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## **Salt efflorescence on leather objects from the Vasa ship**

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Citation for the original published paper (version of record):

Sandström, J., Berg, H., Öhrström, L. et al (2023). Salt efflorescence on leather objects from the Vasa ship. Working Towards a Sustainable Past. ICOM-CC 20th Triennial Conference Preprint

N.B. When citing this work, cite the original published paper.

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## Keywords

waterlogged leather, PEG-treated, salt efflorescence, analysis, iron, sulfur

## Abstract

Deposits of salts on the wood of the *Vasa* ship were previously identified as iron- and sulfur-containing salts, such as pyrite ( $\text{FeS}_2$ ), gypsum ( $\text{Ca}(\text{SO}_4) \cdot 2\text{H}_2\text{O}$ ), jarosite ( $\text{KFe}_3(\text{SO}_4)_2(\text{OH})_6$ ) and rozenite ( $\text{Fe}(\text{SO}_4)(\text{H}_2\text{O})_4$ ). A visually similar salt efflorescence is present on the leather objects found on the ship. A survey of the *Vasa* Museum's leather collection indicated that the salt deposits are negatively impacting the stability of those objects. The similarity of the salts found on the leather objects to those on the ship was therefore investigated. The results will inform further studies on the impact of the salts and enable the development of strategies for the preservation of the collection.

## INTRODUCTION

The Swedish warship *Vasa* sank in Stockholm harbour in 1628. She was raised in 1961, excavated and conserved, and is displayed in the *Vasa* Museum in Stockholm. A large collection of leather objects was found on board, mostly personal belongings such as shoes and gloves, but also book covers, pump packings and others (Hocker 2018, 76). The objects excavated from the ship were conserved in a facility at Beckholmen, in Stockholm, continuously from 1961 to 1996.

During an inventory of the leather collection in 2017, salt outbreaks were noticed on some of the objects. A 2018 documentation of these salt outbreaks showed that, out of roughly 600 leather objects, over 100 were visibly affected. Those objects are partially or completely brittle, hard, fragile and often have a disturbing odour. The salt outbreaks are seen as coloured deposits, commonly red, yellow, white or grey, and often glitter. Salt outbreaks on wooden objects from the *Vasa* and on the ship itself have been well documented and researched, and were found to consist of various compounds of iron and sulfur (Hocker 2018, 105). While the salt outbreaks on the leather objects are similar in appearance and odour, their chemical composition was unknown.

A research project to identify the composition of the salt outbreaks on the leather was initiated in 2019. Samples of salts and leather were analysed by X-ray fluorescence (XRF), scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDX), X-ray diffraction (XRD), and Fourier transform infrared (FTIR) and Raman spectroscopy. Samples from over 20 leather objects have been analysed, as well as samples from textile and wooden objects for reference and comparison. The focus of this article is the results of the analysis of four leather objects, as between them they show the full breadth of the characteristics of salt efflorescence.

## BACKGROUND

### Conservation of the leather finds

The leather objects from the *Vasa* have been conserved in different ways. Early attempts were made using lanolin, lanolin emulsions and polyethylene glycol (PEG) 600, but the results were deemed unsatisfactory (Bengtsson 1979). However, freeze-drying, recently emerged as a conservation treatment, proved to be more suitable for leather objects. Subsequently most of the

leather objects from the *Vasa* were conserved by freeze-drying, after immersion in a 25% solution of PEG 400 in water, with small amounts of boric acid and sodium borate added as a fungicide. The objects were then further treated with British museum leather dressing to improve their flexibility (Bengtsson 1979; Hocker 2018, 76–77).

### Storage of the leather finds

The conserved leather objects were initially stored in wooden crates in a room without climate control in the Beckholmen conservation facilities. In 1996, they were moved to a new storage site under the current Vasa Museum, where they were wrapped in acid-free tissue paper and placed in perforated zip-lock bags (Olsen, pers. comm., 2021). During an inventory carried out in 2017, the objects were transferred from zip-lock bags to acid-free cardboard boxes. Since 2004, when a new and improved climate control system was implemented in the museum and storage areas, the conditions where the objects are currently stored consist of a stable climate at 55% RH  $\pm$  4% and 18°C  $\pm$  1.5°C (Hocker 2018, 123).

### Documentation of the leather finds

It has been difficult to find documentation of individual leather objects after their excavation. They do not appear to have been individually photographed or documented during conservation, and the earliest descriptions are from 1996. Therefore, when the salt outbreaks first occurred is unknown. However, some objects were photographed in 1996, and again in 2017 and 2021, so in some cases the changes that may have developed between 1996 and 2021 can be identified. There are several entries in the museum's objects database that describe some objects as consisting of a certain number of pieces in 1996, and then of a much larger number of pieces in 2017, which indicates that these objects had become increasingly fragile and fragmented in storage.

### Salts on the wood of the *Vasa*

Salt deposits on the wood of the *Vasa* were previously identified as iron- and sulfur-containing salts, such as pyrite (FeS<sub>2</sub>), gypsum (Ca(SO<sub>4</sub>)<sub>2</sub>·2H<sub>2</sub>O), jarosite (KFe<sub>3</sub>(SO<sub>4</sub>)<sub>2</sub>(OH)<sub>6</sub>) and rozenite (Fe(SO<sub>4</sub>)(H<sub>2</sub>O)<sub>4</sub>). Pure sulfur has also been observed. The pyrite was partially converted to sulfuric acid after the ship was salvaged and exposed to oxygen. The areas where salts appear are often very acidic, sometimes as low as pH 1, and sometimes cause the wooden surface to flake, with further breakdown of the wood (Hocker 2018, 105). The iron and sulfur reflect the time the ship spent on the seafloor. Iron entered the wood and other materials through diffusion from corroding iron bolts and objects on the ship. Sulfur came from the surrounding polluted water and through bacterial activity (Hocker 2018, 105–109). The stable conditions of the above-described climate system in the museum contribute to reducing acid development, by minimising the movement of moisture through the wood (Hocker 2018, 123). For the ship, this was the only practical solution to prevent salt and acid development, but re-conservation options are being researched for the smaller wooden objects, including the use of high-performance iron chelators to extract the



Figure 1. Object 06684. Photo: Tony Andersson

iron and thereby remove one of the components causing the salt outbreaks (Almkvist et al. 2013).

## MATERIALS AND METHODS

### Samples

Samples were collected from four leather objects, all of which were already fragmented, sometimes into as many as 100 pieces. For this study one to three such fragments were taken as samples from each object. Depending on the type of intended analysis, the samples were further processed, for example, by removing salt crystals.

### Description of the four leather objects

Object 06684 consists of several parts and is believed to have been a bag or purse (Figure 1). Several of its smaller fragments are covered in orange, grey and glittering salt efflorescence. The object has a strong odour. From this object, one sample was taken (#66080). The sample was found to have three different colours (beige, orange, brown), each of which was analysed, as shown in Table 1. The size of the sample was 1 × 1 cm.

Object 10142 comprises parts of a shoe, found near the remains of a person (Figure 2). Several of the fragments are covered in orange, glittering salt efflorescence. A 5 × 5 mm sample (#66082) was taken for the analysis.

Object 14225 consists of ~100 leather fragments and is probably a shoe (Figure 3). Several of the fragments are covered in orange and yellow glittering salt efflorescence. The object is described as having consisted of 70 pieces in 1996, but ~100 pieces in 2018, indicating further fragmentation. From this object, two samples were taken for analysis (#66102 and #66109,

Table 1. Objects and the corresponding samples: a summary of the results of the different analyses

Object	Sample	FTIR	Raman	pH	EDS (C, O, S, Fe in every area)	XRF (S, Fe in every area)	XRD
06684	66080 beige	FeCO <sub>3</sub>	FeCO <sub>3</sub> ; FeOOH; sulfur	n.a.	Al, Na, K, Si, Mg, Ca	Ca, K, Zn, Cu, Ti, Mn, Si, As (?), Pb (?)	Jarosite
	66080 orange	FeCO <sub>3</sub>	Sulfur; FeCO <sub>3</sub> ; +FeOOH(?)				Jarosite
	66080 brown	Protein	/				/
10142	66082	FeCO <sub>3</sub>	Sulfur	4.9	only C, O, S, Fe	Ca, K, Zn, Cu, Ti, Si, As (?), Pb (?)	N.a.
14225	66102 yellow	Sulfate (jarosite, rozenite); carbonate?	Sulfur; sulfate; Jarosite	3.1	Al	n.a.	Rozenite
	66102 black	Sulfate?	/		Al		Rozenite
	66102 white	Sulfate (jarosite, rozenite); Ferrous oxalate; Carbonate?	Jarosite		Al, (Na, Si)		Rozenite
	66109	n.a.	N.a.		n.a.		n.a.
18297	65999	Sulfate	Sulfur	2.6	Ca, Zn	n.a.	n.a.
	66000	Protein?	Sulfur	n.a.	Al, Si, K, Ca, Cu	n.a.	
	66077 black-yellow	Protein; FeCO <sub>3</sub>	/	3.4	Ca, Al, Si	Ca, Zn	
	66077 black	FeCO <sub>3</sub>					
	66077 black-red	Protein					
66077 transparent	Microcrystalline Wax						

Key: n.a.: not analysed; /: analysed but no identification; (?) indicates uncertainty in the analysis results



Figure 2. Object 10142. Photo: Tony Andersson



Figure 3. Object 14425. Photo: Tony Andersson



Figure 4. Object 18297. Photo: Tony Andersson

7 × 7 mm and >1 mm respectively). Sample #66102 was analysed according to its different colours, from black, to yellow and white (Table 1).

Object 18297 is also part of a shoe (Figure 4). The leather pieces are covered in a grey-white, glittering salt efflorescence and have a strong odour. In the museum database, the object is described as being soft, but all pieces are now hard, indicating a change in the leather. From this object, three samples, ranging from 1 × 1 mm to 5 × 5 mm, were taken for analysis (#65999, #66000, #66077). Sample #66077 has several colours (transparent, black-yellow, black-red and black), each of which was analysed (Table 1).

### Analytical methods

Among the many possible analytical methods that can be used to investigate leather objects, the methods selected in this study were chosen in order to characterise the salt crystals. The topography and visual appearance of the crystals were characterised by stereomicroscopy and SEM. The identity of their elements was determined using SEM-EDS and XRF, and information on their chemical composition using XRD together with FTIR and Raman spectroscopy. The pH of the samples was measured for comparison with that of the wood of the *Vasa*, because highly acidic deposits are known to cause degradation. The combination of methods provided a broader view of the salt efflorescence.

### Stereomicroscopy

A Leica Si9 stereomicroscope was used to image the samples and their salt crystals. Images were collected in reflected light, with the samples placed on glass slides against a white background.

### SEM-EDS

SEM images and EDS spectra were obtained from gold-coated samples (Quorum sputter) using a JEOL JSM-IT500LA with a tungsten filament in high vacuum, at 15 keV, 10 mm working distance and with a standard probe current of 55. The images were collected using a backscatter electron detector, and EDS spectra were measured with an SDD type detector. For semi-quantification of the elements in the differently coloured salts of sample #66102, between nine and twelve EDS spectra were collected from each area of interest.

### FTIR spectroscopy

Small sub-samples consisting of particles of the leather and salts no larger than 300 μm were compressed in a Specac diamond cell. The cell was then opened to measure transmission spectra using a Perkin Elmer Spotlight 200 FTIR microscope with a 100 × 100 μm aperture, a 4000–600 cm<sup>-1</sup> interval and obtaining 16 scans at 4 cm<sup>-1</sup> resolution. A clean area of the diamond window was used as the sample background.

### Raman spectroscopy

The sample in the diamond cell used for FTIR spectroscopy was also used for Raman spectroscopy. Spectra were collected on a BW Tek iRaman instrument coupled to a microscope. The laser wavelength was 785 nm

and the laser power, adjustable up to 495 mW at 100%, was in the range of 10–30%. The integration time was 2000 ms and the scan range 0–4000  $\text{cm}^{-1}$  in Raman shift. Dark spectra were collected using the same settings but with the laser shielded from the detector.

#### **XRF**

The samples were placed on a polypropylene background and analysed without further preparation. Single-point spectra were collected using a Bruker Artax 800 with a molybdenum X-ray tube at 50 kV, 600  $\mu\text{A}$  and 10 s live time. A polycapillary lens with a spot size of  $< 100 \mu\text{m}$  was used. The polypropylene spectrum served as the blank background spectrum.

#### **XRD**

Powder X-ray diffraction (PXRD) patterns were collected on a Bruker D8 Advance diffractometer equipped with a LynxEye detector using Mo  $K\alpha$  radiation ( $\lambda = 0.71073 \text{ \AA}$ ) at room temperature. X-rays were generated by a current flow of 40 mA and an accelerating voltage of 30 kV. The samples, without further preparation, were placed on a Si single-crystal sample holder and scanned over the  $2\theta$  range of  $4^\circ$  to  $130^\circ$  with a step interval of  $0.01^\circ$ . The PXRD patterns were generated from single-crystal X-ray data using the Mercury 3.3 program, which calculated PXRD patterns using the X-ray source also used experimentally.

PXRD data were also collected using a single-crystal X-ray diffractometer (Rigaku XtaLAB Synergy R, HyPix diffractometer) and Cu  $K\alpha$  radiation ( $\lambda = 1.54184 \text{ \AA}$ ). The samples were sub-divided under microscopy and single crystals were mounted on a nylon loop. Data were collected during a constant flow of nitrogen gas (at  $-173^\circ\text{C}$ ). PXRD data were then generated using CrysAlis Pro. Single-crystal data on selected crystals were obtained the same way, with CrysAlis Pro used to evaluate unit cells and space groups.

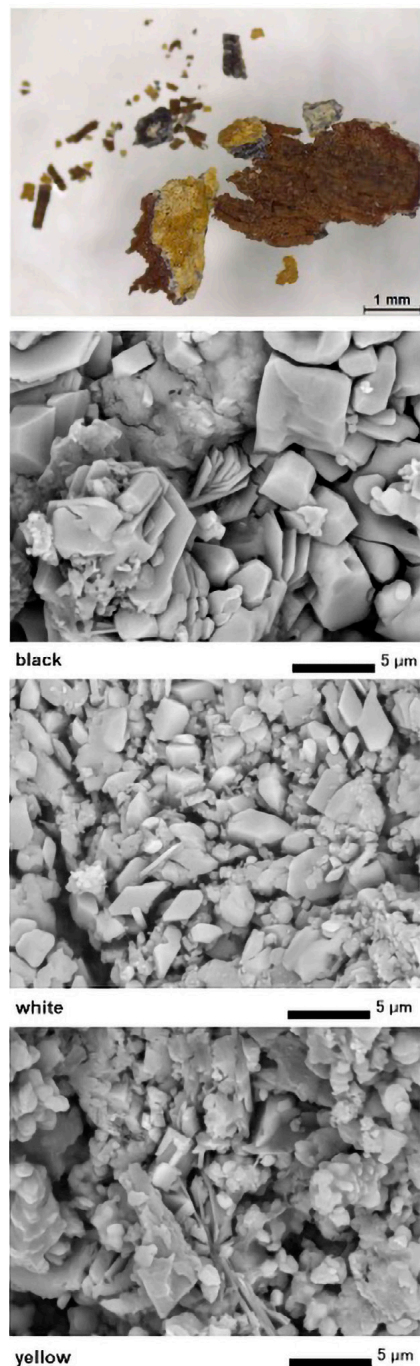
#### **pH measurements**

The samples were sub-divided and fragments weighing roughly 0.5–1 mg were immersed in deionised water and left for 24 hours. The volume of deionised water was adjusted considering the weight of leather as 5% of the total. The pH of the solution was then measured with a Horiba LAQUA twin pH meter, calibrated with a pH 7.0 standard buffer solution. The pH of the deionised water without leather was 5.9–6.0.

## **RESULTS AND DISCUSSION**

Descriptions of the four leather objects, their corresponding samples and the main analytical results are given in Table 1.

All leather samples were of acidic character, with a pH in the range of 2.6–4.9, which is somewhat consistent with the pH of leather tanned using traditional methods, which ranges from 3.5 to 8 depending on the tanning method (Stambolov 1969, 36), and the expectation that objects from the *Vasa* affected by salt outbreaks would be acidic. Measuring the pH was of great relevance considering that acid was produced during



**Figure 5.** Stereomicroscopy image (top) and SEM micrographs of sample #66102 showing salts from black, white and yellow parts of the leather

the processes associated with the salt outbreaks on the ship and wooden objects, as described above. Also, at a pH lower than 3 acid hydrolysis can initiate the breakdown of the collagen fibres in leather (Ibid. 37). Thus, the measurement of pH values below 3 may be an indication that acid is produced in the leather objects and may have a destructive effect on them.

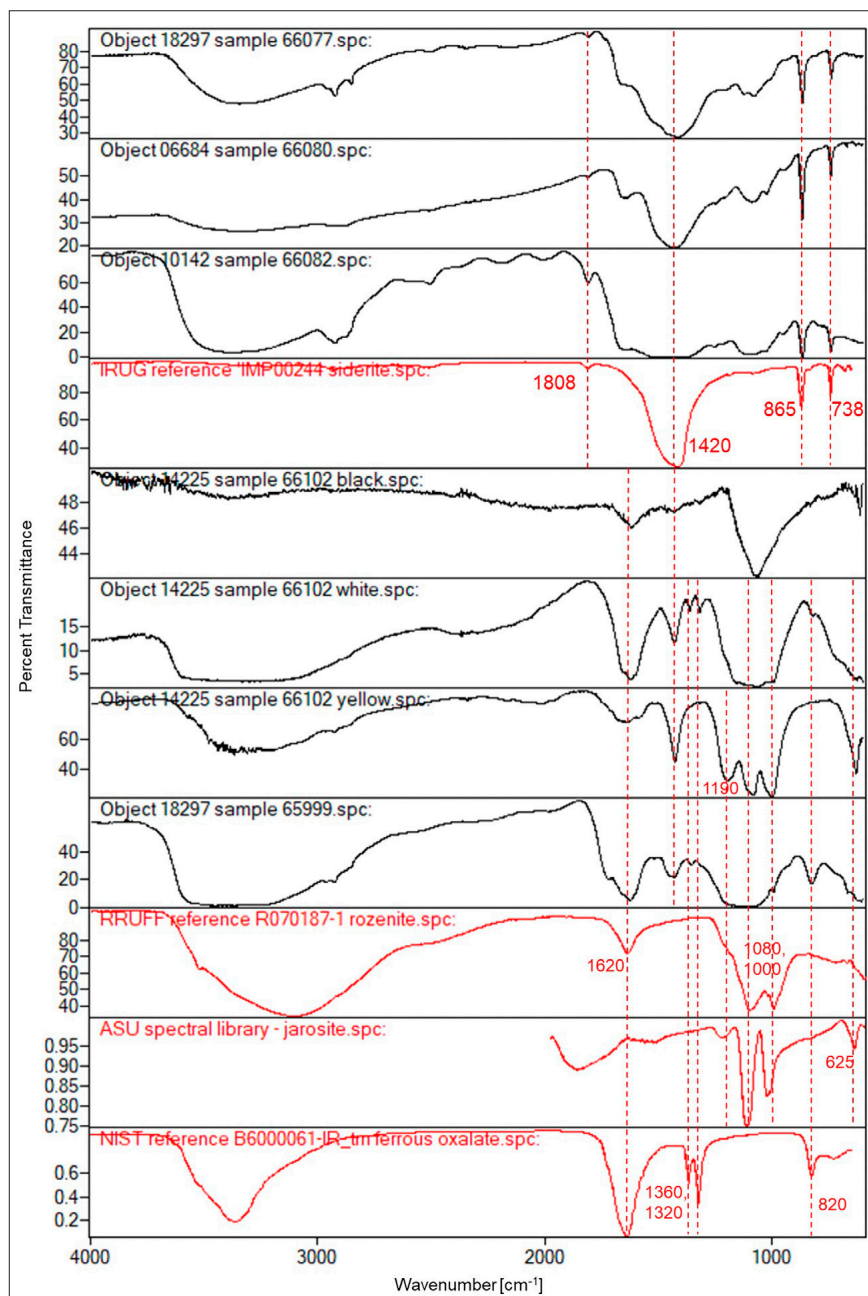
Stereomicroscopy and electron microscopy confirmed the various colours, crystal shapes and sizes of the salt efflorescence, as exemplified in Figure 5, which shows sample #66102. All of these properties were further investigated with the aim of finding the chemical differences that gave rise to the different colours, but none of the results provided a convincing explanation. The EDS analysis of nine to twelve different areas per colour in sample #66102 showed no significant differences with respect to sample colour. The dominant elements detected in every area of analysis were oxygen, at around 40–60 atomic %, and sulfur and iron, at 10–20 atomic %. A significant amount (usually over 20%) of carbon was detected in approximately half of all areas, while aluminium was mostly present at very low levels (below 1%).

The atomic ratio of O/S was in the range of 2–6 and that of S/Fe mostly around 1. These results indicated the presence of  $\text{FeSO}_4$  in the objects. Additional elements detected by EDS were calcium, sodium, potassium, silicon, magnesium, zinc and copper, usually at trace levels and not in every sample (Table 1).

In the XRF analysis, iron and sulfur were the dominant elements in every sample analysed (Table 1). Calcium and zinc were also found in every sample. Other elements detected were potassium, copper, titanium, manganese, silicon, arsenic and possibly lead. The sea area where the *Vasa* sank is polluted and the sources for the various elements found in the leather objects are indeterminable, but they may have stemmed from the bottom sediments or sand or from the pollutants.

In accordance with previous analyses of *Vasa* wood, iron sulfates were detected on the leather samples. Figure 6 shows the FTIR spectra for sulfates, with characteristic absorptions of  $1000\text{--}1200\text{ cm}^{-1}$  and  $500\text{--}700\text{ cm}^{-1}$ . The shape and position of the infrared absorption bands of the sulfate anion ( $\text{SO}_4^{2-}$ ) depend on the cation (e.g.  $\text{Fe}^{2+}$ ,  $\text{Ca}^{2+}$ ,  $\text{K}^+$ ) as well as the hydration of the salt (OH or  $\text{H}_2\text{O}$ ) (Lane 2007). Bands associated with the hydration of sulfate salts were detected at  $\sim 1620\text{ cm}^{-1}$  ( $\text{H}_2\text{O}$ ) and at various positions between  $1000$  and  $400\text{ cm}^{-1}$  (OH) (Lane 2007). Thus, the FTIR spectra suggested the presence of different types of sulfates. Both rozenite ( $\text{Fe}(\text{SO}_4)(\text{H}_2\text{O})_4$ ) and jarosite ( $\text{KFe}_3(\text{SO}_4)_2(\text{OH})_6$ ) were indicated by FTIR and Raman spectroscopy (Table 1 and Figure 6), and later confirmed by XRD (Figure 7 and Table 4).

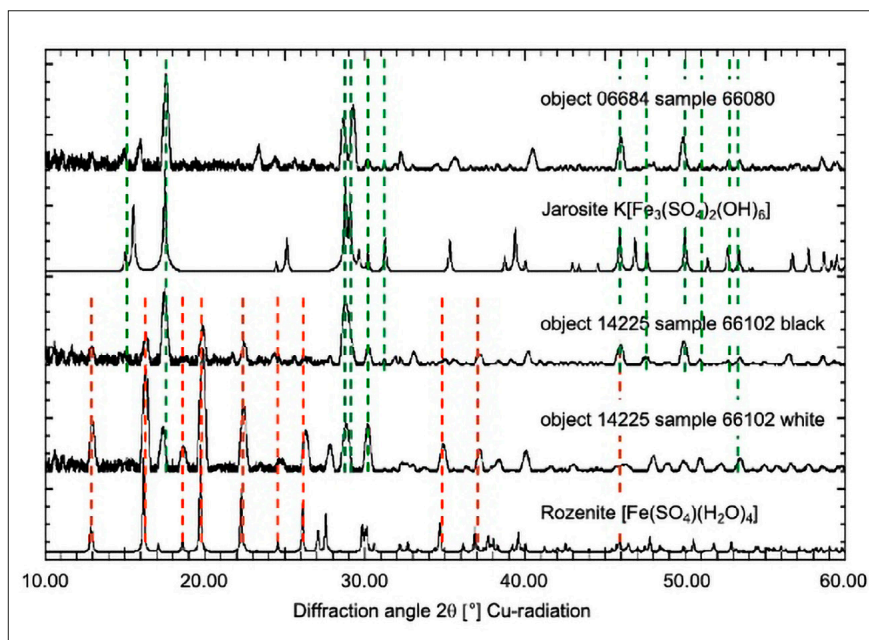
Several compounds other than sulfates were also identified, including iron oxide hydroxide ( $\text{FeOOH}$ ), identified by Raman spectroscopy, and pure sulfur, identified by Raman spectroscopy, XRD and SEM–EDS (Figure 8). The presence of ferrous oxalate ( $\text{C}_2\text{H}_4\text{FeO}_6$ ) in the white sample (#66102) was shown by FTIR, based on characteristic absorptions around  $1360\text{ cm}^{-1}$  and  $1320\text{ cm}^{-1}$  and a smaller peak near  $820\text{ cm}^{-1}$  (Figure 6). High levels



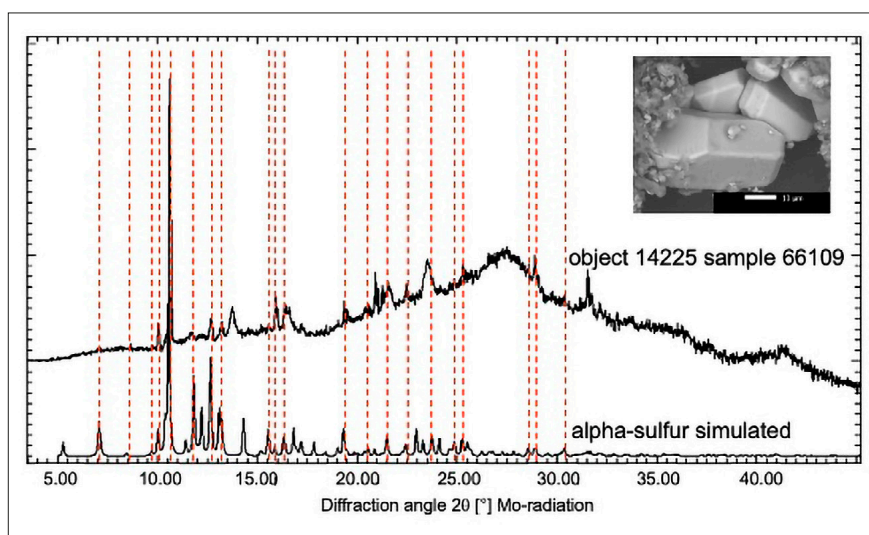
**Figure 6.** FTIR spectra of samples #66102 (yellow, black and white) and #65999 as well as the reference spectra of jarosite ASU, rozenite (RRUFF) and ferrous oxalate (NIST). Also shown are FTIR spectra of samples #66077, #66080 and #66082, showing carbonates, possibly including siderite (see the IRUG reference spectrum, Price et al. 2009)

of oxalic acid and oxalate in wood have previously been reported (Dedic et al. 2013).

The FTIR spectra also showed that samples #66077, #66080 and #66082 contained carbonates (Figure 6). Based on comparisons with the reference spectra, siderite ( $\text{FeCO}_3$ ), with characteristic sharp infrared absorption peaks at  $865\text{ cm}^{-1}$  and  $738\text{ cm}^{-1}$ , associated with the asymmetric bending of  $\text{CO}_3^{2-}$ , was identified. A broader band near  $1420\text{ cm}^{-1}$ , associated with the asymmetric stretching of  $\text{CO}_3^{2-}$ , characterised the spectra of samples #66102 and #65999, which otherwise contained mainly sulfate (Figure 6). It is unclear whether siderite is also present in those samples. Iron carbonates on Vasa wood have not been previously reported or described. However, their potential presence will have to be further investigated, as it could impact the pH measurements, by neutralising some of the acids in the



**Figure 7.** XRD patterns matching rozenite ( $\text{Fe}(\text{SO}_4)(\text{H}_2\text{O})_4$ ) (Bauer 1962) with object 14225, sample #66102 and jarosite  $\text{K}[\text{Fe}_3(\text{SO}_4)_2(\text{OH})_6]$  (Basciano and Peterson 2007) with object 06684, sample #66080. The powder patterns were simulated from single-crystal structures



**Figure 8.** XRD patterns matching elemental sulfur from object 14225, sample #66102 with a simulated powder pattern from alpha-sulfur (Rettig and Trotter 1987). The insert is a SEM image showing a sulfur crystal from the same sample

**Table 2.** XRD unit cell parameters obtained from single-crystal data collection on a crystal from object 14225, sample #66102 (yellow), compared to literature data on rozenite ( $\text{Fe}(\text{SO}_4)(\text{H}_2\text{O})_4$ ) (Bauer 1962)

Unit cell parameter	This study ( $-173^\circ\text{C}$ )	Bauer 1962 (room temp.)
$a$ (Å)	5.95208	5.97
$b$ (Å)	13.59677	13.64
$c$ (Å)	7.97717	7.97
$\alpha$ (°)	90	90
$\beta$ (°)	90.2416	90.26
$\gamma$ (°)	90	90
space group	$P2_1/n$	$P2_1/n$

leather material from the *Vasa*. The fact that carbonate was not detected by XRD suggests that it is present only in amorphous form, but its origin is unknown.

## CONCLUSION

All of the analytical methods used for this project detected high levels of iron and sulfur compounds in all samples, both on the surfaces and inside the leather. Most of the salts contained high levels of carbon and oxygen along with many other elements. The absence of chlorides is likely due to the fact that the water where the *Vasa* sank is brackish, such that the salt concentration is lower than that typical for seawater (Hocker 2018, 8); in addition, the objects were immersed and rinsed in freshwater prior to their conservation (Bengtsson 1979).

SEM-EDS, XRF, Raman and FTIR spectroscopy, and XRD identified the following compounds within the salts of the *Vasa* leather samples: jarosite, rozenite and possibly other sulfates, ferrous oxalate, iron oxide hydroxide, pure sulfur and siderite. The latter has not been previously reported in *Vasa* materials nor has the presence of carbonate in the leather samples, but they further demonstrate the complexity of the chemistry of the *Vasa* objects.

Several different types of salts were detected on the leather objects and their chemistry was shown to be similar to that of the salts found on *Vasa* wood. Assuming that the salts behave the same in the leather as they do in the wood, then it seems probable that they have already negatively impacted the leather and likely contributed to the fragmentation of some of the objects.

Identification of the salts has helped to evaluate the need to examine possible future conservation steps, sustainable storage in terms of temperature and relative humidity, and handling. However, further investigation is necessary to understand the specific effects of the different salts on the leather objects. Whether our findings reflect the effects of previous conservation treatments of the *Vasa* and her objects was not determined in this study but also needs to be investigated. Although the objects are now kept in a stable storage environment, it is currently uncertain whether the salt efflorescence is an ongoing process that continues to pose a danger to the so-far-unaffected parts of the collection. Furthermore, tests need to be carried out to examine what can be done to improve the condition of objects already affected by salt outbreaks.

## ACKNOWLEDGEMENTS

The authors wish to thank Tom Sandström, from the Swedish National Heritage Board, for help with the FTIR and XRF data collection, and Chalmers Materials Analysis Laboratory and Dr Michal Strach for access to the X-ray diffraction instruments.

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**To cite this article:**

Sandström, J., H. Berg, M. Hacke, L. Öhrström, and F.M. Amombo Noa. 2023. Salt efflorescence on leather objects from the *Vasa* ship. In *Working Towards a Sustainable Past. ICOM-CC 20th Triennial Conference Preprints, Valencia, 18–22 September 2023*, ed. J. Bridgland. Paris: International Council of Museums.