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The Use of Particle Accelerator Analysis to Identify the Origin and Production Processes of 16th-Century Stained-Glass Windows at St Mary's Cathedral in Auch

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L'analyse par faisceau d'ions pour identifier l'origine et les procédés de fabrication des vitraux du XVI^e siècle de la cathédrale Sainte-Marie d'Auch – Résumé

Parmi les vitraux conservés dans la cathédrale d'Auch (Gers, Occitanie), dix-huit, situés dans le déambulatoire, ont été créés entre 1510 et 1513 et signés par le peintre verrier Arnaud de Moles. La restauration des vitraux de la cathédrale a commencé avec la chapelle Saint-Louis (2017-2019), et se poursuit dans les autres chapelles et les baies hautes du chœur. Ce projet de restauration et de conservation a été réalisé par une équipe interdisciplinaire.

Les trois baies de la chapelle Saint-Louis représentent des scènes bibliques. Les premières observations en atelier ont montré un très bon état de conservation. Le projet de restauration visait à nettoyer et réparer les casses des vitraux, ainsi qu'à les protéger par une verrière extérieure. Une restauration est toujours l'occasion d'étendre la connaissance de l'œuvre. Ce projet a bénéficié d'un temps de faisceau à l'Accélérateur Grand Louvre d'Analyse Élémentaire (AGLAE), un accélérateur de particules développé par le Centre de Recherche et de Restauration des Musées de France (C2RMF, Paris). Les spectroscopies

The Use of Particle Accelerator Analysis to Identify the Origin and Production Processes of 16th-Century Stained-Glass Windows at St Mary's Cathedral in Auch – Abstract

Among the stained-glass windows conserved in Auch Cathedral (south of France, Occitanie), eighteen, located in the ambulatory, were created between 1510 and 1513 and signed by the glass-painter Arnaud de Moles. A recent restoration of the cathedral started with Saint Louis' Chapel (2017-2019), then moved on to the remaining chapels and the upper windows of the choir. This restoration and conservation project was carried out by an interdisciplinary team.

The windows of the three bays in Saint Louis' Chapel depict holy characters and biblical scenes. The first observations in the studio showed few lead repairs and a very good state of conservation. The restoration project aimed to clean the stained glass, mend any broken sections and protect the three windows with protective glazing.

A restoration is always an opportunity to extend the knowledge of the artwork in question. This project benefitted from a session at the Accélérateur Grand Louvre d'Analyse Élémentaire (AGLAE), a particle

PIXE et PIGE ont été utilisées pour effectuer des analyses non invasives et non destructives sur trois panneaux afin d'identifier la composition chimique quantitative des verres.

Les résultats ont révélé une composition très homogène pour tous les verres à l'exception des verres pourpres. Des analyses ont été effectuées sur les deux faces et ont révélé des verres rouge, vert et bleu plaqués. La composition du verre a été comparée à celle d'autres sites en France, et une similitude a été trouvée avec la composition du vitrail réalisé dans les premières années du XVI^e siècle pour la cathédrale Saint-Étienne de Sens. Les archives du site de Sens notent que le verre a été acheté à un marchand se fournissant en Lorraine. On peut donc supposer que les feuilles de verre utilisées par Arnaud de Moles, soufflées en manchon suivant la méthode lorraine, provenaient du même fournisseur, situé à mille kilomètres d'Auch.

La qualité exceptionnelle du verre et la remarquable conservation des verrières nous ont permis d'approfondir les connaissances existantes sur les procédés de fabrication du verre coloré et sur les routes commerciales du XVI^e siècle en France.

accelerator developed by the Centre for Research and Restoration of Museums of France (C2RMF, Paris). PIXE and PIGE spectroscopies were used to carry out non-invasive and non-destructive analyses on three panels to identify the quantitative chemical composition of the different pieces of glass.

The results revealed a very homogeneous composition for all the glass samples except for those with a murrey colour. Analyses were carried out on both sides of the glass and revealed the use of flashing for the red, green and blue glass samples. The glass composition was then compared with those of other sites in France, and a similarity was found with the composition of the stained glass made in the first years of the 16th century for Saint-Etienne's Cathedral in Sens. Archives from Sens note that the glass was purchased from a glasshouse in the Lorraine region. We can therefore assume that the glass sheets used by Arnaud de Moles came from the same supplier, located a thousand kilometres from Auch. The exceptional quality of the glass and the remarkable preservation of the windows have enabled us to extend existing knowledge on the manufacturing processes of coloured glass and on the 16th century trade networks used for their commerce in France.

1. Introduction

A conservation-restoration project is always a unique opportunity to examine stained-glass windows in detail. Indeed, their removal from their original position provides an ideal occasion to observe them carefully at very close quarters and understand the production processes used by the glass-painter. The possibility to combine these observations with chemical analyses to determine the composition of both the glass and paints used and then compare the findings with those obtained in other sites and other periods in time is an exceptional opportunity to deepen our knowledge of a work of art. We seized this opportunity to study the stained glass of St Mary's Cathedral in Auch (south of France). The windows were made by the glass-painter Arnaud de Moles between 1510 and 1513. Their restoration began in 2017 and involved an interdisciplinary team made of curators, conservators, architects, historians and scientists. Three window panels were analysed with ion beam techniques to identify the chemical composition of the glass and improve our knowledge of 16th-century French stained glass. This data was compared with findings collected from other French sites from the same period, and the results are presented in this paper.

2. Historical background

The reconstruction of the cathedral choir began ca. 1493. Shortly afterwards, the chapter clergy funded the complete decoration of the choir, including the glazing of the ten chapels surrounding the ambulatory. A total of eighteen monumental stained-glass windows were installed in their triple or quadruple lancet bays, crowned by complex and flamboyant tracery lights. The completion date of June 1513 is inscribed on a window panel of the last southern bay beside the name of the glazier Arnaud de Moles, presumably the main actor of this ambitious project, which was carried out over just three or four years¹.

This spectacular series is striking in terms of the formal coherence of its iconographic program, an illustration of the concordance between the Old and New Testament that is organized into an innovative scenography. This comprises three subjects in three windows which resume the redemption (Original sin, Crucifixion and Resurrection), and in the fifteen remaining windows, forty-nine tall standing figures which are depicted on various decorative backgrounds. These patriarchs, prophets, sibyls and apostles compose typological groups placed in each of the fifteen remaining windows, above small-format « predellas » illustrating related scenes. In addition to the architectonic variety of the compositions, the disparate tracery lights are filled with innumerable motifs, angelic musicians, allegories, coats of arms and other details.

Despite a clear scholarly interest from the early 19th century to the current day,² little is known about the origin of templates and patterns which appeared through new approaches that closely resembled sketches by the Milanese contemporaries of Leonardo da Vinci, and were provided for those implementing the stained-glass window projects. From a technical standpoint, these designs were applied by highly skilled craftsmen who used grisaille to obtain different visual effects on a range of glass colours. The remarkable quality of the pieces of glass allowed large sections to be cut and the insertion of numerous « jewels » in the panels, as well as abrasion or etching on the surfaces of coloured flashed glass. This material was very resistant to alterations, and needed little restoration throughout the following centuries, as confirmed by documentary evidence and archaeological observation. The main restoration campaign was carried out in 1872-75 by the Parisian glass-painter Emile Hirsch, who introduced minimal modern additions to this well-preserved glazing.

3. Restoration project

The restoration project at St Mary's cathedral in Auch began with the chapel of Saint-Louis (2017-2019). This project was launched by the regional cultural affairs directorate (the main project manager) and was carried out by Stéphane Thouin, the chief architect and contractor. The restoration of the stained-glass windows was entrusted to Anne Pinto and Claire Babet, two qualified conservators who worked together on this project. A scientific committee was named to attend the restoration and participate in decision making. It was made up of a cross-section of the different restoration actors: architects, conservators and curators, inspectors from the Ministry of Culture; art historians from the

¹ GATOUILLAT 2020, p. 191.

² GATOUILLAT 2020, p. 195.

Centre André Chastel and conservation scientists from the *Laboratoire de Recherche des Monuments Historiques (LRMH)*. Saint-Louis' chapel has three bays (numbers 4, 6 and 8) with windows representing holy characters and biblical scenes (fig. 1). All the panels were removed and brought to the conservators' studios for restoration under the supervision of the scientific committee. The project manager then decided to protect the stained-glass windows with a protective glazing placed in the original frame rebates.

3.1. Conservation process



The first observations made on the panels showed a very good state of conservation of both the glass and the paint. Little mending lead was noted and the glass corrosion was very limited. The restoration was carried out in the conservators' studios and included the cleaning of the glass and the restoration of broken pieces of glass. Following the code of ethics, the mending lead was then replaced by silicone or epoxy edge-bonding when the two pieces of glass were contemporary or by copper foils when they were from different periods and restorations. Lead came was preserved as much as possible and only replaced when needed. The metalwork was treated as follows: when set into the masonry, lug-bars were stripped and treated with anti-corrosion products on site. Since the panels were placed in front of their original frame after restoration, the saddle bars were too short. New ones were made for the stained-glass windows and the original ones were preserved and placed on the protective glazing. A wide lead frame was fixed on all panels to fill the gap between the window and the masonry. Finally, new lug-bars were fixed to the old ones with clamps and steel-threaded rods in order to prepare the metalwork for the protective glazing. All original metals and new metallic objects added to this framework were treated to prevent corrosion, which is particularly common when different metals are used.

Fig. 1. Auch, St Mary's Cathedral, bay 6, internal face, transmitted light. Panels chosen for AGLAE analyses: a) Bay 6, panel A1; b) Bay 6, panel c8; c) Bay 6, panel b6. © C. Babet, 2018.

3.2. Protective glazing

The protective glazing prevents any further deterioration of the stained glass by stopping the formation of condensation on the internal face of the panels and protecting their external face from rain, wind and pollution. In order to be efficient several parameters must be respected, namely a distance of 3.5cm between the protective glazing and the stained-glass window, a natural air flow from inside the building and the installation of lead flashing at the base of the protective glazing to collect water from the condensation. The choice of window is purely based on the aesthetic considerations for each site, and in the case of Auch cathedral, the curator and the architect chose to use lead-glazed windows, placed in the original frame rebate.

4. Glass analysis research

Since 2006, a long-term research project by the LRMH and the *Centre de Recherche et de Restauration des Musées de France* (C2RMF) has concentrated on the use of the AGLAÉ particle accelerator (*Accélérateur Grand Louvre d'Analyses Élémentaires*) to analyse stained glass. Beam time access is granted twice a year and when stained-glass windows are under restoration, this provides a unique opportunity for researchers to study the chemical compositions of both glass and paints and thus extend current knowledge of French stained glass. AGLAÉ has high analytical performances and is non-invasive and non-destructive, as no samples are taken from the objects and the materials analysed are not modified in any way. In the case of stained glass, the panels can be placed directly in front of the beam. A mechanised system then obtains numerous measurements from their surface. This analytical technique is therefore well-fitted for inorganic compounds and precious works of art such as stained glass.

4.1. Experimental protocol

4.1.1. Instrumentation

AGLAÉ is the only particle accelerator that is solely dedicated to works of art, and has thus been located at the Louvre museum since 1988³. This instrument uses ion beam methods and is based on the interaction between light particles (protons, deuterons and alpha particles) and the atoms constituting the materials under study. Several detectors can be used to collect different emissions, including Particle Induced X-ray Emission (PIXE) and Particle Induced Gamma Emission (PIGE). The PIXE method is based on X-ray emission following the interaction between the inner electrons of an atom and the ion beam. This method allows the quantitative detection of chemical elements and particularly trace elements. It is coupled with the PIGE method, which is based on the emission of gamma-rays and can detect light elements such as sodium or magnesium.

4.1.2. Analytical parameters

Analyses are carried out on the surface of the panels (glass and paint) after prior selection of the least affected or unaffected areas. A proton beam set at a power of 3 MeV scans a surface of 500 x 500 µm. A detector is used to collect the gamma-ray from the PIGE emission, whilst the X-ray from the PIXE emission is collected by other detectors dedicated to low or high energies. All detectors are used simultaneously. A 40-mm helium flux is set up in front of the low energy detector to reduce the absorption of both the beam and the X-rays in the air. Aluminium filters with a thickness of 50 µm are placed in front of two high energy detectors (HE1 and HE3), and a 100 µm-thick aluminium filter is placed in front of the last high energy detector (HE4). This thick filter is used to enhance the detection of antimony and tin reflecting the amount of lead in the paintings. Three measurements are collected and averaged for each sample. National standards and reference materials (Brill standards and NIST 620) are used at the beginning and end of every day of analyses in order to calibrate PIGE data and monitor the accuracy of the quantitative data obtained from PIXE results. PIXE spectra are processed with GUPIX© software,⁴ using iron as the pivot element. Calibration is then carried out using the PIGE values collected for sodium.

³ PACHECO et al. 2016, p. 63-69.

⁴ PICHON et al. 2015, p. 48-54.

4.2. Samples

Three panels were chosen in the conservators' studios to obtain a representative sampling of all the colours and paints found across the three bays. These were B6A1, B6b6 and B6c8 (fig. 1). This choice was made after discussion with the art historians and conservators to ensure that panels with as many original glass pieces as possible were selected. Analyses were carried out on both sides of the panels when possible and were obtained for the glass (fig. 1) and the paint, namely grisaille, yellow stain and sanguine. Only the results for the glass samples are presented here. The list of all the glass samples is presented in Table 1.

4.3. Results and discussion

The quantitative analysis of the different pieces of glass was instrumental in determining the proportions of the different raw materials used by the glass-maker during the production process. The main raw materials today are the same as those used in the past, namely vitrifying agents (sand, quartz), fluxing agents (natron, plant ash) and stabilizers (alumina and alkaline earth oxides). Fining agents include manganese oxides (which are used as decolorizing agents), sulphates, chlorides and nitrates that are all already present as impurities in raw materials⁵.

Around 60 % of the composition of the glass at Auch cathedral is attributable to silicon dioxide (SiO₂), and the remaining 40 % is made up of high levels of calcium, potassium and/or sodium oxides (CaO, K₂O and Na₂O), with significant amounts of chlorine, as well as magnesium and phosphorus oxides (Cl, MgO, P₂O₅) depending on the type of ash used (forest or marine plants)⁶.

4.3.1. Quantitative composition of the glass samples

A similar composition was revealed for all the colours on all the glass pieces studied in this project, except for the murrey samples (Tables 2a and 2b). Indeed, the blue, red, green, white, yellow and orange glass was composed of approximately 60 % of silicon dioxide (SiO₂), 20 % of calcium oxide (CaO), 5 % of potassium oxide (K₂O) and around 2.5 % of sodium oxide (Na₂O). They can be classified in the high-lime, low-alkali (HLLA) group⁷. The composition of these coloured sections also contains around 2.5 % of magnesium oxide (MgO), 0.5 % of chlorine (Cl) and nearly 3 % of phosphorus oxide (P₂O₅). The composition of the murrey glass sections is different, with 55 % of SiO₂, 16 % of CaO, 15 % of K₂O and less than 1 % of Na₂O, whilst MgO, Cl and P₂O₅ concentrations are around 5 %, 0.1 % and around 1.5 %, respectively. Given that the hierarchical classification of stained-glass fragments is based on their major composition,⁸ the murrey glass samples cannot be placed in the HLLA group due to their potash content.

Table 1: List of samples

Colour	Sample	Face analysed	
		External Face (EF)	Internal Face (IF)
Blue	B6a1_B1	×	-
	B6b6_B1	×	-
	B6b6_B2	×	-
	B6c8_B1	×	×
	B6c8_B2	×	-
Green	B6a1_G1	×	×
	B6b6_G1	×	-
	B6b6_G2	×	×
	B6b6_G3	×	-
	B6b6_G4	×	-
	B6c8_G1	×	-
	B6c8_G2	×	×
Murrey	B6b6_M1	×	-
	B6b6_M2	×	-
	B6b6_M3	×	×
	B6c8_M1	×	-
Orange	B6a1_O1	×	-
Red	B6a1_R1	×	×
	B6b6_R1	×	-
	B6b6_R2	×	-
	B6c8_R1	×	×
	B6c8_R2	×	×
B6c8_R3	×	-	
White	B6a1_W1	×	×
	B6a1_W2	×	-
	B6a1_W3	×	-
	B6b6_W1	×	-
	B6b6_W2	×	-
	B6c8_W1	×	-
	B6c8_W2	×	×
Yellow	B6b6_Y1	×	-

⁵ MORETTI & HREGLICH, p. 23-47.

⁶ HENDERSON 2013, p. 83-126.

⁷ HARTMAN 1994, p.103-128 and SCHALM et al. 2007, p.663-668.

⁸ SCHALM et al. 2007, p.663-668.

4.3.2. Flashed glass

When considering the concentration of metallic oxides responsible for the coloration of glass, the surface on which the analysis has been made (i.e. internal or external face) appears to play an important role. Indeed, the literature reports that although the use of glass-flashing is necessary to ensure transparency when making red glass, this procedure is not necessary for the other colours⁹. However, the present study reveals that the glass-flashing process was used not only for the red samples but also for the blue and green sections. The concentration of cobalt and copper oxides (CoO, CuO) producing the blue colour in the blue glass sections ranges from 0.01 % to 0.09 % and from 0.02 % to 0.17 %, respectively. The lowest concentrations of CoO and CuO are found in the same sample (B6c8_B1). They are quite low for a blue glass,¹⁰ and are similar to the concentrations found in the white glass samples. The analysis of sample B6c8_B1 was only carried out on one face of the piece, and the use of flashed glass can therefore only be presumed. It is however clear that the flashed glass process was used for the green glass. Indeed, the analysis of one sample on both sides shows different concentrations of CuO, with 0.6 % on the internal face and 0.3 % on the external face. Another sample reveals equal concentrations on both faces, with just under 0.2 % CuO and less than 0.05 % of lead oxides (PbO) which seems quite low for green glass¹¹. The hypothesis of a double-flashed glass can then be made, the green glass core being covered by white glass layers on both sides. Finally, analysis of the red glass samples revealed an identical CuO concentration of 0.05 % on both sides of all three samples. This concentration is relatively low compared to the CuO concentrations found on stained-glass samples from other sites. For example, the CuO concentrations in the red glass on the rose of the Sainte Chapelle in Paris range from 0.03 % to 0.5 % on the same piece of glass, depending on the side that is analysed¹². The assumption can therefore be made that double-flashed glass was also used for the red glass pieces in Auch cathedral.

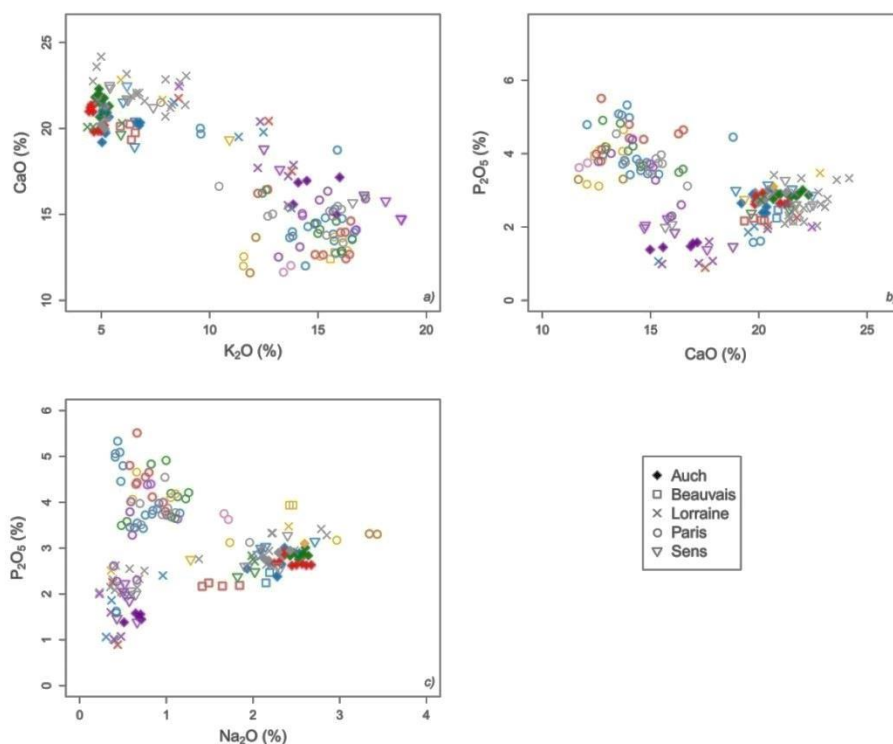


Fig. 2. The chemical compositions of 16th-century stained glass from different sites in France. The graphs show the ratio between two chemical elements:

a) $\text{CaO}/\text{K}_2\text{O}$

b) $\text{P}_2\text{O}_5/\text{CaO}$

c) $\text{P}_2\text{O}_5/\text{Na}_2\text{O}$

The colour of each symbol indicates the colour of the glass sample analysed, and uncoloured glass is shown in grey.

⁹ KUNICKI-GOLDFINGER et al. 2014, p. 89-105.

¹⁰ WEDEPOHL 1997, p. 883-891.

¹¹ WEDEPOHL 1997, p. 883-891.

¹² The analyses were carried out by the LRMH using an identical protocol for all samples, and the results have yet to be published.

4.3.3. Comparison with other French sites

The results of this study were compared to the concentrations recorded for other contemporary French stained-glass samples from three different locations, namely the western rose of the Sainte Chapelle in Paris, the southern rose of Sens cathedral and the northern rose of Beauvais cathedral. The windows in Sens cathedral were made between 1500 and 1502 with glass produced in the Lorraine region¹³. The original source of the glass used for the Sainte Chapelle rose, built between 1483 and 1498, is not known¹⁴. This information is also lacking for the glass in panels at Beauvais cathedral, which are inscribed with the date 1537¹⁵. The samples from Beauvais cathedral are no longer on site in their original bay (323) as it was destroyed by bombing during the Second World War. A further set of archaeological samples from Lorraine (La Vôge) were considered in this data analysis. These fragments of glass were retrieved in 1981 during excavation on the site of a glass factory that was probably operational between 1448 and 1612¹⁶. The chemical compositions of the stained-glass samples from Paris and Beauvais were obtained with AGLAÉ between 2014 and 2019, whilst composition data for the stained glass from Sens and the archaeological samples from Lorraine were obtained with an electron microprobe¹⁷ between 2015 and 2016. The results show two to three distinct groups. Indeed, in figure 2.a two clusters can be observed. The first mainly concerns the samples from Sens, Lorraine and Auch, with a few exceptions for some (mostly murrey) coloured glass samples. The majority of the murrey samples are found in the second group, which is mainly made up of the Sainte-Chapelle samples. The glass samples from Beauvais are divided between the two groups. In terms of the CaO/K₂O ratio, the first group is described as wood ash lime glass (with a CaO/K₂O ratio of around 3.4) and the second as wood ash glass (CaO/K₂O ratio is around 1)¹⁸. However, figures 2.b and 2.c highlight that there are three groups as the murrey glass samples from Auch, Sens and Lorraine are not located within the Sainte-Chapelle cluster. However, with these chemical ratios, it is not clear which group the samples from Beauvais belong to.

The glass samples from bay 6 in Auch cathedral have a very homogeneous composition. They can be considered to form two groups: HLLA glass, and potash glass (i.e. the murrey samples). These two groups are defined by Schalm et al as *type A* and *type B*¹⁹. Type A glass is made using beechwood ash and lime as fluxing agents, while beechwood ash is the only fluxing agent used to make type B. The concentrations of P₂O₅ and MgO in the third group containing the Sainte-Chapelle samples are higher than those observed in the other groups, indicating that these pieces of glass may have been made with fern ash²⁰.

5. Conclusions

The conservation and restoration project of the Saint Louis chapel in Auch cathedral provided a unique opportunity to carry out a detailed study of the stained glass made by Arnaud de Moles. The observations and the analyses indicate that the glass came from the Lorraine area, and suggest that a different manufacturing process was used to produce the murrey glass. Future restoration projects for the other chapels will hopefully bring this interdisciplinary team together again and will provide further details to enrich our knowledge of stained glass. The AGLAÉ analyses will continue to allow the collection of multiple data and will provide the opportunity to use statistical treatments to pinpoint the differences between different types of glass and broaden our understanding of 16th-century stained glass in France. This restoration has also been a means to promote Auch Cathedral via the six-month exhibition run by the *Musée des Augustins* (a fine arts museum) in Toulouse presenting the panels to visitors after their restoration²¹.

¹³ MINOIS 2005, p. 111, 429.

¹⁴ HEROLD et al. 2016, p. 283-294.

¹⁵ GRODECKI et al. 1978, p. 177-181.

¹⁶ DELEMONTEY 2009, p. 461-465.

¹⁷ CAMPARIS, CNRS-UMR 7097, University of Jussieu (Paris).

¹⁸ WEDEPOHL & SIMON 2010, p. 89-97.

¹⁹ SCHALM et al. 2004, p. 1647-1656.

²⁰ WEDEPOHL & SIMON 2010, p. 89-97.

²¹ COHENY 2018, p. 100-104.

Correlated with the results obtained for the chemical composition of the glass, the good state of conservation observed for these windows are a clear testimony to the well-preserved identity of the cathedral. The installation of protective glazing is therefore crucial for the long-term conservation of this monument.

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