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# Potentials and limitations of provenance attribution through material analyses: new insights on the Imperial Crown of the Holy Roman Empire

# Vantagens e limitações da atribuição de proveniência através da análise material: novas perspetivas sobre a Coroa Imperial do Sacro Império Romano

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**Abstract**

This study discusses the potentials and limitations of non-destructive material analysis by providing insights into the current state of research on the composition and specific characteristics of the materials used for the Imperial Crown, including gold, gemstones, pearls, *niello* and enamel. The *cloisonné* enamel plaques are of particular interest, given that glass is a complex mixture of different raw materials. The following complementary non-invasive methods were applied: digital microscopy, X-ray fluorescence analysis, Raman, optical absorption and reflection spectroscopy and multiband imaging. Determining the materiality proved to be a complex task, due to a number of restrictions. These included the limited range of suitable methods, a lack of knowledge regarding specific materials and established manufacturing traditions, such as recycling, or the presence of overlapping characteristics among materials of different origins. Although the origin of the crown itself cannot yet be determined, the analytical results provide a reliable basis for further research.

**KEYWORDS**
Imperial Crown
Medieval
Gemstones
Enamel
Material analyses
Provenance

**Resumo**

Este estudo discute o potencial e as limitações da análise não destrutiva de materiais, fornecendo perspetivas quanto ao estado atual da pesquisa sobre a composição e as características específicas dos materiais usados na produção da Coroa Imperial, incluindo ouro, pedras preciosas, pérolas, *niello* e esmalte. As placas de esmalte *cloisonné* são de particular interesse, pois o vidro utilizado é uma mistura complexa de diferentes matérias-primas. Foram usados métodos complementares não invasivos: microscopia digital, fluorescência de raios X dispersiva de energias, espectroscopia Raman, espectroscopia de absorção e reflexão ótica e imagem multiespectral. Determinar os materiais foi complexo, devido a restrições, como a gama limitada de métodos usados, a falta de conhecimento sobre os materiais e os processos tradicionais de fabrico (reciclagem), ou a presença de características sobrepostas entre materiais de diferentes origens. Embora a origem da coroa ainda não possa ser determinada, os resultados analíticos forneceram uma base fiável para investigação futura.

**PALAVRAS-CHAVE**
Coroa Imperial
Medieval
Pedras preciosas
Esmalte
Análises de materiais
Proveniência

## Introduction

The Imperial Crown of the Holy Roman Empire, in the following referred to as Imperial Crown, is a medieval goldsmith object consisting of eight crown plates connected by hinges, a cross in the front and a single arch on top (Figure 1). Notably, the circlet, cross and arch originate from different manufacturing processes. All structural and decorative components are made of gold and adorned with gemstones and pearls. A distinctive feature is the presence of four cloisonné enamel plaques integrated into the systematic gemstone-pearl-arrangement on the crown plates. Niello is used on the back of the cross. An additional velvet cap from the seventeenth or eighteenth century completes the ensemble. This crown stands out as a unique symbol of European history in addition to its precious materials and exceptional craftsmanship. In contrast to other crowns, this one has been preserved over time due to its legendary association and adoration with Charlemagne (r. 768-814) and its persistent use in coronation ceremonies in the Holy Roman Empire. Since 1827 the crown is on exhibit in the Imperial Treasury in Vienna, Austria.

Since the eighteenth century this crown has attracted scholarly interest, resulting in an extensive body of literature from various disciplines. Several attempts have been made to summarize the large number of publications [1, pp. 23-28; 2, p. 11; 3, pp. 125-133; 4, pp. 13-50]. A frequently discussed question deals with the time and place of its origin, leading to a complex array of different opinions and hypotheses. The proposed dates cover a period of over 200 years (approximately 960-1150). Similarly, the localization of its origin has also been disputed, suggesting regions within present-day Germany, France, Italy or "Byzantium". In recent decades, there has been an increasing awareness of the necessity for a comprehensive technological and archaeometric investigation into the crown's structure and materials, as well as the changes, damages and repairs undergone over its long history of use, in order to provide a reliable basis for all studies on the crown as an artefact [5, p. 314; 6, p. 188; 7, p. 17].

**Figure 1.** Imperial Crown of the Holy Roman Empire, 10th-12th century, diameter approx. 26 cm (photo: Christian Mendez, KHM-Museumsverband).


**Figure 2.** Interdisciplinary research questions covered within the CROWN project.

Hence in 2022 the CROWN project was conceptualized at the Kunsthistorisches Museum, aiming to gain insight into the technology, materials, and state of preservation of the Imperial Crown. It covers a large number of research questions given by art historians, historians, restorers and conservation scientists (Figure 2). This paper focuses on the systematic investigation and interpretation of the materials used. Suitable and accessible analytical methods are presented, including the discussion of their potentials and limits in provenance attribution. Moreover, challenges – such as managing different types of data, as well as general limitations in the methodical concept – are addressed. Research questions concerning art historical and historical perspectives or manufacturing techniques are beyond the scope of this study.

## Materials and methods

The applied methodological concept for the material analysis of the Imperial Crown was developed based on the project's research questions and available facilities, all within the limitation of a non-destructive, in-situ approach. Table 1 provides an overview of the materials investigated and the analytical methods used.

**Table 1.** Investigated materials of the crown and the applied analytical methods.

| Material | Analytical method | Scope |
|---|---|---|
| Gold components (~1,000) | digital microscopy | ~ 15,000 images/ scans/ videos |
| | µ-XRF | ~ 60 measurements |
| Gemstones (172) | digital microscopy | ~ 5,000 images/ scans/ videos |
| | Raman spectroscopy | ~ 200 measurements |
| | PL spectroscopy | ~ 100 measurements |
| | µ-XRF | ~ 280 measurements |
| | UV-Vis-NIR spectroscopy | ~ 110 measurements |
| | Multiband imaging | ~ 80 images |
| Pearls (233) | digital microscopy | ~2,000 images/ scans/ videos |
| | µ-XRF | ~200 measurements |
| | Multiband imaging | ~ 80 images |
| Niello (1 plaque) | digital microscopy | 1 scan (30×) |
| | µ-XRF | 5 measuring points |
| Enamel (4 plaques, 11 colours) | digital microscopy | ~ 6,000 images/ scans/ videos |
| | µ-XRF | ~ 200 measurements |
| | MA-XRF | 8 scans, ~ 30 measurements |
| | Raman spectroscopy | ~ 140 measurements |
| | FORS | ~ 50 measurements |
| | Multiband imaging | ~ 60 images |

## Materials of the Imperial Crown analysed

### *Gold*

As all parts of the crown are made of gold, the question of the composition of the alloy used for each individual component is of special interest. From the eighteenth century onwards, gold compositions are cited in the literature referable to Christoph Gottlieb Murr [8, pp. 1-15] However, the method used at that time is unclear, though it probably involved touching tests. Murr described the crown as being made of 21-karat gold, noting that the enamel plaques were crafted with 24-karat gold, while the arch was produced using a 19-karat alloy. These historic values are being reassessed and complemented by using modern analytical methods as far as possible.

### *Gemstones and pearls*

The lavish use of gemstones on the Imperial Crown is one of its most distinctive features, reflecting its status and value during the medieval period and beyond. However, the preserved inventory of 172 coloured stones and 233 pearls has undergone numerous alterations and cannot be directly equated with the crown's original appearance. Many of them – especially pearls – were secured to their settings with repair wires over time, remaining in this provisional state until today (Figure 3).

The identification of the gemstones on the Imperial Crown has been addressed in the literature at a rather general level thus far [9]. Determining the geological and, in some cases, even the geographic origin of a gemstone is a highly complex task [10]. Nonetheless, one objective of the investigation was to expand knowledge by means of clustering groups according to their similar optical and/or chemical characteristics in order to draw conclusions about historic manufacturing processes and ancient trading practices.


**Figure 3.** Garnet (C_St_30) secured to the damaged setting (C_Fa_30) by a golden repair wire (C_Ei_10) (photo: Herbert Reitschuler, KHM-Museumsverband).

***Niello***

On the backside of the front cross of the crown is a depiction of Christ crucified (Figure 4). The black material used to highlight the fine incised lines is known as niello – a material produced by fusing metals (silver, copper, lead) with sulphur. This technique has evolved over time, leading to several manifestations that coexist and are expressed through different base components or application methods [11-13].


**Figure 4.** Detail of the niello decoration on the backside of the front cross (KK_NT) (photo: Herbert Reitschuler, KHM-Museumsverband).

In art-historical observations, the niello plaques on the Imperial Cross of Conrad II are cited as being stylistically similar [14]. As this object is also in the Imperial Treasury, material analyses could be applied to study this hypothesis on the basis of a comparison of the chemical composition.

***Enamel***

The Imperial Crown features four plaques with enamelled images executed in cloisonné technique (Figure 5)**.** Each plaque is mounted on the crown plates in bezel settings that match the unique shape of the plaques, distinguished by small indentations on the left and right sides beneath the semi-circular fields. The figurative depictions and inscriptions on the plaques were subject to many iconological studies [6, 15]. In contrast, technological observations have not been available in a sufficient and reliable form so far [1, p. 78; 16, p. 103]. Enamel is defined as glass fused onto a recipient – usually metal [17]. Six opaque (white, light blue, turquoise, yellow, flesh tone, red) and five transparent (light green, blue, brown, black) to semi-transparent (dark blue) coloured glasses were used to fill the cloisonné cells on the plaques of the Imperial Crown.

Glass is a complex material consisting of numerous ingredients that vary according to changing processing traditions and raw material sources across different regions and historical periods. Different glass compositions therefore can provide insights into production-related characteristics; thus, the enamel plaques are a central focus of the current research. However, non-destructive glass analysis also posed some challenges that had to be taken into account in the interpretation of the results.

The main component of glass is silica, historically derived from quartz sand or pebbles. Alkaline fluxes – primarily sodium (from mineral natron or halophyte plant ash) or potassium (from wood ash) – were added to lower the melting point. Calcium, either naturally present in the sand or intentionally added (from shells or limestone), acts as a stabilizer within the glass matrix [18]. Based on their composition, different glass types can be identified. Roman and Islamic soda-lime glass, following long-established traditions, are divided into types based on mineral sodium sources (natron glass) or plant-based sodium sources (plant-ash glass) [19]. Another type, known as wood-ash glass, was available in Northern Europe from the ninth century onwards [20]. The raw materials for glass production were likely sourced locally, allowing researchers to define sub-groups to a certain extent, that are sometimes linked to specific regions [21].


**Figure 5.** Solomon plaque (plate B): detail of the cloisonné enamel (photo: Herbert Reitschuler, KHM-Museumsverband).

Producing this so-called raw glass from the basic ingredients required high temperatures accomplished by specially constructed furnaces. Consequently, it is believed that only a few centralized production sites existed from where the raw glass was distributed along various trade routes [22]. Based on archaeological findings, primary production centres of soda-lime glass were probably located in Egypt, the Levant, and South-west Asia [23]. For wood-ash glass, however, less geographically bound raw materials were required, making the identification of production sites even more challenging [24]. The raw glass is further modified by adding colouring agents or opacifiers in a secondary production step, either at the primary production sites or in specialized centres alongside the trade routes [25]. Evaluation of glass composition results from the cloisonné plaques may repeal the extent to which they can be integrated into the current state of research.

**Data management**

To facilitate a detailed description of the crown, the object was conceptually divided into approximately 1,750 components, identifiable through a customized labelling system. Each individual element – from hinge tubes and settings to gemstones and enamel colours – was assigned a unique inventory number (e.g. A_Fa_1 for the first setting on the front plate and A_Pe_1 for the pearl mounted in this setting; or F_ET_Em_otue for the opaque turquoise enamel on the Ezechias plate). This step was essential for ensuring the traceability of all collected data. A workflow was developed based on a customized version of the museum´s internal database (TMS, Getty Trust) that enabled the documentation of specific information on each component. Including various phenomena from the optical appearance and the occurrence of damage or repairs extending to details like inclusions in a gemstone or particle sizes in the enamel layer. As the integration of images into the internal database did not proceed as required, an additional system (Goobi, Intranda) had to be implemented to facilitate image evaluation and tagging.

**Analytical methods**

***Digital microscopy***

Using a modern optical 3D microscope (HRX-01, Hirox), numerous high-resolution, multi-focused scans and detailed images of all components were captured using various lenses, stands, and lighting conditions. Approximately 30,000 images, videos and scans were collected, providing a reliable basis for further research.

***Energy dispersive X-ray fluorescence analysis (XRF)***

To determine the chemical composition of the materials, two energy dispersive X-ray fluorescence (XRF) spectrometers from the museum's Conservation Science Department were utilized: a micro-XRF instrument (PART II) and a macro-XRF scanner (CRONO).

The Portable ART Analyzer II (PART II) was developed addressing challenges specific to heritage science [26]. The detection head of this instrument is equipped with a vacuum chamber, enhancing sensitivity to light elements (Na upwards). A close 1 mm distance to the object minimizes absorption in air. Additionally, a *polycapillary* lens focuses the excitation beam to approximately 150 µm diameter, allowing for highly targeted measurements, though with reduced sensitivity to higher-energy fluorescence lines. This instrument, equipped with a Pd tube, was applied to analyze all crown materials (gold, gemstones, pearls, niello, enamel), with measurement parameters (kV, mA, measuring time, Al-filter) individually adjusted for each material.

The CRONO MA-XRF scanner by Bruker [27], also equipped with a Pd tube, was employed for both single-point measurements and elemental scanning on the enamel plaques, with spot sizes of 0.5 and 1 mm achieved using different collimators. Measurements were performed in air at approximately 1 cm distance, limiting the detection of light elements (Na-Ca). However, compared to the μ-XRF instrument, the CRONO instrument shows a better detection in the high-energy region, as demonstrated in the results.

***Raman and photoluminescence spectroscopy***

With two different setups, the gemstones and enamel of the Imperial Crown were examined for spectroscopic characteristics. The 172 gemstones were analysed using Raman and laser-induced photoluminescence (PL) spectroscopy using a confocal dispersive WITec UHTS 300 VIS spectrometer system equipped with fibre-coupled probe head. Spectra were excited using a blue laser (457 nm; maximum power 8.5 mW). A 20× objective (free working distance 25 mm) was used to focus the laser light onto the sample and to collect the scattered/emitted light. For more details see [28].

The enamel was analysed by means of a portable fibre-coupled BWTEC i-Raman Plus system (supported by the European programme IPERION HS). Excitation was mainly achieved with a 532 nm laser source and on some additional measuring points with 785 nm. Focusing on three measuring points per colour and plaque, 20× and 40× objectives were used.

***Complementary material-specific analytical methods***

Multiband imaging was performed on all parts of the crown. Images were taken under visible (3000 K and 5000 K light colour), ultraviolet (UV: 254 nm and 365 nm), and infrared light illumination, from which false-colour images were generated. Material-specific phenomena such as luminescence of gemstones, pearls and some enamel colours are made visible through this method.

Optical absorption (often referred to UV-Vis-NIR) spectra from 250 to 950 nm of the gemstones were acquired using a mobile instrument from Ocean Insight USB2000+ coupled via fibres with a DH-2000 deuterium-halogen light source, with an acquisition time of 2 s (2 cycles) and with a spectral resolution of 2 nm.

Enamel was studied with fibre optic reflectance spectroscopy (FORS) in the spectral range 300-1000 nm. It was performed on the eleven different colours on all four plaques. The instrument used was a CHSOS Gorgias reflectance spectrometer for art equipped with a fibre-coupled halogen light source and Toshiba TCD1304DG detector.

Although generally considered as non-invasive, computer tomography (CT) was excluded from the concept, as the risk of any changes associated with high levels of radiation to the gemstones and pearls was deemed too unpredictable.

***Comparative studies***

To enhance understanding of the Imperial Crown by contextualizing the optical and analytical data obtained from the enamel plaques, comparative measurements – based on the same analytical methodology – were performed on gold cloisonné objects in Essen, Munich, Cologne and Vienna. These comparative objects were selected from the limited number of preserved medieval examples based on their frequent citation alongside the crown in the literature and the feasibility of investigating them within the project's resources. Optical investigations were performed at the respective localisations using the digital microscope from the Kunsthistorisches Museum, while XRF, Raman and FORS analyses were facilitated through collaborations with the Rathgen-Forschungslabor, Berlin, and the Bayerische Staatsbibliothek, Munich. Extending the investigations to a larger group of enamel objects not only allows the identification of potential correlations with the crown, but also enhances the general knowledge of medieval goldsmithing traditions, especially enamelling techniques.

## Results and discussion

### Gold

Due to the intricate three-dimensional structure of the gold work and the small measuring distance provided by the µ-XRF instrument, it was not feasible to systematically examine all components. For example, it was not possible to determine the alloys used for the base plates at all. Consequently, the gold analysis remains incomplete, preventing further statistical evaluation of alloy compositions that might provide insights into the crown's manufacture. µ-XRF analyses, however, confirmed differences in composition among the circlet, the arch, and the cloisonné plaques. The latter consist of metal sheets that, when compared to other parts of the crown, show a higher gold content (Au ~98 wt %). This result comes very close to the 24-karat gold mentioned by Murr [8, pp. 1-15]. However, whether these significant differences result from technological factors or suggest a separate production process for the circlet and the enamel plaques cannot be determined from this evidence alone.

Insights into a possible provenance attribution of gold would involve analytical methods capable of revealing trace element and isotope chemistry. Such highly sensitive analyses require sampling (e.g. Laser Ablation) and were therefore not suitable for this project. Even with access to these methods, attributing gold to specific sources or mining regions remains challenging to impossible, as recycling and metallurgical modifications were already common practice in the Middle Ages. For non-antique gold objects it is therefore not possible to assign gold compositions to specific sources or mining regions [29].

### Gemstones

Visual examination reveals a diversity of shapes and traces of processing, suggesting that many stones were reused from earlier contexts before being mounted on the crown. This is particularly evident in the case of two ancient intaglios that were documented in the course of the microscopic investigation on the inner sides of the circlet [30]. Unfortunately, there is still limited knowledge about early traditions in gemstone processing. Considering ancient Indian diamond-drilling techniques and the emergence of polished pyramidal surfaces on sapphire in seventh century Europe, nearly all of the crown´s gemstone processing techniques are consistent with medieval or earlier traditions. Analysis of the gemstones using Raman and PL spectroscopy identified 71 blue corundum (sapphire), 50 differently coloured garnets, 20 green Cr-bearing beryl (emerald), three pink to red spinel, 13 purple quartz (amethyst), four variably coloured chalcedony and 11 artificial glass imitations [28]. Raman analysis also enabled the assignment of different garnet types by comparing characteristic band positions with secured reference materials and provided additional details on specific stones. One of the key findings of the spectroscopic analyses was that Raman and PL spectra (Figure 6) provided evidence for heating to at least 1000 °C of the huge central spinel mounted on the front plate (A_St_25) [28]. Moreover, it represents – to our knowledge – the earliest documented example for the use of such a transparent red gemstone.

To assess the geological and/or geographical origin of a gemstone, it is essential to combine various observations and analytical investigations, such as characteristic inclusions, the chemical composition and UV-Vis-NIR spectroscopy. Challenges arise from overlapping features of gemstones from different mining areas [10]. This is particularly true for sapphire [31] and the quartz variety amethyst, where, due to the mineral's common occurrence, similar inclusion patterns and/or chemical composition the determination of a stone´s provenance is nearly impossible [32]. Furthermore, there is a lack of knowledge about mining sites that were active over a thousand years ago.

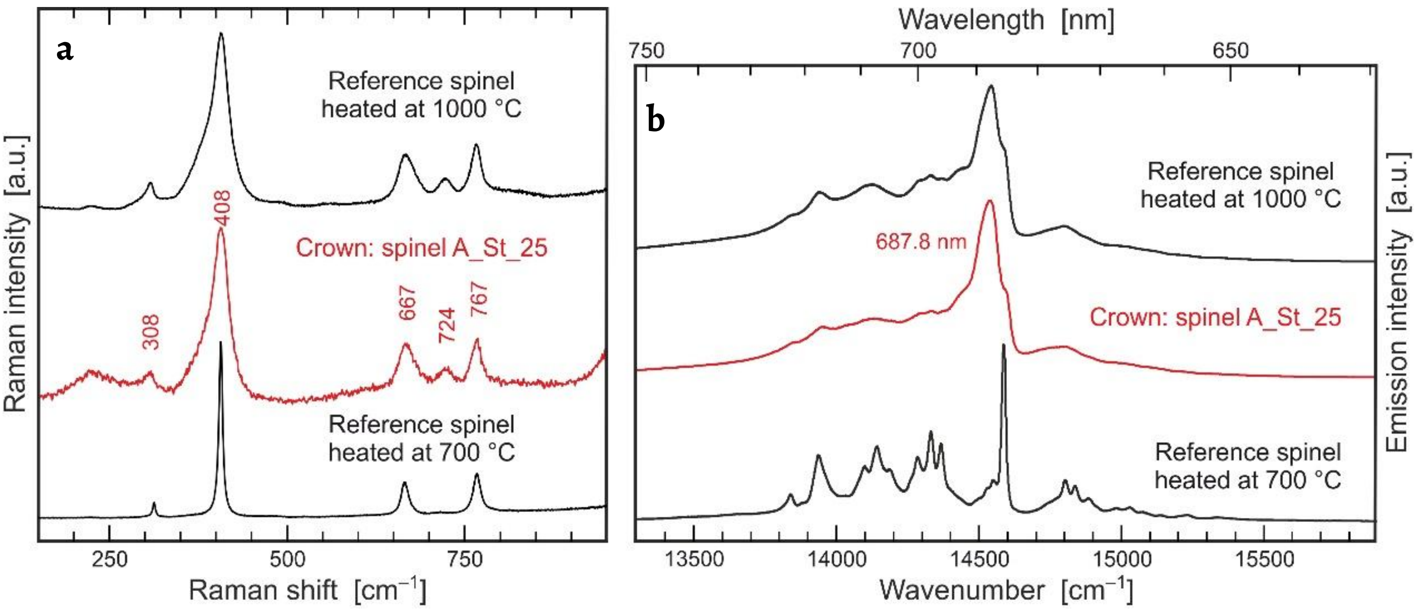


**Figure 6.** Spectra of the largest gemstone in the crown (red spinel A_St_25; see Figure 1): *a)* Raman and *b)* laser-induced PL. Spectra reveal cation disorder that is known to occur only after heating at elevated temperatures of about 1000 °C. Reference spectra of a heated, gem-quality spinel from Sri Lanka are shown for comparison (note that spectra obtained after heating at 700 °C still correspond to that of the unheated stone). Spectra are adjusted to the same height and plotted with vertical offset for clarity [28].

Nevertheless, an attempt was made to gain insights into potential geological/geographic origin of the crowns´ gemstones by comparing analysed data with verified reference materials and published data with known provenance. For example, the attribution of the 50 garnets to six distinct clusters identified by Raman analysis was confirmed by chemical analyses, allowing for a more detailed characterization of the stones. Some of these clusters can be attributed to known geographic sources. Many garnets in the crown are thus from South Asia (Sri Lanka, India), and some originate from deposits in Europe (Portugal) or Africa (Nigeria). All these garnet types and their deposits – except for the later added hessonite (E_St_16) – have been known and mined since in Antiquity. Bohemian garnets are notably not present in the crown, although they were in use since Late Antiquity [33-35].

As previously noted, determining the geographical origin of sapphire is especially challenging. UV-Vis-NIR was performed to distinguish between metamorphic and basalt related origins [31, 36]. It was found that all large sapphires on the circlet and front cross are of metamorphic origin (e.g., Sri Lanka, Myanmar, East Africa), while six small stones in bezel settings on the cross are derived from basaltic volcanic fields (e.g., France, Thailand-Cambodia). Considering the microscopic inclusion patterns, it is most likely that all of the metamorphic sapphires originated from Sri Lanka, as frequently proposed in the literature on the crown [4, 9]. Taking into account the historic context, France appears to be the most plausible source for the six basalt related sapphires.

Archaeological evidence and literary sources [37] suggest that the emerald deposits of Gebel Zabara and Wadi Sikait in Egypt were actively mined during the period when the crown was probably created. The gemmological characteristics of 18 out of 20 emeralds in the crown are consistent with this source. However, two emeralds exhibit different chemical and spectroscopic properties. One of these even containing large carbonate inclusions identified through Raman spectroscopy as magnesite and dolomite. Such inclusions are absent in Egyptian emeralds but suggest an origin from the Swat Valley in Pakistan [38].

**Pearls**

Microscopic examination of the Imperial Crown confirmed that all pearls were nacreous, identifiable through the terraced-like structure of the nacre, composed primarily of aragonite calcium carbonate crystals (Figure 7) [39]. Some of them were identified as blister pearls – i.e. pearls partially attached to the shell during formation. No replacement materials commonly used in other medieval objects, such as glass, wax, bone, or "mother-of-pearl" (nacre cut from the shell) were detected. Considering the historical context, all pearls must be natural, as the production of well-shaped cultured pearls began only in the early twentieth century, when the crown was already stored in a museal environment. The microscopic analysis also included detailed documentation of each pearl's shape, size, colour, and lustre, as well as drill hole characteristics, which varied in diameter and traces of processing.

Natural pearls can be classified as either freshwater or saltwater based on their chemical composition, primarily by comparing the concentrations of strontium and manganese – i.e. higher strontium and low manganese levels indicate saltwater origins [40]. The analysis of the crown's pearls was conducted using μ-XRF, though some of the smaller pearls were beyond the instruments reach, resulting in gaps in the dataset. Results indicate that most pearls are saltwater in origin (147), with a smaller number of freshwater pearls (69) distributed across all parts of the crown. Even the 858 small, irregularly shaped pearls used for the pearl string on the arch appear to be of saltwater origin (measurements were taken at 13 pre-selected reference points).

It is not possible to determine the exact geographic origin of pearls based on their chemical composition. However, historic sources and trade patterns suggest that the saltwater pearls most likely originated from the Persian Gulf region [41-42], though a source from the Red Sea and Southern Asia, cannot be completely ruled out. The natural freshwater pearls could be sourced from European rivers [42]. Notably, some of the saltwater pearls on the front and back plates – likely dating back to the original manufacturing period, as indicated by their precise fit in the settings – have a diameter of approximately 12 mm, significantly larger than those on comparable medieval objects. Pearls of this size only became more common in Europe after the discovery of the Americas and were for sure regarded as extremely rare and valuable before that time [42, pp. 23-32].

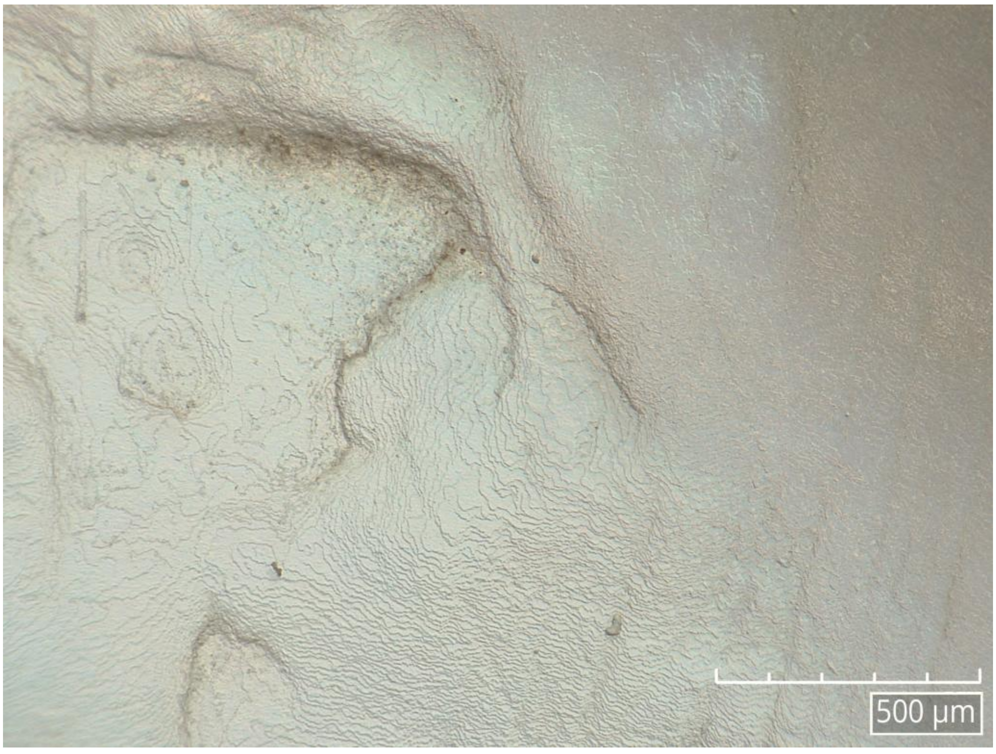


**Figure 7.** Pearl (A_Pe_31) surface showing a terrace-like structure of nacre (photo: Herbert Reitschuler, KHM-Museumsverband).

**Niello**

Archaeometric interest in niello has advanced since the 1950s, though it remains a niche area, still within the realm of fundamental research. Topics such as historical sources including recipes, applied techniques, and chemical compositions have been taken into focus, yet only a limited number of preserved objects have been examined in detail [11-13, 43-44]. Literature distinguishes three primary niello types by composition: silver sulfide, silver-copper sulfide, and silver-copper-lead sulfide, excluding rare forms containing zinc or gold [13]. Analyses suggest – based on significant variations in chemical composition, occasionally even within a single object – that niello was not a commercially traded material like glass. Instead, it is believed that niello was locally prepared by goldsmiths using available raw materials [44].

The niello on the Imperial Crown was identified via μ-XRF as a silver-copper-lead containing type. The established assumption that lead was not introduced into niello techniques until the eleventh century has been challenged by a few early finds [44]. Nevertheless, a general trend indicates that this lead-based niello type became a common technological advancement in the High Middle Ages, as the addition of lead lowered the melting point which enabled finer motifs through complete liquefaction during processing [11]. Consequently, in the attempt of dating the niello plaque of the front cross only a tendency toward production in the eleventh-twelfth centuries can be suggested.

However, comparative investigations are considered more effective in providing profound insights. Therefore, the Imperial Cross of Conrad II, exhibited alongside the crown in the Imperial Treasury, was also analysed. The absence of lead in the niello composition of the Imperial Cross refutes a material-based connection between the two objects despite the stylistic similarity.

**Enamel**

Microscopic examination – especially the few areas where the enamel layer is missing – provided greater insight into the applied cloisonné technique (Figure 8). Detailed information on manufacturing-related characteristics are omitted here as this article focuses on material-analytical aspects. As mentioned above, coloured and opacified glass consists of a complex mixture of different raw materials. Insights into the chemical composition can provide an identification of the glass types used for the manufacturing of the enamel plaques and a possible interpretation considering the state of research.

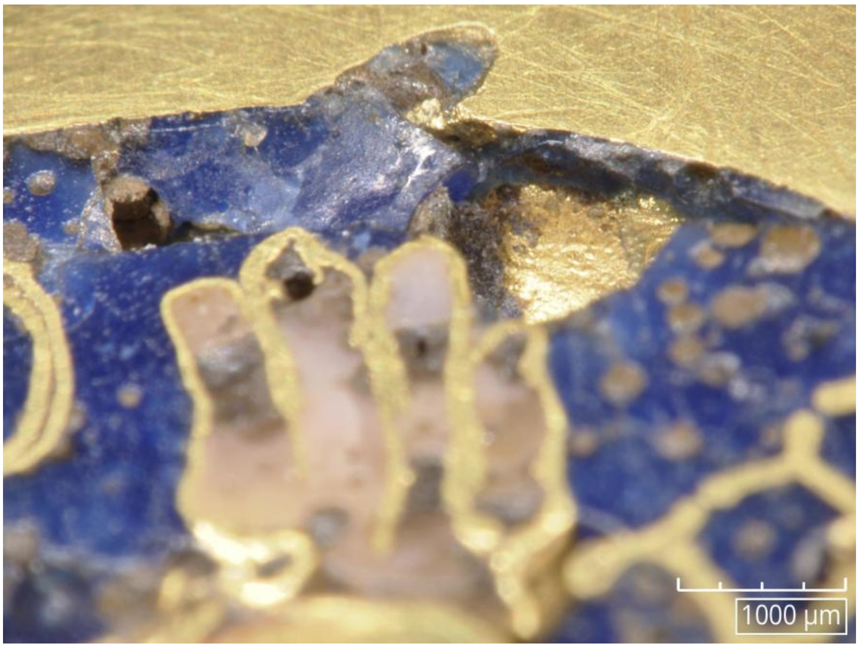


**Figure 8.** Solomon plaque (plate B), insight into the recess of the enamel recipient trough a damage in the enamel layer (photo: Herbert Reitschuler, KHM-Museumsverband).

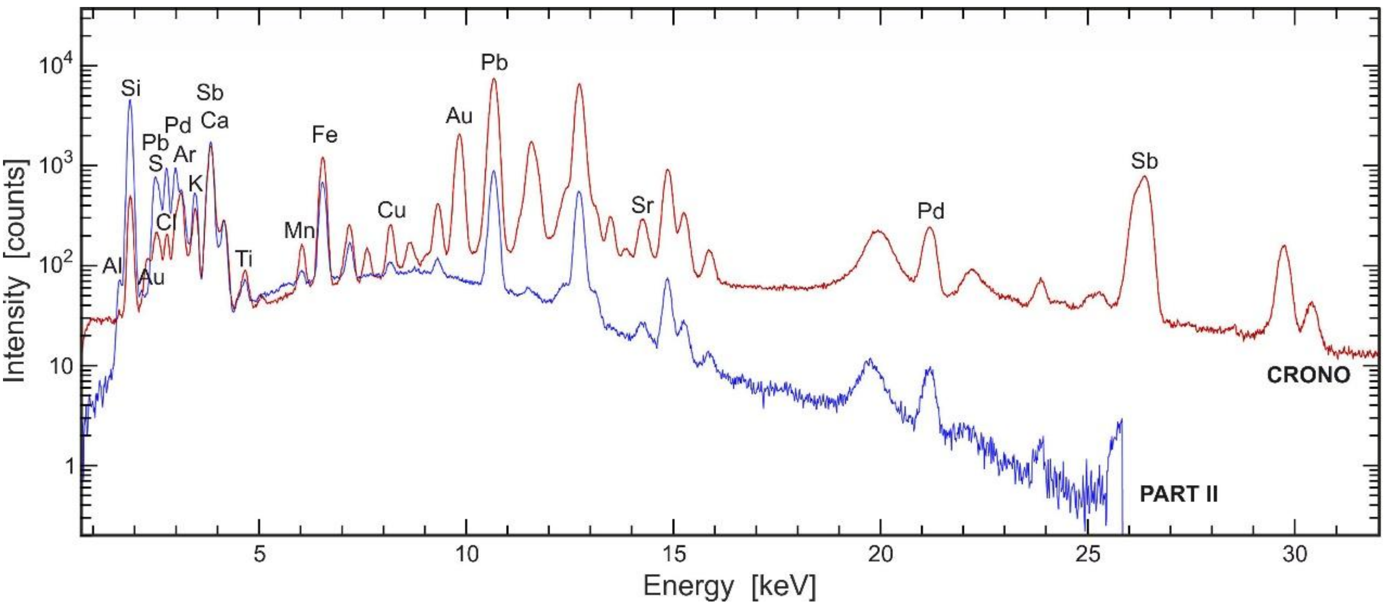


**Figure 9**. Comparison of two XRF spectra of the opaque yellow enamel on plaque D, recorded with the PART II (blue) and the CRONO (red) System (image: KHM-Museumsverband).

As mentioned in the instrumentation section there are some limitations bound to the applied methods. For example, the detection of different chemical elements is affected not only by the instrument used, but also by the measurement conditions. This makes it difficult to compare published data. Especially the detection of light elements like sodium is usually not possible with XRF performed in air. Moreover, XRF is a surface-sensitive method and, therefore, strongly affected by degradation processes. Nevertheless, under special considerations a semi-quantitative evaluation of the chemical composition is possible, but it is essential to measure glass standards of known composition under the same measurement conditions in order to obtain approximate quantification results. [45]. The example in Figure 9 illustrates how different the results can be using two XRF-devices PART II (μ-XRF) and CRONO (XRF) at the same measuring point (opaque yellow enamel). This demonstrates that the spectra of the two methods are not directly comparable; however, since the information was obtained from the same measurement point, they can be interpreted in a complementary way. For instance, the presence of antimony is apparent only in the higher-energy range covered by CRONO, whereas the μ-XRF instrument is optimized for detecting lighter elements. Briefly summarized, the results indicate that the Imperial Crown's enamels include various types of soda-lime glass, along with two wood-ash glasses (red and black).

Complementary to the XRF analyses, Raman spectroscopy was also performed for more insight into the composition. Because glass is a weak Raman scatterer, the characteristic bands of its individual components are often masked by strong background noise, luminescence, and the presence of multiple constituents and impurities, making them difficult to resolve. These limitations are especially pronounced when using mobile instruments, as in this study. Thus, reliable information from Raman spectra is often limited to identifying opacifying agents and, in some cases, detecting colouring additives. [17, 46] Using this method, opacifiers typical of first-millennium glass were identified, including calcium antimonate ($Ca_2Sb_2O_7$ and $CaSb_2O_6$) in most opaque colours and lead antimonate ($Pb_2Sb_2O_7$) in the yellow enamel.

FORS generates colour-specific spectra based on optical properties, providing objective information on comparability and, in some cases, identifying colouring additives and their oxidation levels [47]. For instance, FORS confirmed that the red enamel's colour and opacity were achieved through colloidal copper what equals also the XRF results of low copper and low lead concentrations typical for this colorant [48].


**Figure 10**. Detail of the Ezechias plaque (plate F) under different light conditions (image width 3.6 cm): *a)* visible (light colour 5000K); *b)* UV (254 nm); *c)* UV (365 nm) illumination (photo: Andreas Uldrich, KHM-Museumsverband).

Multiband imaging was also applied to document the distinctive features of the various enamel colours. The enamel exhibits specific luminescence in UV light; for example, the white enamel appears pink at 365 nm illumination, while the yellow enamelled areas appear bright blue at 254 nm being in accordance with the luminescence of the pigment Naples yellow (lead antimonate) (Figure 10). Unfortunately, there are hardly any published studies on the luminescence phenomena of coloured and opaque glass, which significantly limits the interpretation of the observed effects. UV light also revealed various organic residues on the surface and within recesses or cracks, which may result from contamination over the crown's history or indicate past restoration interventions.

Although the evaluation of the enamel analyses of the crown and the comparative objects has not been fully completed yet, preliminary results are consistent with the state of knowledge about medieval glassmaking published in literature. Combining multiple glass types within a single object is not uncommon and reflects a well-established tradition of glass recycling [49]. The frequent reuse and remelting of different glass types – sometimes in combinations – adds an extra layer of complexity to the interpretation of chemical compositions. The presence of red wood-ash glass was also detected on some German comparative objects, the earliest of which is dated to the late tenth century (e.g. Mathilden-Otto Cross, Treasury Essen). These results correct the current state of research, according to which the earliest known enamels made from red wood-ash glass dates from the twelfth century [50]. The presence of black wood-ash glass on the crown plaques, with an as yet unidentified colouring agent – presumably resulting from the presence of a sulfidic compound – is even more distinctive. For a further refinement and interpretation, the data will be processed in various statistical approaches such as binary element plots or principal component analysis (PCA).

## Further research

Upon completion of the CROWN project, the collected data will offer numerous opportunities for further research. Future scientific publications will address specific topics, such as those specifically about gemstones already published in the gemmological studies [28, 51]. A comprehensive final publication will follow in 2027 aiming to address, explain and contextualize the project's central research questions and to synthesize the various findings. Furthermore, a user-friendly, open-access presentation of the data will be developed. All these forms of dissemination will provide a robust, evidence-based foundation for future studies on the crown's provenance, use and history, as well as for further comparative studies with other early medieval goldsmithing objects.

## Conclusions

A detailed examination of the materials used for the Imperial Crown revealed that existing knowledge often relies on probabilistic constructs which become increasingly complex the deeper the investigation. Material analyses allow us to occasionally infer how the crown was manufactured and how it has changed over time. At the same time, however, the complexity of the topic is also revealed, leading to more refined research questions. Using state-of-the-art techniques and moving away from centuries-old hypotheses has established a foundation of well-documented and reliable data.

Careful interpretation of the collected data has already revealed new insights into the potential provenance of the materials used in the Imperial Crown. For instance, many of the gemstones appear to be reused, some of them traceable as far back as Antiquity. A combined evaluation of inclusion patterns, spectral signatures and chemical compositions suggests that most of the gemstones originate from Asia, while all but two emeralds most probably originate from Egypt. The majority of the set pearls exhibit features indicative of a saltwater origin. The analysis of the glass used in the four enamel plaques proved particularly complex. Optical and chemical analyses suggest that the enamels reflect medieval recycling practices, having characteristics associated with Roman and Islamic glass traditions. Certain results, such as the red and black wood-ash glasses, are particularly striking due to the absence of published data in a comparable context. Consequently, comparative analysis with well-dated objects serves as the most reliable basis for interpretation.

When investigating the provenance of an object, it is essential to acknowledge the limitations of optical and analytical methods. In the case of the Imperial Crown, one major challenge is the scarcity of securely dated and geographically localized medieval reference objects. Difficulties in attributing material origins often arise from overlapping characteristics between different sources, as well as from natural or production-related variations in appearance and chemical composition within the same material. Additionally, the historical recycling of materials can result in a blending of properties associated with different provenances, further complicating interpretation. As the analysis of valuable objects usually requires non-destructive methods, one must also accept their limitations, such as reduced sensitivity, which sometimes results in limited outcomes. Therefore, a comprehensive analysis of chemical compositions including trace elements, or even more advanced methods that could provide further insights into the material's nature, such as identifying specific isotopes, remain unattainable. Additionally, degradation effects caused by environmental exposure, especially prevalent in glass, can impact both optical appearance and chemical composition. The comparability of results of energy-dispersive methods such as XRF also requires scrutiny, as differences in instrumentations, measurement parameters, and data evaluation protocols can lead to incongruities. Awareness of these limitations is essential for both planning methodological approaches and interpreting results effectively.

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