

## RESEARCH ARTICLE

# Detecting Chloride Contamination of Objects and Buildings – Evaluating a New Testing Process

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Soluble salts play a key factor in damage to a variety of materials, including stone, ceramics and metals. Particularly, salt contamination can lead to weakening of porous materials through salt crystallisation events, and increases the rate of metal corrosion. Over time, this results in physical damage to affected objects and buildings. It is therefore important to be able to monitor the salt content of materials, in order to understand levels of salt contamination and the potential for damage to occur.

This research discusses the further development of the testing method for surface chlorides originally proposed by Piechota and Drake Piechota (2016) in their article “A simple survey kit for chloride detection on cuneiform tablets and other collections”. It introduces new and revised steps into the original protocol in order to make the achieved results semi-quantifiable, as well as identifying the limits of detection of the test kit. A comparison to alternative testing methods showed that comparable results were achievable using this methodology. The revised methodology was tested for efficacy on a range of salt contaminated objects, as well as on samples from buildings.

**Keywords:** Chlorides; soluble salts; conservation; historic buildings; porous ceramic; limestone

## 1. Introduction

Soluble salt damage to materials is an ongoing conservation issue, with the potential to affect heritage buildings, statuary, and other historic objects. Understanding the types of salts present on the surface of materials is an important first step in the process of conservation treatment, as it allows conservators to trace back the potential sources for the salts, and to understand how the contamination may be resolved. Furthermore, being able to quantify the levels of the different salts can aid in the decision making process for treatments. This research discusses the evaluation of a novel method for detecting the presence of surface chlorides, one of the principal types of salts involved in damage to historic materials.

The main types of salt contamination found in objects and buildings are those from chlorides, sulphates, nitrates, phosphates and carbonates (Winkler, 1997, pp. 156–158). Carbonates are known as insoluble salts, forming on the surface of materials hard crusts that do not re-dissolve in the presence of moisture. Insoluble carbonates may originate from external sources, or from the material itself (Lehmann, 1971). Soluble salts, such as chlorides, may form salt crystals both on (efflorescence) and below (sub-efflorescence) the surface of materials; testing of surface salts is a good indicator of the salts present throughout a material, and provides an indicator of the possibility for sub-efflorescence. While unsightly,

efflorescence tends to be less damaging than sub-efflorescence, where salts crystallise and expand within the pores of materials such as low fired ceramics and porous stone (Sawdy *et al*, 2008, p. 2). This expansion generates stress on the material, which can cause the stone or ceramic to become weakened, and may eventually cause the material to disintegrate (Lombardo *et al*, 2004; Brady *et al*, 2006; Doehne & Price, 2010, p. 15). Salts will often undergo repeated cycles of dissolution and crystallisation, as relative humidity and material moisture levels change, thus increasing the stress and potential damage to the material (Lombardo *et al*, 2004).

Salts can be transferred to objects and buildings in many ways, with both outdoor and indoor sources. Outdoor sources include air pollution, where atmospheric pollutants such as NO<sub>x</sub> or SO<sub>2</sub> may react to form nitrate or sulphate salts within materials (Doehne & Price, 2010, p. 15). Nitrate salts can also be the product of an atmospheric reaction between nitric acid and ammonia, product which then precipitates in dust or in rainfall (Sharma, 2005, pp. 45–47). Nitrates may also have a soil based origin, as they are readily soluble in ground moisture. Other salts, including chlorides, may contaminate the material through rainfall, through de-icing salts from roads, from sea spray or from within the material itself (Charola, 2000). In the case of archaeological materials, salt contamination can come from the burial environment, such as the sea or the ground water moving through soil. Often the objects may become contaminated with salts in an anoxic or low oxygen environment; the lack of air

exposure, for instance while buried, prevents evaporation, meaning that the items start to effloresce once evaporation can take place, after removal from water or ground (Brady *et al.*, 2006). Indoor sources of salts can include off-gassing materials from display and storage environments (Ellis *et al.*, 2007, p. 62).

The most commonly used method for identification of surface salts is spot testing. Spot tests are useful techniques for conservators because of their low cost, speed, and minimal need for equipment. Other analytical methods are available to investigate and quantify chloride levels, but they involve more investment in equipment, and therefore are considerably more costly. For example, more precise quantification of soluble salt levels throughout a material can be obtained by ion exchange chromatography either from core sampling of the stone or through salt extracted by poultices (Vicente & Vicente-Tavera, 2001; Dionísio *et al.*, 2013).

There are currently two main spot testing methods used. In both cases, a dry sample of the salt is removed from the surface of the object, and then dissolved in water. In the silver nitrate test a small amount of the silver solution reacts with free chloride ions, producing a cloudy white precipitate (Riss, 1993). Alternatively, the salts may be tested using commercially available chloride test strips, which give basic quantification, such as those available from Quantofix®. Both methods have some limitations. The standard silver nitrate test cannot easily be carried out on site. The presence of precipitate shows a positive result, but is not viewed as a quantifiable test, whereas the test strips allow some level of quantification through a colour chart system. Nevertheless they have a limited quantifiable range (0–>3000 mg/L for the standard test strips). Furthermore, when testing for surface chlorides on ceramics, Ellis *et al.* (2007) found that the Quantofix® indicator strips were insufficiently sensitive to the presence of the salts when compared to the use of the silver nitrate test. The authors suggest this may be due to the low concentration of salts being below the level detectable by the test strips, or to the method of removal of the salts (rolling swabs or using a poultice on the surface).

Other alternative options for testing not only look at the surface efflorescence, but analyse salts found throughout an object, and can be combined with the use of test strips or the silver nitrate method. Chloride contamination may for example be determined by soaking an object and testing the wash water. Although chloride test strips were ineffective in detecting surface salts levels in research by Ellis *et al.* (2007), when testing the wash water (and therefore determining overall chloride contamination) Odegaard *et al.* (2011) found that test strips were a successful method of semi-quantifying the chloride levels of the wash water during the desalination of ceramics. Analysing the salt content of poultices may also be used to indicate the salt content of materials, and to evaluate the success of poultice treatments in desalination (Bläuer Böhm, 2005). While these methods of wash water and poultice testing tend to be more quantifiable and reliable (in terms of positive or negative readings), they are not always suitable methods, either because of the fragility or because

of the size of the object (Ellis *et al.*, 2007). They also take some time to perform, due to the time needed to extract the salts from the item. This means that it is important to further develop quick testing methods for efflorescence of salts, to help overcome these difficulties.

A recent publication by Piechota and Drake Piechota (2016) described a revised method for the silver nitrate test for chloride efflorescence on cuneiform tablets. In their study, they used a smartphone light to give a stable light source for the test. A light source is not used in the standard method for testing with silver nitrate; where low levels of chlorides are present, particularly in lower light levels, it can be problematic to decide between a positive and a negative result. The additional light made it possible to detect levels of chlorides more consistently, cheaply, and quickly, and could be used on site. The authors suggested that their method may be more quantifiable, but did not expand on how this could be achieved. In addition, their paper focussed on the use of this test specifically for cuneiform tablets (clay), and did not investigate its potential for use in relation to other material types.

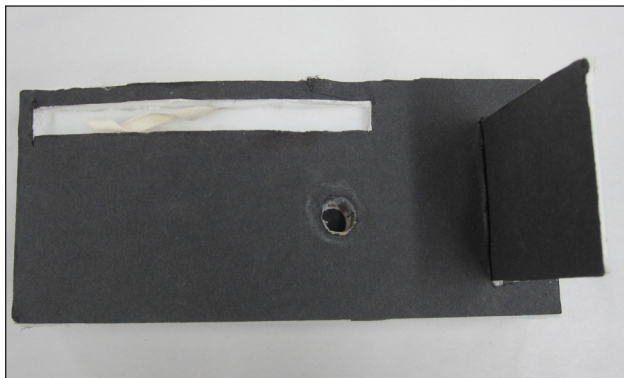
In the study presented in this paper, we revised the Piechota protocol, adding a novel step in order to quantify results, and identified the limits of detection for the kit. The results obtained using the revised protocol are compared to those of the standard silver nitrate test and of the use of test strips for chlorides, in order to establish the accuracy of the test. In order to demonstrate that the kit has wider applications to a range of different materials, surrogate objects were contaminated with salt: limestone, earthenware, pine wood and iron alloy were chosen. Earthenware, wood and iron alloy are typical of the material types found at archaeological sites or in marine environments, and are more likely to be contaminated with chlorides from those environments (e.g. Cronyn, 2003). While iron alloy is not a porous material, the presence of chlorides significantly increases the rate of its corrosion, and if untreated is likely to result in the total loss of the artefact. Limestone was also tested as it is a common building material, found in over 36,000 listed buildings in England alone (English Heritage, 2017), and is vulnerable to chloride contamination from a range of sources (Sowden, 1990, p. 122).

## 2. Methods

### 2.1. Construction of testing kit base

The chloride test kit was constructed according to the instructions from Piechota and Drake Piechota (2016), with a modification made to the phone slot using inserts made from strips of cut Ethafoam E220, in order for a variety of phone sizes to be utilised. For this modification, fabric tape was attached to the foam insert to allow it to be removed. These foam strips are shown in **Figure 1**. The phones used for testing in this paper were a Sony Xperia Z3 Compact smartphone and an Alcatel Pixi 4 (4") smartphone with a flashlight function. Lux levels from the phone flashlights were measured with an Elsec 765 Environmental Monitor, and shown to be approximately 11,500 lux at the sample measurement point.

Foam board was prepared in the following sizes:



**Figure 1:** The completed test kit base.

- Two pieces of black Plastazote polyethylene foam board (Kewell Converters Ltd), 0.3 cm × 9 cm × 20 cm
- One piece of black Plastazote foam board, 0.3 cm × 6 cm × 9 cm
- One piece of Ethafoam E220 (Kewell Converters Ltd), 2.5 cm × 9 cm × 20 cm

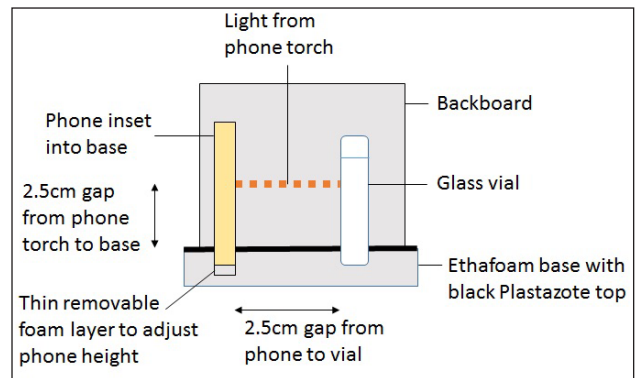
Using a hot melt glue gun (Bostik, supplied by Homebase), one of the two 20 cm black foam boards was attached onto the top of the Ethafoam plank (**Figure 1**). A slot wide enough to fit the smartphone was cut along the left side of the foam. The hole was backfilled with the cut foam, so that the light from the phone was approximately 2.5 cm above the base.

For this testing, 4 ml Wheaton Specimen glass vials with push fit plastic stoppers were used (product code ST5012); however, alternative glass vials could be considered – for alternative products, the diameter of the cut-out hole would need to be adjusted to fit the vial size. At right angles to the phone light a 1.6 cm diameter circle was removed, approximately 2.5 cm from the phone. The hole was backfilled using the cut-out circle, to create a depression between 1 and 1.3 cm in depth. The vial should fit securely in the hole. A slot was cut along the back of the foam board, approximately 5.1 cm from the vial. This should be sufficiently long (just over 6 cm in length) to fit the smaller piece of foam board, which functions as a backdrop for viewing the sample. The backboard was not glued in place, as it should be removable for storage and transportation. Finally, using a hot melt glue gun, the second black foam board was attached to the underside of the testing base to provide support. **Figure 2** shows this set-up in cross-section.

## 2.2. Testing for the presence of chlorides

Salt solutions of known concentrations were made as instructed in Riss (1993), using 99.5% pure sodium chloride (NaCl) (Fisher Scientific). Stock solutions of 4000 ppm (parts per million) and  $2.725 \times 10^5$  ppm were prepared and diluted using distilled water as required, giving a wide range of concentrations, from 10 ppm to  $2.725 \times 10^5$  ppm, which were used during testing as required. Zero ppm (water) was used as a negative control.

Salts for testing samples from buildings were obtained from visible efflorescence on the stonework of St Mary



**Figure 2:** Cross-sectional diagram showing the layout of the test kit.

le Wigford, Lincoln, and of Lincoln Cathedral (both limestone), and from the University of Lincoln's Student Union bar (Tower Bar) brickwork, Lincoln, UK.

For testing of stock solutions, or other solutions which were already available in a liquid form, 4 ml test vials were prepared with a little less than 3 ml of distilled water using a plastic pipette, and 220  $\mu$ l of 10% nitric acid (Fisher Scientific) was added. Four drops of salt solution were added, and the vial was shaken for 15 seconds. The vial was placed into the test kit and 440  $\mu$ l of 1% silver nitrate solution (Fisher Scientific, made in house from 1M stock solution) was added. The phone flashlight was switched on and the vial examined against the light. A positive result showed a precipitate. To carry out a semi-quantifiable test, vials with known concentrations of chlorides were prepared, alongside the unknown samples tested.

When testing a solid sample, wearing gloves, an area of approximately 4 cm<sup>2</sup> was brushed using a soft paintbrush (size 6, Liquitex Basics) or glass-fibre brush in order to obtain the sample. The vial containing distilled water and nitric acid was placed into the support base and the salts were carefully placed into the vial. The vial was shaken by hand with the lid closed for approximately 40 seconds, or until salts had fully dissolved. The solution was filtered into another vial using a plastic funnel and filter paper (Whatman, Grade 1, 1001–070). 440  $\mu$ l of 1% silver nitrate solution were added, and the vial was examined using the test kit as described previously.

Quantofix® test strips (from Macherey-Nagel GmbH) were used in accordance with the manufacturer's instructions (Quantofix, 2013) to test the NaCl stock solutions of known concentrations. The strips used indicated a range of positive readings for chlorides from 500 mg/L (mg/L being equivalent to ppm) to a maximum level of greater than 3000 mg/L, in increments of 500–1000 mg/L.

The silver nitrate boiling test was used as described by Riss (1993) on known salt stock solutions for comparison. Where the samples were initially obtained as salt crystals, in order to dissolve the salts this test was used with the following modifications: a 0.5 g sample was boiled in 5 ml distilled water for 2 minutes. The solution was then filtered to remove undissolved components (see section 3.2). After cooling, 550  $\mu$ l of 10% nitric acid and 1.1 ml of 1% silver nitrate were added. A positive result was indicated by a white cloudy precipitate.

### 2.3. Salt contamination of objects

Samples of new unglazed earthenware (fragments of a modern flower pot); 5 cm × 3 cm pine blocks; 1 cm<sup>3</sup> Lincoln limestone blocks (from Lincoln Cathedral quarry); and iron alloy nails were contaminated with salts. One sample for each material type was used as a control. A concentrated salt solution was created by dissolving 109 g NaCl in 400 ml of distilled water at room temperature (20°C), the point at which no further salt would clearly dissolve. This created a solution of 27.25% NaCl, or approximately  $2.725 \times 10^5$  ppm. In order to saturate the materials with chlorides, three specimens of each material type were placed into covered glass beakers of the salt solution for 7 days at room temperature. The control samples were placed in individual covered beakers of distilled water, which was confirmed to be free of salt using Quantofix® and the silver nitrate test. After one week the samples were removed from the soaking solutions, and placed onto individual labelled tiles before being placed into a Genlab Mino Economy oven to dry at 40°C for 42 hours, which was determined experimentally to give a suitable level of surface salt efflorescence. Soak solutions were retained and tested for salts to determine uptake; however, as the salt levels were all outside the maximum limit of detection this was not reliably quantifiable. In order to test the effectiveness of the protocol at lower salt levels, this method was repeated using a 5% w/v NaCl solution, prepared using 20 g of salt in a total volume of 400 ml distilled water.

Samples were brushed to test for the presence of salts with a size 6 paintbrush (Liquitex Basics), over an area of 4 cm<sup>2</sup> in the case of the ceramic, wood and limestone; the entirety of the iron alloy nail and limestone cubes were brushed. The salts were collected onto filter paper and then tested as described in section 2.2.

## 3. Results

### 3.1. Testing limits of detection and optimising quantification

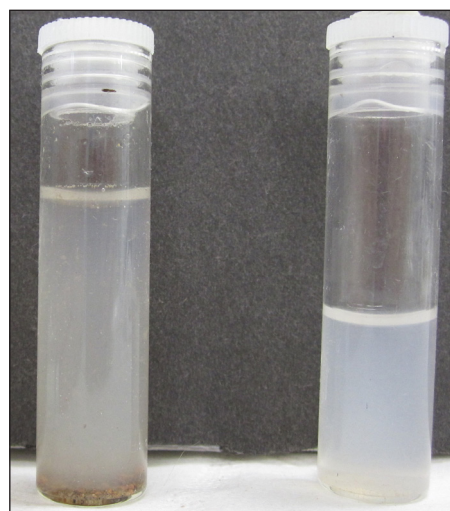
In order to determine the extent of quantification possible using this surface chloride detection method, it was necessary to ensure that all stages of the sampling and testing process were as repeatable as possible. In order to achieve this, a more consistent method was developed for the taking of initial samples. In their original paper, Piechota and Drake Piechota (2016) suggest using a soft brush for a few strokes of a small area of the object's surface. The problem with this was that there was no way to quantify samples, as there were too many unknown variables to deal with, such as unknown amount of sample and what qualified as a few brush strokes. Therefore, it became apparent that a set area would be more reproducible. A sample size of 4 cm<sup>2</sup> was chosen when collecting samples from site, as this gave sufficient salts to give a detectable reading. Testing was also carried out to determine the optimum quantity of distilled water, silver nitrate and nitric acid used in the vials (data not shown).

After shaking samples by hand and timing the results, it appeared that 40 seconds was sufficient time for the majority of the sample to dissolve. However, testing

samples from three building sites across Lincoln showed that parts of each sample were not soluble, but affected the results. The presence of crumbled stone and brick particles, being removed with the efflorescence, affected the appearance of the solutions by reflecting their own colours when the phone light was present, as well as making the solution appear cloudy before adding silver nitrate. Due to this it became hard to determine accurate results from the samples. To remedy this, a filtration step was added to the method, as it removed undissolved impurities from the solution, thus returning it to a clearer state (**Figure 3**).

The limits of detection of the kit were determined by using a range of concentrations, from 10 ppm to the maximum dissolved salt concentration of  $2.725 \times 10^5$  ppm. The respective cloudiness of the solution was compared visually to the control solution. While other methods of assessment of the solution colour may have been more accurate, such as spectroscopy, they would all require additional laboratory equipment, while the test kit of this study was designed as a quick on-site method to evaluate the tests' effectiveness.

The lowest level of detection that could be semi-accurately identified using the testing kit method was 30 ppm. When compared to the control sample of water, silver nitrate and nitric acid without additional salts, 10 ppm was visually identical. A concentration of 25 ppm was too similar to one of 10 ppm and to the control to give any reliable visible difference. It was only once 30 ppm concentration was reached that it was possible to see a visible difference in cloudiness of solution between the control and the test solution. Although 30 ppm and 40 ppm were clearly different from 10 ppm and 50 ppm, due to their similar appearance it was not possible to accurately differentiate between the former two. When 50 ppm levels were reached, there was a clear differentiation visually between the quantities of precipitate in comparison to 40 ppm; therefore it is at the 50 ppm point that the quantification of the test becomes more realistic.



**Figure 3:** Samples from the Tower Bar, Lincoln, showing the effect of filtration. The unfiltered sample is on the left, and the filtered sample on the right.

The upper limit of detection was also tested, using solutions ranging from 100 ppm chlorides upwards. It was clear from the testing that all of these solutions gave a strong positive result. However, quantification becomes visually indistinguishable from 4000 ppm upwards. While it was possible to see that there was a higher level of precipitate at, for example,  $1.4 \times 10^5$  ppm, it was not possible to reliably determine the actual concentration (**Figure 4**).

Therefore, it is possible to give a semi-quantifiable level of chlorides between 50 ppm and 4000 ppm, or to state the level is greater than 4000 ppm, but without more specific accuracy. Results obtained that appear below 50 ppm do not necessarily denote a lack of chlorides, and may be noted as possible low level chlorides that cannot, however, be accurately given a ppm concentration level.

However, it was noted that while the quantification is possible, it was difficult to determine this accurately without reference samples. A reference photograph across a range of concentrations was taken as a guide for comparison (instead of carrying out stock concentration tests in the field). The exact accuracy of the reference photograph depends on the quality of the printer and computer colour calibration. This approach does have significant limitations, and so preparing a set of stock reference solutions would give a more reliable set of references.

### 3.2. Evaluating the accuracy of the test kit in comparison to alternative methods

The testing kit method was compared to Quantofix® testing strips for chlorides and to the standard silver nitrate test in order to evaluate its accuracy and ease of use.

Testing strips are a supposedly quantitative way of identifying the amount of salts in a solution; however, it is more precise to state that the range of detection depends on the exact range of the test strip, and will give a rough range (semi-quantitative) rather than an exact quantity. In order to ensure the test strips gave reliable semi-quantitative results, the pre-made stock solutions were tested using the strips, and the results were compared to expected values. The results of the testing can be seen in **Table 1**.

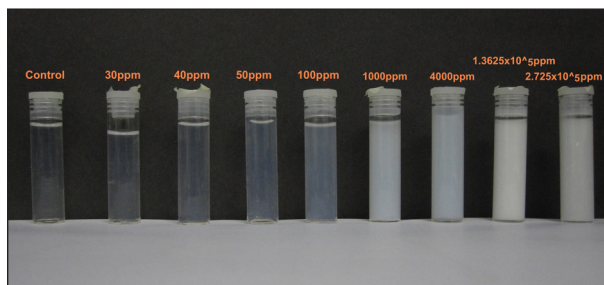
As expected, the test strips read the salt concentrations within their given range correctly. The reading was less accurate when presented with a solution slightly above the maximum detection limit; for example, for the 4000 ppm solution the test strip returned a colour consistent within the range of 1500–3000 mg/L. When presented with

concentrations much higher than the strip's maximum detection limit of 3000 mg/L, the reading correctly showed a strong positive for being greater than or equal to 3000 mg/L. In comparison the silver nitrate test kit used in this study provides a greater potential range for semi-quantifiable readings than the chloride test strips, as it gave a reliably visually quantifiable range of 50–4000 mg/L.

The other standard method used for testing of chloride concentrations, in particular of solid samples, is the silver nitrate test described by Riss (1993) with the addition of a boiling step to reduce the time taken for salts to dissolve, which is carried out as a standard method at the University of Lincoln. Salt samples were collected from the Tower Bar brickwork at the University of Lincoln and were tested using the standard silver nitrate method and with the test kit method. Results were compared visually against pre-prepared stock solutions to determine whether both methods gave similar results. In order to determine the accuracy and reliability of testing across a range of sample sizes, different sample weights, from 0.1 g to 0.5 g, were tested.

Initial tests took place on unfiltered samples; however, the unfiltered sample tended to be cloudy prior to the addition of silver nitrate because of the presence of particulates from brickwork or stonework removed alongside efflorescence, and was much more difficult to interpret. While it was possible to state whether the results were positive or negative, giving any semi-quantitative estimate of concentration of chlorides in unfiltered solution was not possible due to the initial discolouration of the liquid. In order to refine the methodology further, the addition of filtration to the original method was proposed by the authors, in order to remove insoluble matter from the solution before testing.

With the addition of filtration, the results from the silver nitrate test kit method could be accurately compared with those of the standard silver nitrate test (including boiling) method. Both methods reproducibly gave comparable results, with readings from 100 ppm for 0.1 g to just under 1000 ppm for 0.5 g, meaning that they could be effective for quite small samples. Given the level of accuracy of the testing, and the fact that chlorides may not be evenly distributed throughout the sample collected, the methods appear equally effective. A negative aspect of filtration is the loss of some of the sample, as it was absorbed by the



**Figure 4:** Examples of test solutions used to determine quantification and limits of detection of chlorides.

**Table 1:** Comparison of chloride level results from testing strips with known concentrations of salt stock solutions.

Stock concentration	Result from Quantofix® chloride test strips (0–3000 mg/L)
100 ppm	Between 0 and 500 mg/L
1000 ppm	1000mg/L
4000 ppm	Between 1500 mg/L and 3000 mg/L
$1.3625 \times 10^5$ ppm	Greater than or equal to 3000 mg/L

filter paper. However, this did not appear to affect the quantification of the readings, as the filtered sample gave a reading comparable to that of the other method. The use of the black background and standardised light source from the phone flashlight made quantification easier and more reproducible under varying or low light conditions. Avoiding boiling of the samples means that testing can be carried out on site using the modified, portable test kit. It also gives a very quick, clear, visual indicator of the presence of chlorides, which can be easily understood by non-specialist audiences. **Figure 5** shows the kit being demonstrated on site by undergraduate conservation students during a public engagement event at St Mary le Wigford church, Lincoln.

Salt testing strips can be a useful way to measure a range of chloride ion concentrations in rinse water from object desalination treatments, as results can be obtained in a matter of seconds after the process of desalination has taken place (Odegaard *et al*, 2011). The testing kit method would also be beneficial for testing surface salts on objects and buildings in addition to testing of rinse water, as results can be obtained without having to submerge or introduce water to the objects' surfaces.

### 3.3. Testing the efficacy of the kit on chloride contaminated objects

In order to determine the testing kit's ability to detect chlorides on various objects, tests were performed on a range of materials pre-treated with both a high concentration of salts and a lower level, to check effectiveness. This pre-treatment step was carried out on new, surrogate materials (pine, iron alloy, limestone and earthenware) as these are commonly affected by the presence of salts. The aim of this test was to determine whether a positive result could be obtained from objects.

After soaking the objects in a saturated salt solution, they were oven dried to encourage surface salt crystallisation (see section 2.3). After this step, it was clear that the salt had led to surface damage of the samples, in particular the limestone, where pitting and losses were observed.

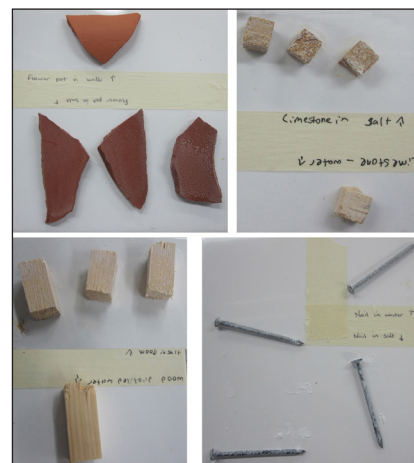


**Figure 5:** Undergraduate Conservation of Cultural Heritage students demonstrating the use of the testing kit to the public, on site at St Mary le Wigford church in Lincoln, UK.

The pine, earthenware and limestone all showed a clear colour change after the treatment with the salt solutions, and had visible surface salt crystals. The nail showed surface corrosion, with salt crystallisation on the surface. The control samples, which had been soaked in distilled water and dried using the same process, did not demonstrate any visible surface change, and no salts were present. Examples of crystallisation can be seen in **Figure 6**, showing salt crystals and colour change of the chloride contaminated materials.

Salts were gathered from each of the four types of contaminated objects using either a paintbrush or a glass-fibre brush, and tested using the kit as described previously. The results were compared to readings prepared using stock solutions of known concentrations. Since the samples were from an area of 4 cm<sup>2</sup> the readings are measured in parts per million for 4 square centimetres (ppm/4 cm<sup>2</sup>). Salt concentration results are shown in **Table 2**. The weight of salt obtained from each sample was minimal, measuring between 0.00 and 0.02 g. The control samples all gave a negative result, with no salt recorded (data not shown).

The results confirmed that a 4 cm<sup>2</sup> sample size was sufficient to give a positive result for the materials tested. Within the readings for each material, there was considerable variability in the salt levels found when salts were removed using a soft brush. As the materials came from the same source, and were all exposed to the same levels of salt over the same time period, it would have been expected that variability would have been generally relatively low. From the appearance, it was clear that the earthenware and limestone samples had significant salt crystal growth on the surface, but when it came to brushing the salts off, not all the salts were removed as the crystals were so strongly bonded. The samples were re-tested using a glass-fibre brush (**Table 2**). This was effective at removing a greater quantity of salt from the surface, and consequently gave a more representative indication of the high salt levels in each material. Therefore, while it was possible to state whether the results were positive or



**Figure 6:** Pine, limestone, iron alloy and earthenware after soaking in a saturated salt solution and drying for 42 hours, showing the presence of salts.

negative from using a paintbrush, in this case the test kit was unable to give reliable semi-quantification without the application of an alternative salt removal method, ie the use of a glass-fibre brush. This alternative removal method, however, could be potentially damaging to the surface of an object and should be used with caution. Where salts are less strongly bonded, this would not be such an issue, as a paintbrush would be effective.

In order to study the success of the method at lower salt levels, additional samples were prepared using a 5% salt solution, which may be more representative for real-world salt contaminated historic objects. Observation of the samples showed corrosion of the nail, and a small amount of salt crystals were visible to the naked eye on other materials, though considerably less than seen with the saturated salt samples. Results of testing are shown in **Table 3**.

**Table 2:** Chloride concentration readings obtained from 27% salt contaminated materials.

Material	Sample number	Paintbrush, ppm/4 cm <sup>2</sup>	Glass-fibre brush, ppm/4 cm <sup>2</sup>
Wood	W1	>4000	>4000
Wood	W2	>4000	>4000
Wood	W3	>4000	>4000
Nail	N1	2500	4000
Nail	N2	>4000	>4000
Nail	N3	2500	4000
Earthenware	F1	75	>4000
Earthenware	F2	550	>4000
Earthenware	F3	550	>4000
Limestone	L1	100	4000
Limestone	L2	4000	4000
Limestone	L3	4000	>4000

**Table 3:** Chloride concentration readings obtained from 5% salt contaminated materials, using either a paintbrush or a glass-fibre brush to remove salts for testing.

Material	Sample number	Paintbrush, ppm/4 cm <sup>2</sup>	Glass-fibre brush, ppm/4 cm <sup>2</sup>
Wood	W4	75	100
Wood	W5	50	50
Nail	N4	2000	2000
Nail	N5	2000	3000
Earthenware	F4	50	100
Earthenware	F5	50	200
Limestone	L4	100	100
Limestone	L5	50	50

The results showed a generally lower salt concentration, as would be expected from the initial lower salt levels in the soaking solution. Use of a glass-fibre brush to obtain salts resulted in levels that were either higher or the same as those obtained when using a paintbrush, as was observed with saturated salt tests, demonstrating that, as before, the use of a harsher removal technique does give increased or similar removal of salts from the surface. However, as the paintbrush method did detect the presence of salts, it is still an effective and quick indicator of the presence of chlorides, even if the quantification is less reliable. It was particularly noticeable that the low positive levels, around 50 ppm, were only a clear positive when viewed in the light from the test kit; when examined by eye without the test kit, they did not appear to be significantly different to the control. The test kit therefore is particularly helpful for detecting these low chloride contamination levels.

#### 4. Discussion

The value of the testing kit is that it can help conservators optically confirm the presence of chlorides during surveys and work on site, as well as that it has the potential to give a semi-quantifiable part per million concentration range, indicating how strongly the object or building might be contaminated.

The testing kit allows conservators to carry out the silver nitrate test on site where access to laboratory equipment is not possible. The use of a consistently strong light source and dark background ensures that chlorides can be detected in various environments, such as in storage rooms where light levels may be low, and outdoors where lighting may not be very even. By itself, the revised silver nitrate test is not a quantitative test; however, by creating stock ppm salt solutions and a reference image, the results of the test can be compared to known concentrations, making the results gathered more quantifiable.

The results gathered on site using the revised method are comparable to those of tests carried out in the laboratory using the boiling method with the standard silver nitrate test, as well as to those of test strips for the presence of chlorides. All these methods gave roughly the same results when tested against known concentrations of chlorides, indicating that the tests were able to reliably give semi-quantifiable information.

When testing takes place on only a small area of a building or object, results may be misleading, as this does not necessarily mean that the entire building or object, for example, has the same chloride levels throughout. However, such testing can still provide a useful guide. Due to this, the testing kit may also prove useful when it comes to on site desalination work. There are limitations to any surface salt testing method, since such methods do not detect salts within the object; if this latter information was also needed, it would be important to carry out additional testing in order to establish non-surface salt levels. For example, the method could be used as a cheaper alternative to chloride test strips to test rinse water from object immersion, which extracts salts from within the material, as well as to test chloride concentrations of poultices.

We have demonstrated that the revised testing kit method worked with multiple materials; however, the sampling methods meant that it was problematic to gather samples where the salts were tightly bound to the surface. This led to a large variability in results, although all salt contaminated materials tested positive as expected. While the paintbrush was not always able to easily remove salts from objects where salts were tightly bound to the surface, it was a non-damaging tool and did not cause any damage to the surface of the object. The glass-fibre brush was more effective, but risked surface scratching and damage or loss of friable surfaces. An alternative approach, where salts are tightly bound, could be to use a scalpel, although this also has the potential to damage the surface of the object being tested. Another possibility for collecting samples would be to use a damp brush or cotton swab, and soaking the brush or swab in the vial before carrying out filtration. While the use of damp cotton swab could help to remove chlorides without scratching the surface, it would introduce moisture to the surface of an already contaminated object, potentially leading to further salt distribution within the object.

## 5. Conclusion

The revised method of testing for the presence of salts using a mobile silver nitrate testing kit with light source has reliably given positive results for the presence of chlorides collected as efflorescence from building surfaces (both limestone and brick), and was able to reliably detect the presence of chlorides in a range of materials such as wood and metal. No false positives were found during testing.

All the methods used for comparison during this study had the potential to give semi-quantifiable results for chloride levels. However, the new test kit had the potential to give more precise results than test strips, when combined with stock salt concentrations as reference. Test strips are limited to testing on aqueous solutions only, such as through the rinse water method. The test kit is more versatile, since it can also be used with dry samples, including salts taken directly from the surface of objects and buildings. The use of the black background and even light source, as suggested by Piechota and Drake Piechota (2016), meant that the results were more comparable than the standard silver nitrate test for chlorides. Preparing the vials in advance gives the potential to carry out a number of high speed tests on a large quantity of objects in situ. The test may not be appropriate for all situations, such as where brushing the surface could cause damage, and so a careful evaluation of the purpose of testing should be carried out. For dry surface salts, especially for on site conditions, this method is particularly suitable.

One unexpected benefit of this kit was its ability to provide an opportunity for public engagement. Salt testing is usually carried out under laboratory conditions; however, with increasing interest in the work of conservators, the potential for using this as an engagement tool should not be underestimated. When this kit was used as part of a public display, with live testing taking place, it did an excellent job of engaging interest. The reference photograph for standard stock salt solutions allowed members of the

public to interact more in depth, through comparing the reference image to the test vial, under the guidance of the conservator. Increasing public awareness of the presence, and risks, of salt may have benefits for protection of our cultural heritage, through greater understanding of the risks involved in the use of salts in cities, for example in relation to the use of sodium chloride as a de-icing material. In addition, the straightforward testing process means that this work can be carried out by interns after some initial training.

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## Competing Interests

The authors have no competing interests to declare.

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