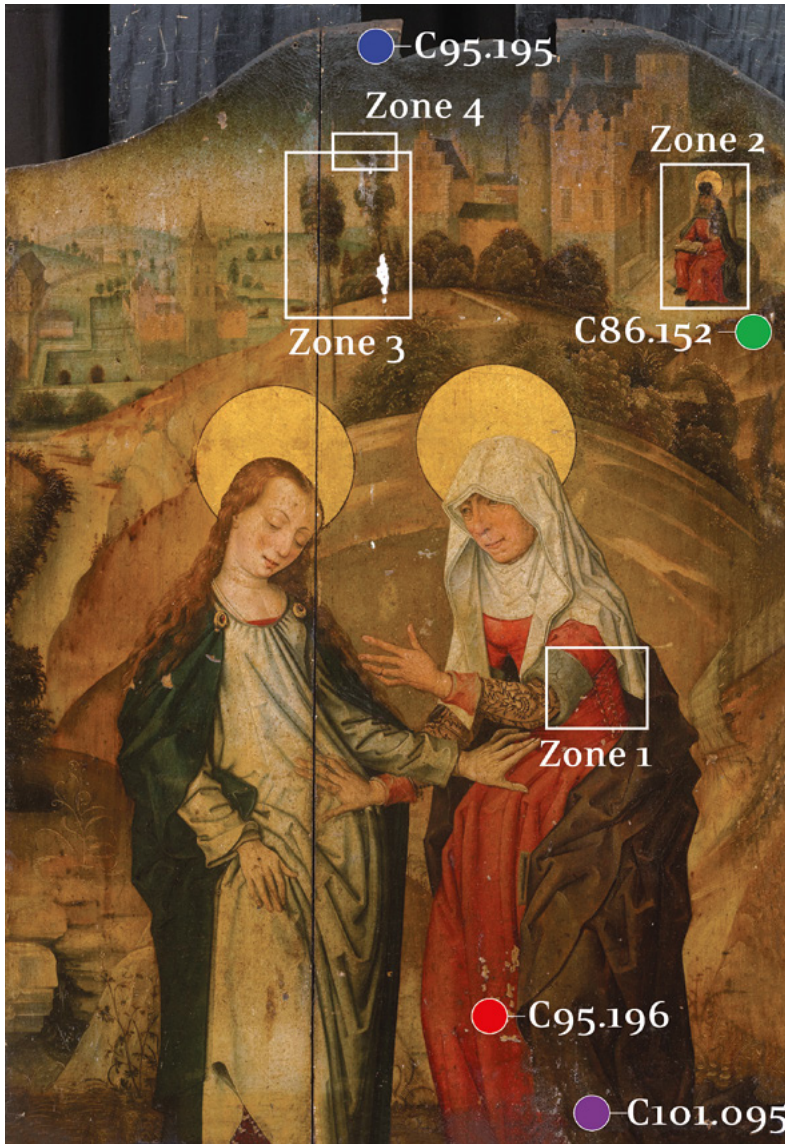
The conservation-restoration of the *Visitation* panel (fragment 2 recto) at the Royal Museum of Fine Arts of Belgium took place in parallel with the MetOx[2] research project. The latter provided scientific support for the material study of the painting as well as its alteration, focusing more particularly on the as-found metal oxalates species. The current chapter will briefly illustrate the scientific results obtained during the macro-to-microscale multi-analytical investigation of the *Visitation* panel. It will firstly present the results relating to the characterisation of the painting materials and technique. It will then offer an overview of the metal oxalate alteration phenomenon observed in the panel's pictorial layers.

The MetOx macro-to-micro analytical methodology

The MetOx analytical approach consisted of combining a large array of analytical techniques that encompass the painting from the macro to the microscale. At the macroscale, three main imaging techniques were employed: macro X-ray fluorescence (MA-XRF), macro reflection Fourier-transformed infrared spectroscopy (MA-rFTIR) and macro X-Ray powder diffraction (MA-rXRPD). In the case of the *Visitation* panel, these techniques were applied distinctively in four different analysis zones [fig. 30]. Each one of these techniques, developed by the Antwerp X-ray Imaging and Spectroscopy laboratory (AXIS) of the University of Antwerp (UA), yields different types of chemical information from the paintings in a non-invasive and non-destructive manner. MA-XRF makes it possible to visualise the distribution of chemical elements in the paint layers. Even though this information is crucial to producing refined hypotheses on the composition of such paint layers, in this research project this technique was mostly employed to facilitate the interpretation of both MA-rFTIR and MA-rXRPD data. The two other techniques made it possible to locate spatially either molecular moieties (MA-rFTIR) or crystalline phases (MA-rXRPD) attributable to different paint materials or metal oxalates species located at the painting's surface. Further specifications on the analytical instrumentation employed can be found in Table 1.



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White rectangles indicate the zones of the *Visitation* panel scanned with both MA-rFTIR and MA-rXRPD. The entire painting was scanned with MA-XRF except for its bottom-left area. Red, green, blue and purple paint layers were also micro-sampled in pinpoint locations of the panel, indicated by the like-coloured dots. Associated KIK-IRPA crosssection numbers are indicated as a reference.

Likewise, a series of four microsamples[3] were extracted from the *Visitation* panel [fig. 30] and prepared into cross-sections. These samples were documented by optical microscopy, under polarised and ultraviolet light (OM-VIS/UV). Subsequently four complementary analytical techniques were selected for the in-depth chemical characterisation of the paint strata at microscale level [see Table 2]: Attenuated Total Reflection Fourier Transform Infrared microspectroscopy (ATR-μFTIR); Scanning Electron Microscopy - Energy Dispersive using X-Ray analysis (SEM-EDX); microRaman spectroscopy (μRaman); and Time of flight secondary ion mass spectroscopy (ToF-SIMS).

Table 1 - Imaging techniques employed for the macroanalysis of the *Visitation* panel

Acronym	Name	Instrument specifications
MA-rFTIR	Macro reflection Fourier-transformed infrared spectroscopy	Bruker Alpha FTIR spectrometer with a frontal reflection module mounted on motorised stages (10 x 25 x 10 cm^3)
MA-XRF	Macro X-ray fluorescence	XOS Xbeam microtube with a Rh anode coupled to a Vortex EX-90 SDD detector on a motor stage of 57 x 60 cm^2
MA-rXRPD	Macro X-Ray powder diffraction	Custom-built scanner featuring an Incoatec Cu^{HB} X-ray source, a Dectris PILATUS 200K 2D diffraction detector and motor stages (30 x 30 x 10 cm^3)

Table 2 - Analytical techniques employed for the microanalysis of historical paint microsamples

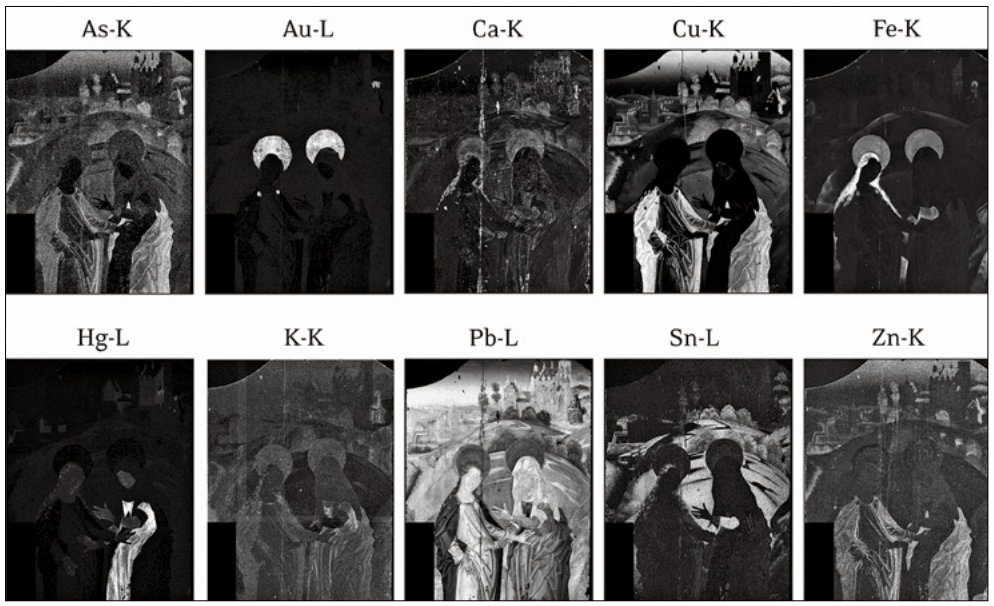
Acronym	Name	Instrument specifications	Institution
OM-VIS/UV	Optical microscopy under polarised (VIS) and ultraviolet (UV) light	Zeiss Axio Imager M1 equipped with a CCD Deltapix camera	KIK-IRPA
ATR-μFTIR	Attenuated Total Reflection Fourier Transform Infrared microspectroscopy	Bruker Vertex 70 FTIR coupled to a Bruker Hyperion 3000 FTIR microscope equipped with a 250 mm germanium ATR tip	
μRaman	microRaman spectroscopy	Renishaw InVia coupled to a Leica DMLM microscope	
SEM-EDX	Scanning Electron Microscopy - Energy Dispersive using X-Ray analysis	Zeiss EVO LS 15 SEM coupled to an Oxford Instruments $X\text{-}Max^N$ EDX detector	
ToF-SIMS	Time of flight secondary ion mass spectrometry	IONTOF GmbH	UCLouvain

Finally, as a result of the possibilities offered by the new Historical Materials BAG[4] access to the ID13 beamline of the European Synchrotron Radiation Facility (ESRF), two historical paint cross-sections (C95.195 and C101.095) were investigated using synchrotron-based micro X-ray powder diffraction (SR-μ-XRPD) to investigate their crystalline composition at microscale level.

Characterisation of the painting materials and technique used in the *Visitation* panel

The *Visitation* panel was scanned in its entirety with MA-XRF (bottom-left area excluded) to gain insight into the pigment composition through the elemental distributions [fig. 31] as well as to highlight areas of interest for analysis with MA-rFTIR and MA-rXRPD.

While pigment identification with MA-XRF is indirect, several preliminary conclusions can be drawn from the elemental composition. For instance, the halo surrounding the two women was created using metallic gold while St. Anne's red robe was painted using a mercury-based pigment, very likely vermillion or cinnabar. The Ca-K signal seems most intense in areas where the ground layer is exposed, (such as lacunae) but might also originate from calcium-based compounds (chalk, gypsum, ...) in the paint layers. The presence of tin in the yellow landscape suggests the use of a lead-tin yellow pigment. The Fe-K distribution suggests the use of earth pigments, most notably in the Virgin Mary's brown-colored hair. The Pb-L distribution implies the



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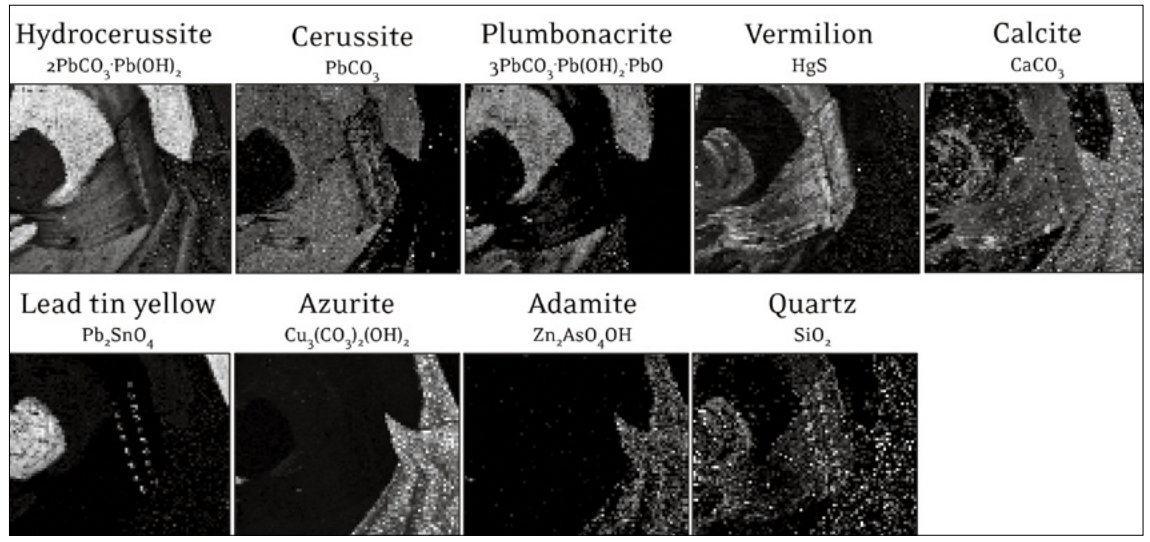
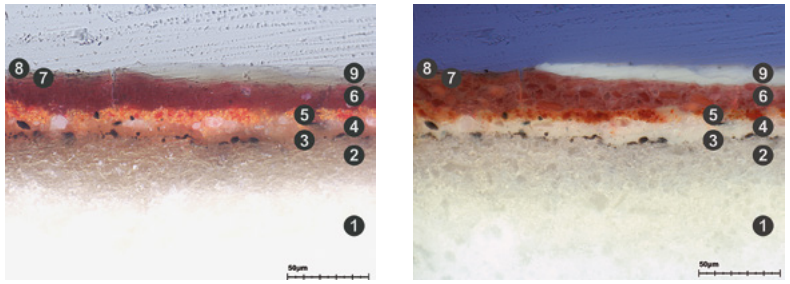
MA-XRF elementary distribution images obtained for the Visitation panel. Brighter zones indicate higher relative concentrations of the selected species in the different mappings.



32
MA-rXRPD distribution images for zone 1 of the *Visitation* panel. Brighter zones indicate higher relative concentrations of the selected species in the different mappings.

usage of lead white, and its co-localisation with tin also supports the identification of lead-tin yellow in certain areas. Copper is closely related to the blue areas on the panel, as in the Virgin Mary's blue robe or the sky, but also to St. Anne's violet drapery or the green foliage in the background. Cu-K is co-localised with As-K and Zn-K, possibly hinting at the presence of an impurity in these areas. Finally, a weak signal for potassium was detected, being the most intense in the depicted figures' red robes, suggesting the use of a red lake in these areas.

Based on the MA-XRF results, several smaller areas were scanned with MA-rXRPD [fig. 30] to unambiguously identify the employed crystalline pigments. The MA-rXRPD distribution images for zone 1 are shown in fig. 32. MA-rXRPD confirms the presence of vermillion or cinnabar and lead-tin yellow (type I). Hydrocerussite and cerussite, the two main components of lead white, were identified alongside the rare compound plumbonacrite. Two different subtypes of lead white were identified based on the hydrocerussite-to-cerussite ratio (HC-C): a hydrocerussite-rich lead white (HC-C = 90%-10%) was used in the blue sleeves and white headscarf and a cerussite-rich subtype in the red dress (HC-C = 65%-35%). A second difference between these two areas is that plumbonacrite is present only in the hydrocerussite-rich zone. Nonetheless, it is unclear whether plumbonacrite is a remnant of the lead white production process or has been formed *in situ* from the alteration of the original painting materials. The purple robe contains azurite along with minor amounts of malachite and quartz. Chalk was also identified alongside the azurite and vermillion pigments. As MA-rXRPD is limited to the identification of crystalline compounds, it cannot confirm the presence of a red lake in the red or violet robes, as suggested by the MA-XRF results.

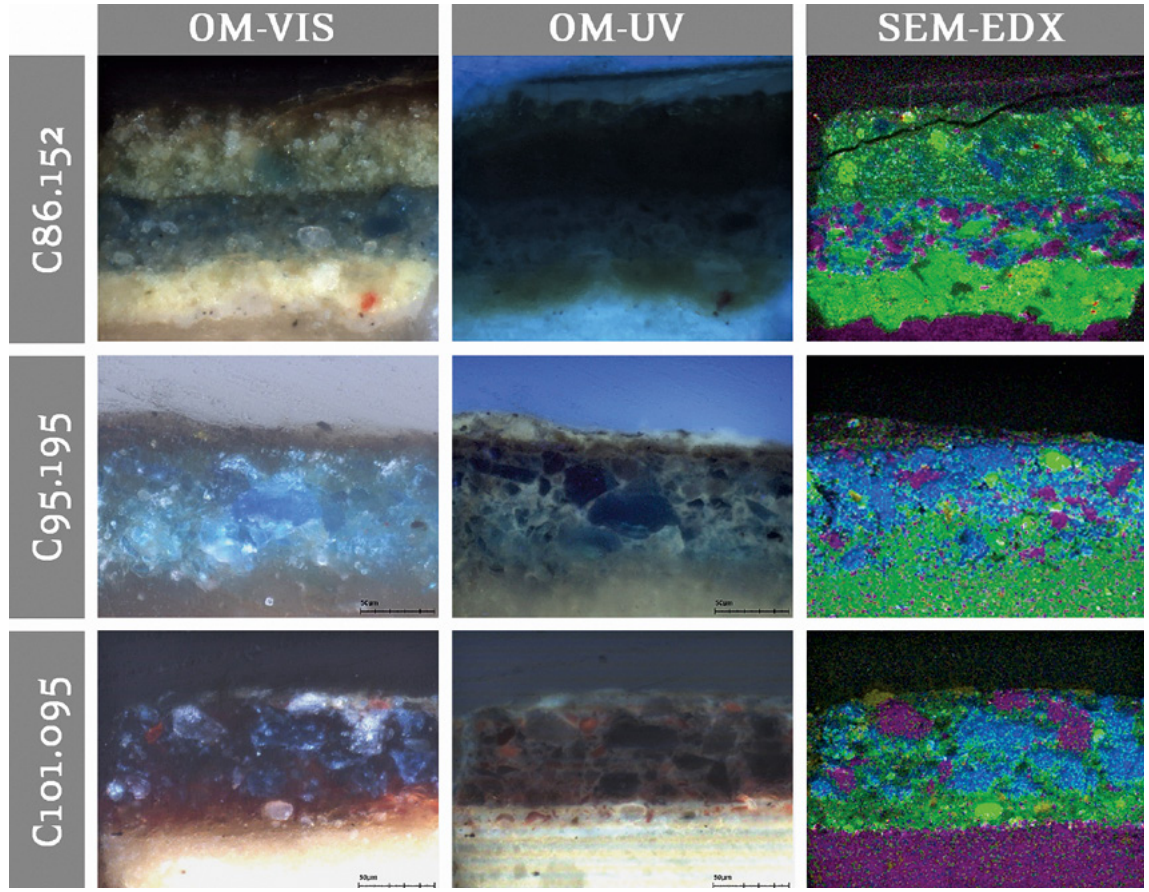


33
Sample C95.196, taken from St. Anne's robe. Microphotographs under polarised (left) and ultraviolet (right) illumination illustrate the panel painting's layer build-up, considered to be typical of Early Netherlandish oil paintings:
(1) ground;
(2) impregnated ground surface;
(3) drawing;
(4) priming;
(5) underpaint;
(6) paint;
(7) glaze;
(8) alteration crust;
(9) varnishes.

The paint materials and general layer build-up [fig. 33] revealed by the study of the microsamples correspond to what is usually observed in other early Netherlandish paintings. The preparatory layers include a sizing of the panel onto which was applied a white ground (c. 100-140 µm) in at least two distinct layers, recognisable on the cross-sections. Under SEM-BSE, fragments of coccoliths are discernible while ATR-µFTIR analysis indicates the presence of proteins, suggesting the use of chalk bound with an animal glue. The upper part of the ground (c. 20-30 µm) was likely impregnated with an oil in order to impermeabilise it before applying the subsequent oil-based layers. In this zone of the ground, calcium sulphate aggregates were found, dispersed in the ground matrix, indicating a possible local alteration of the chalk.

A black carbon based underdrawing was then applied, as suggested by the morphology of the black particles [fig. 33][5]. The drawing was then covered and fixed with a thin (c. 7-10 µm), transparent, binder-rich and highly fluorescent layer, usually called priming. The priming is an intermediate layer between the preparatory, colourless strata and the coloured paint layers. It is a distinct layer observed not only in early Netherlandish paintings but also in other Northern European artworks including some pre-Eyckian paintings[6]. The formulation of such an intermediate layer is quite varied. Moreover, it has been observed that its composition can even vary in different areas/panels constituting the same artwork, as has been recently demonstrated for the Van Eyck brothers' Ghent altarpiece[7]. In the Master of the View of Saint Gudula's painting, the priming layer contains small amounts of lead white [present as both hydrocerussite ($2PbCO_3.Pb[OH[_2$) and cerussite ($PbCO_3$)) and calcite ($CaCO_3$)] and some carbon black particles bound with oil. The combination of ATR-µFTIR and SR-µ-XRPD results, which indicate the presence of lead carboxylates[8], and those obtained by SEM-EDX, in which lead is diffusely observed throughout this layer, seem to suggest that the employed oil binder was heat-bodied with a lead siccative.

The paint layer is applied in a succession of two or three strata: underpaint, paint and glaze layers. The employed pigments, such as azurite, verdigris, lead-tin yellow (type I), lead white, madder and kermes lakes, vermillion and carbon black, were bound with a siccative oil-based binder. This relatively simple colour palette was skilfully mixed and applied in different layer build-up



34
Microphotographs (500x) under polarised (OM-VIS) and ultraviolet (OM-UV) illumination of the green (C86.152), blue (C95.195) and purple (C101.095) samples taken from the *Visitation* panel. Corresponding SEM-EDX elemental distribution maps for lead (green), copper (blue) and calcium (pink) are shown in the right column.

schemes in order to produce the rich chromatic diversity observed in the panel painting.

For instance, St. Anne's purple drapery (see microphotographs of sample C101.095 in fig. 34) was produced by the superposition of a mauve paint layer, composed of azurite mixed with red lake, calcite and lead white (composed of a mixture of cerussite and hydrocerussite), over a red underlayer containing red lake manufactured from wool shearings and mixed with lead white. The particle size of the azurite grains in the upper layer is quite variable, the largest of them measuring as much as 50 µm.

Likewise, the green background vegetation was executed with two kinds of pigment mixtures (see microphotographs of sample C86.152 in fig. 34), applied over a pale yellow underlayer composed of a mixture of lead-tin yellow (type I), lead white and a minute amount of red particles. The topmost pigment mixture producing a green-yellowish paint layer is composed of verdigris combined with lead-tin yellow and lead white. This paint layer is applied over a blue-green layer containing azurite, lead white, some lead-tin yellow and large particles of calcium carbonate. The latter material is found in quite a large quantity, suggesting that it is an additive rather than an impurity of one of the employed pigments. The calcite particles are as large as the azurite pigment grains, suggesting the use of ground marble.

The particularity of the azurite pigment, found in the underlayer of the background vegetation, in the blue hue of the sky or in Saint Anne's violet mantel, resides in the presence of rare mineral impurities. Indeed, besides common impurities, such as iron (hematite, Fe_2O_3) and silicon oxides (quartz, SiO_2) present in small amounts, there are several particles containing arsenic and zinc. This composition suggests the presence of adamite (zinc arsenate hydroxide, Zn_2AsO_4OH), which was confirmed both at the macro and the microlevels by MA-rXRPD and SR-µ-XRPD, respectively [fig. 32]. The XRF-based co-localisation of arsenic and zinc in an azurite-containing blue paint layer was previously reported for a Late Medieval painting from Cologne[9]. The presence of this impurity has been linked to the exploitation of copper sulphide ores in Schwaz-Brixlegg, an ancient copper mining area in Tyrol[10].

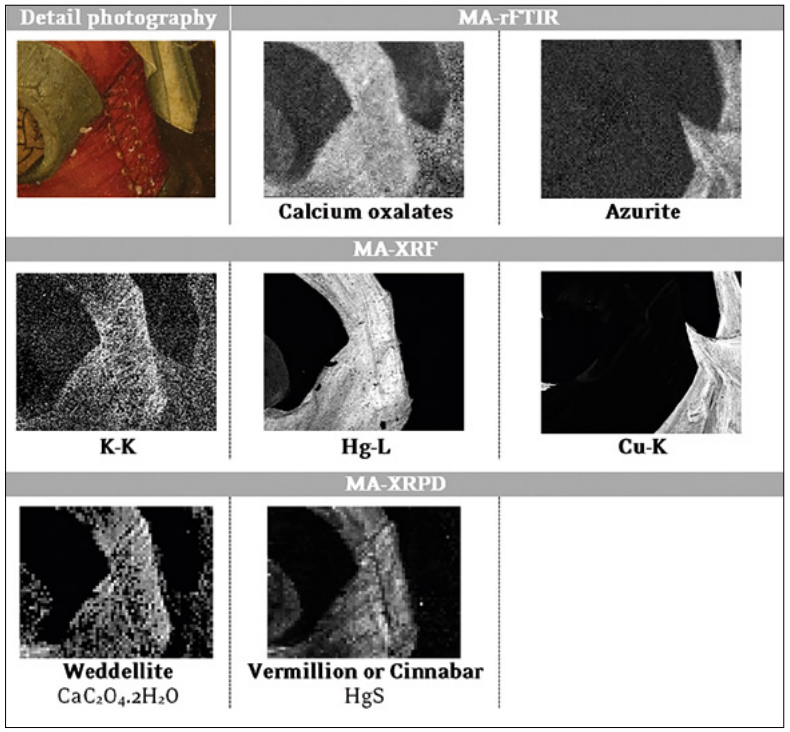
The layer build-up of St. Anne's red robe [fig. 33] is also worth discussing since such garments seem to have been executed in the same way in the two panel paintings composing this ensemble[11]: two glaze layers are applied over a vermillion underlayer. The lower, thicker glaze layer (c. 20 µm) is composed of two different lakes manufactured from wool shearings. These are characterised by large, irregularly-shaped particles, containing small amounts of alumina [hydrated aluminium oxide, $Al(OH)_3$] together with proteins from the wool. Some of these particles present a characteristic orange fluorescence under ultraviolet illumination, suggesting the presence of madder lake, while those with a more red-purplish fluorescence are likely composed of a kermes lake. The upper glaze is thinner (c. 5-8 µm) and is coloured by smaller red-purplish lake particles having higher amounts of alumina, likely prepared from silk textile waste dyed with *Kermes vermilio Planchon.*

The use of these two different, biologically-sourced red lake types was confirmed during the HPLC analysis of a non-embedded sample from St. Anne's dress. The obtained chromatogram revealed the presence of three red anthraquinones in different proportions: abundant purpurin moieties were found alongside small amounts of alizarine and kermesic acid. The first two compounds are indeed typical for madder lake prepared from *Rubia tinctorum* L, while the latter indicates the using of a kermes lake. The SEM-EDX detection of phosphorus in certain lake particles is considered as confirmation of the use of a lake from kermes[12].

The metal oxalate phenomenon in the *Visitation* panel

When applying the non-invasive microanalytical imaging techniques, MA-rFTIR measurements were hindered by the presence of the thick, oxidised varnish layer that covered The *Visitation* panel. Indeed, the spectra obtained were dominated by spectral features related to such a layer, obscuring the clear visualisation of other chemical species underlying this topmost stratum. MA-rFTIR scans were thus repeated on the panel once the varnish removal operation had been completed. As shown in fig. 35, results obtained during the analysis of zone 1 made it possible to spatially locate calcium oxalates at the surface of the unvarnished painting and thus at the topmost section or within the pictorial layers themselves. Their distribution is not homogeneous and they seem to be mainly localised in the red and purple areas, where higher potassium concentrations observed with MA-XRF suggest the use of a red lake, mixed with or superposed on either vermillion or cinnabar in the red areas or azurite in the case of the purple hue, as previously discussed. The MA-rXRPD analysis of this zone also made it possible to detect the presence of crystalline calcium oxalate of the weddellite type ($CaC_2O_4 \cdot 2H_2O$). As with the MA-rFTIR results, the spatial distribution of this oxalate species seems to correlate to red and purple areas.

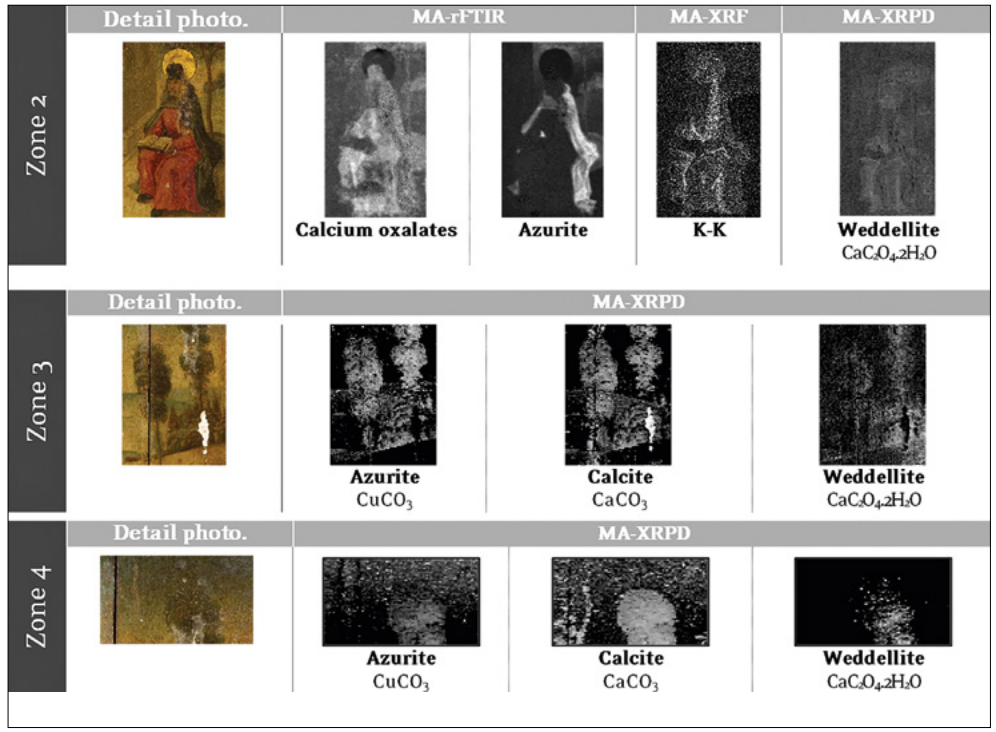
In zone 2, similar results were found using MA-rFTIR, detecting calcium oxalates in the red and purple robes of the sitting elder saint found in the background of the painting [fig. 36]. Likewise, the MA-XRF results suggest the use of a red lake in the red areas, mixed with azurite in the case of the purple cape. Nonetheless, only faint signals of crystalline weddellite-type calcium oxalate, close to the noise signal, were detected with MA-rXRPD, matching those areas indicated by MA-rFTIR. These results suggest that in these areas crystalline and amorphous calcium oxalates coexist. Nonetheless, it is not clear if the weaker crystalline signals indicate that such phase is present in smaller quantities than its amorphous counterpart or if it is located in a deeper paint layer and thus its characteristic signals are attenuated. Interestingly, weddellite was also found in the brownish-green tree and bushes analysed in zones 3 and 4 [fig. 36]. These elements of the



35
Selected element or compound-specific distributions obtained with MA-rFTIR, MA-XRF and MA-rXRPD for zone 1 of the *Visitation* panel. Brighter zones indicate higher relative concentrations of the selected species in the mappings of the different techniques.

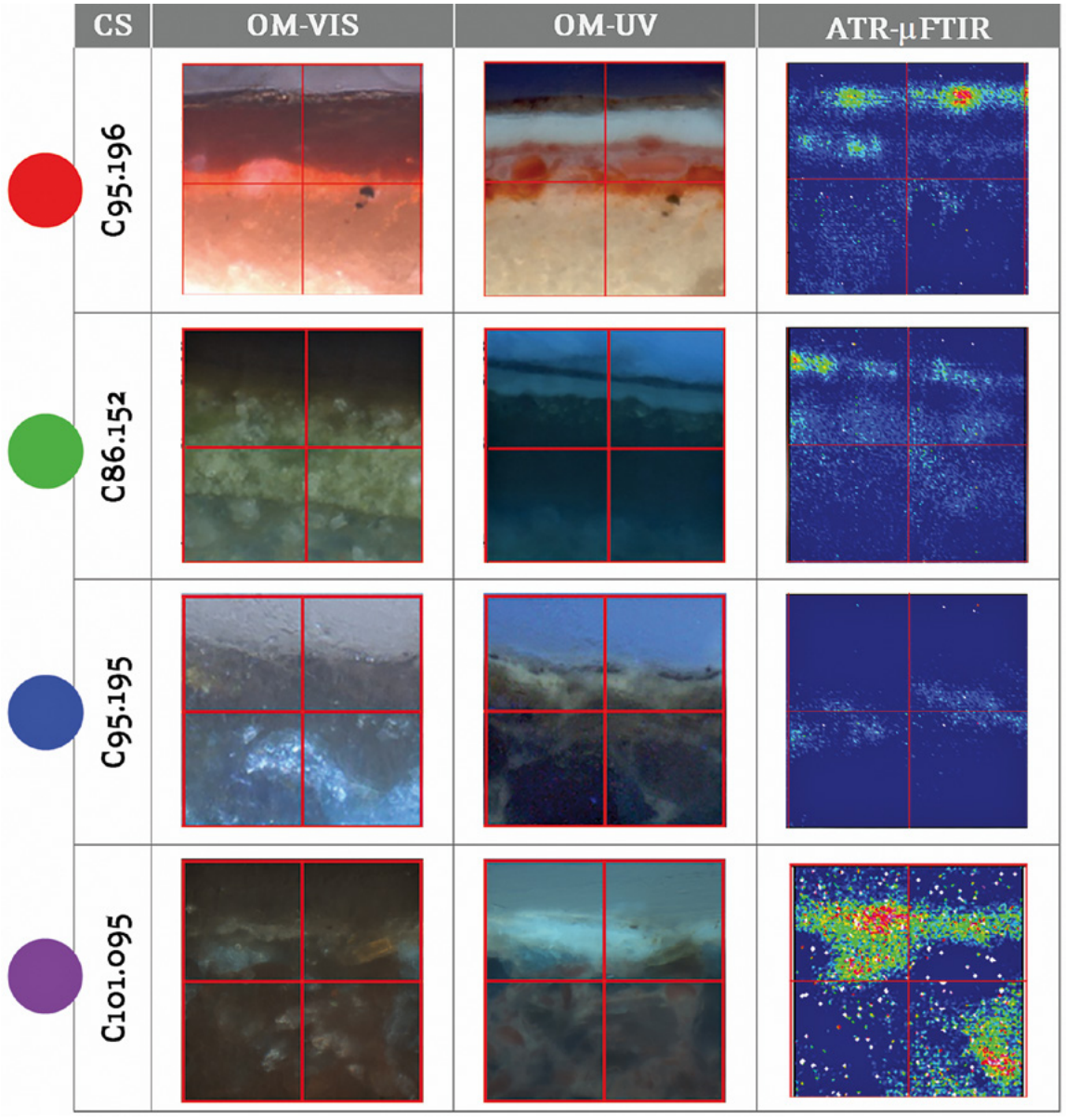
composition are mostly azurite-based and rich in calcite, present as an additive as described in the previous section.

These results demonstrate that both MA-rFTIR and MA-rXRPD are able to detect metal oxalates in historical paintings in a non-invasive manner. Contrary to MA-rFTIR, MA-rXRPD makes it possible to identify and discriminate closely chemically-related metal oxalates types [like differentiating weddellite ($CaC_2O_4 \cdot 2H_2O$) from whewellite ($CaC_2O_4 \cdot H_2O$)], only if they are crystalline. However, MA-rFTIR is sensitive to the presence of such alteration compounds, irrespective of their solid phase state (crystalline or amorphous). The application of both techniques thus makes it possible to obtain a comprehensive picture of the surface spatial distribution of metal oxalates on historical paintings. It also makes it possible to evidence pigment-to-metal oxalate correlation patterns, as those outlined for the *Visitation* panel. In the case of this panel painting, only calcium oxalates were found. Nonetheless, these techniques have made it possible to detect other metal oxalate species, such as copper oxalate, in other historical paintings such as the Ghent Altarpiece by the Van Eyck brothers.
Regarding the use of microanalytical techniques, ATR-μFTIR was the technique that provided the fastest and most reliable results concerning the composition

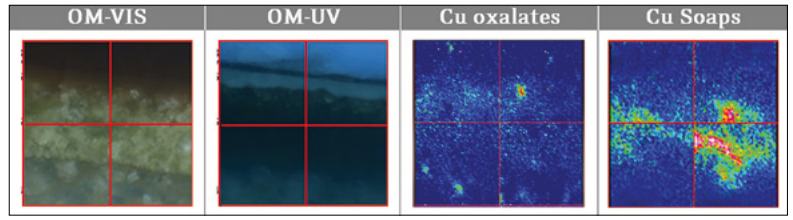


36
Selected element or compound-specific distributions obtained with MA-rFTIR, MA-XRF and MA-rXRPD for zones 2-4 of the *Visitation* panel. Brighter zones indicate higher relative concentrations of the selected species in the mappings of the different techniques.

and spatial distribution of the metal oxalate moieties found in such paint layers. SR-μ-XRPD, while able to differentiate between various metal oxalates types, showed only weak signals for weddellite-type calcium oxalate in one (C95.195) of the two analysed samples. Fig. 37 summarises typical results obtained for the localisation of calcium oxalate (CaOx) in the four extracted microsamples. As shown there, calcium oxalate is invariably found in the uppermost stratum of the paint structure, either in surface degradation layers (samples C95.196 and C86.152) or within the surface varnish (C101.095), in some cases agglomerated in nodules. However, it is also found within the paint layers themselves or even at the surface of the chalk-containing grounds (results not shown). In those containing madder red lakes (C95.196 and C101.095), characteristic calcium oxalate signals are more intense, possibly indicating a higher concentration of the oxalate moiety, and also tend to agglomerate in nodules. In paint layers containing azurite (C95.195) or verdigris (C86.152), they seem less concentrated and present a hazy spatial distribution.

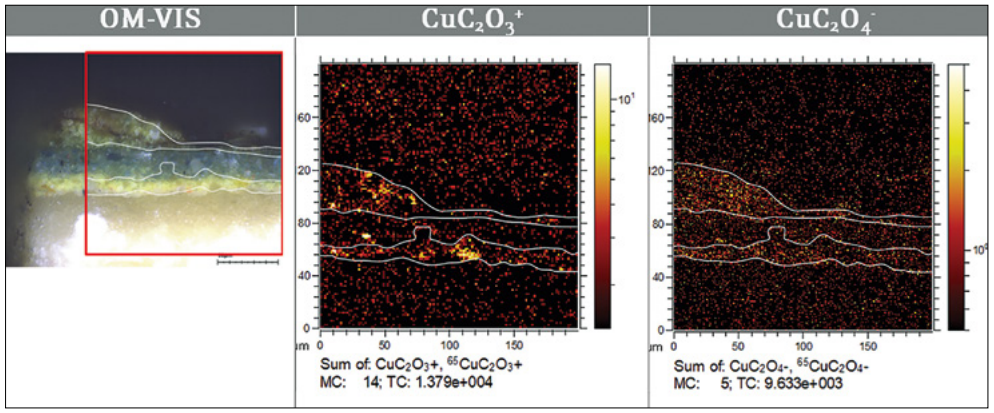


37
ATR-mFTIR mappings showing calcium oxalate C-O symmetric stretching distribution (13361296 cm^{-1}) in the red, green, blue and purple cross-sections (CS). In each mapping, the falsecolour scale ranges from deep blue to pink-white, indicating the absence or the highest relative intensity of the visualised vibration respectively. The corresponding microphotographs of the analysed zones are shown under polarised (OM-VIS) and ultraviolet (OM-UV) illumination.



38
ATR-mFTIR mappings showing the spatial distributions of copper oxalate and copper soap in the verdigris-rich paint layer of cross-section C86.152. In each mapping, the false-colour scale ranges from deep blue to pink-white, indicating the absence or the highest relative intensity of the visualised vibration respectively. The corresponding microphotographs of the analysed zone are shown under polarised (OM-VIS) and ultraviolet (OM-UV) illumination.

Copper oxalates (CuOx) were also found in sample C86.152 using ATR-µFTIR [fig. 38]. Contrary to what was found for CaOx, copper oxalates are not found at the surface of the paint structure but exclusively within a paint layer in which verdigris is expected to have been used, mixed with lead-tin yellow. No CuOx were found in the underlying azurite-based paint layer nor in sample C95.195 which is also primarily composed of azurite. Interestingly, the detected CuOx are located in close spatial vicinity to other types of copper carboxylates, such as copper soaps. The presence of CuOx in the verdigris-rich layer was confirmed with ToF-SIMS [fig. 39]. Interestingly, characteristic CuOx ions were also detected in the bottom lead-tin yellow-rich paint layer as well (not analysed by ATR-µFTIR). Nonetheless, the technique was not capable of detecting CaOx, possibly due to its low ionisation probability.



39
ToF-SIMS mappings showing copper oxalate spatial distribution in a zone of cross-section C86.152 (marked in red in the OM-VIS microphotograph). Characteristic CuOx ions were detected in both positive ($CuC_2O_3^+$) and negative ($CuC_2O_4^-$) mode.

Conclusions

The interdisciplinary investigation resulting from the association of the restoration of the *Visitation* panel and the MetOx research project proved to be a fruitful synergy. On one hand, the scientific investigation of the panel from the macro to the microscale using state-of-the-art analytical techniques not only offered a thorough characterisation of its painting materials and comprehension of the artist' pictorial technique, but also supported decision-making during the conservation-restauration process, especially when deciding on cleaning choices. On the other hand, it also allowed the MetOx researchers to clearly define the contribution of each analytical technique to the multiscale comprehension of the metal oxalate phenomenon (chemical composition, spatial distribution and pigment-specific reactivity patterns), both at a painting's surface as well as inside its pictorial layers, thus expanding our understanding of how historical painting materials transform in time.

1 Polychromed Artefacts Laboratory, Laboratories Department, Royal Institute for Cultural Heritage (KIK-IRPA): Francisco Mederos-Henry and Jana Sanyova; Research Centre in Archaeology and Heritage (CReA-Patrimoine), Free University of Brussels (ULB): Francisco Mederos-Henry; Antwerp X-ray Imaging and Spectroscopy laboratory (AXIS), University of Antwerp (UA): Steven de Meyer and Frederik Vanmeert; Paintings Laboratory, Laboratories Department, Royal Institute for Cultural Heritage (KIK-IRPA): Frederik Vanmeert.

2 The MetOx project was an international research venture, coordinated by the Royal Institute for Cultural Heritage (KIK-IRPA) and funded by the Belgian Science Policy (BELSPO) through the Belgian Research in Action (BRAIN) initiative, aimed at characterising fundamental aspects of the metal oxalate phenomena in major Southern Netherlandish oil paintings created from the 15th to the 17th centuries.

3 The study of this panel painting was started in 2017 by Dr Steven Saverwyns' research group (KIK-IRPA) analysing two cross-sections and carrying out two varnish analysis, before the painting was included in the MetOx project. The authors are grateful to Dr Saverwyns for making these samples available for the project.

4 The Historical Materials BAG, proposal HG-172, is a pilot project for the new access models implemented thanks to support of the European Union's Horizon 2020 research and innovation programme under grant agreement No 870313, Streamline.

5 Only one cross-section shows the underdrawing material. Although this single microsample does not allow to conclude about the nature of the employed drawing materials for the entire panel, it seems likely that at least one of the drawing steps was executed with a dry media, possibly charcoal, perhaps combined with other wet and/or dry techniques.

6 J. Sanyova *et al.*, 'The Challenges of a Complex Stratigraphy from a Chemical Point of View', in G. Steyaert, M. Postec, J. Sanyova, H. Dubois (ed.), *The Ghent Altarpiece. Research and Conservation of the Interior: the Lower Register*, Brussels, Brepols, 2021.

7 *Loc. cit.*

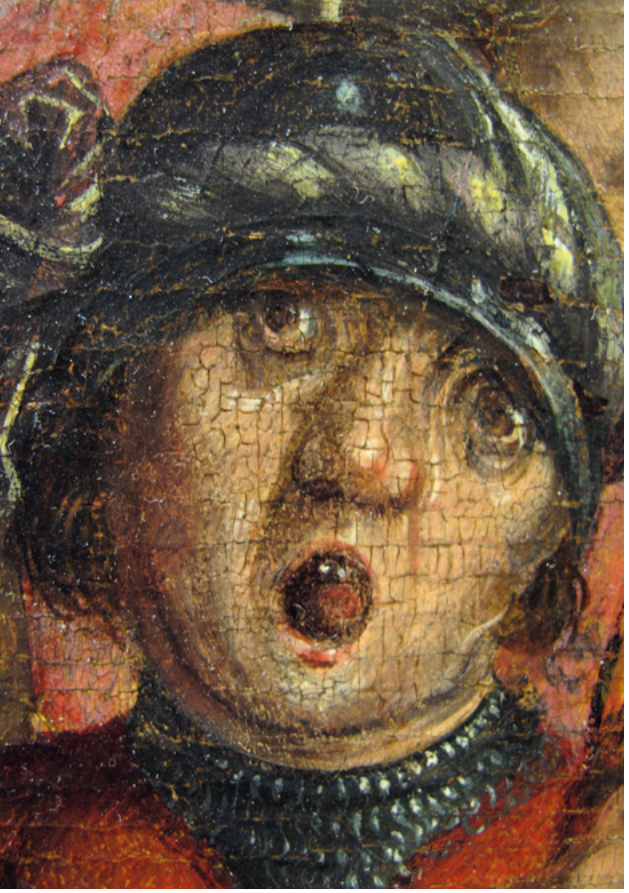
8 In sample C95.195, SR-µ-rXRPD results clearly show that the priming layer, located between the calcite ground and the azurite-containing paint layer, contains lead carboxylates whose diffraction signals agree well with data provided by Koci for mixed Pb-stearate/palmitate soaps [E. Koci *et al.*, 'Mixed lead carboxylates relevant to soap formation in oil and tempera paintings: the study of the crystal structure by complementary XRPD and ssNMR', in *Dalton Transactions*, 2019, 48 (33), pp. 12531-12540 (DOI: 10.1039/C9DT02040C)].

9 The painting in which Zn and As were co-detected is *Epitaph. Virgin Mary with Child, St Jerome and Donor*, 1440-50, inv. WRM 71, Wallraf-Richartz-Museum.

10 H. Paschinger, 'Die Wandmalerei im Kreuzgang des Franziskanerklosters in Schwaz/Tirol', in A. Vendl, B. Pichler (ed.), *Wiener Berichte über Naturwissenschaften in der Kunst*, 1, 1984.

11 Another red robe microsample, taken from the praying virgin in the forefront of the Saint Ursula outer-face panel, shows an identical layer build-up to the one described in this chapter.

12 Phosphorus has been found to be typically present in the substrate of lakes produced from scale insects as discussed in Kirby *et al.*, 'The technology of red lake pigment manufacture: study of the dyestuff substrate', in *National Gallery Technical Bulletin*, vol. XXVI, London, 2005.



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