

## Salt Weathering of Porous Structures Related to Climate Changes

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### Abstract

The relationship between environmental conditions and the growth and dissolution of salts has been studied in specific areas of three different monuments. For this purpose, a non destructive method „Monitoring of the crystallization of salts in combination with climate measurements“ was developed which allows an understanding of the interaction between the existing salts and the ambient climate. In the crypt of St. Maria im Kapitol damage and decay of stones and wallpaintings have been caused by cyclic crystallization and dissolution of the salts halite [NaCl] and nitrate [NaNO<sub>3</sub>] controlled by climate changes through the heating of the crypt. In the choir of St. Johannes in Mußbach wallpaintings are weathered by temperature controlled crystallization of niter [KNO<sub>3</sub>]. In the Grotzensaal of the New Palace in Potsdam the walls are deteriorated by crystallization of mirabilite [Na<sub>2</sub>SO<sub>4</sub>·10 H<sub>2</sub>O] and its transformation into thenardite [Na<sub>2</sub>SO<sub>4</sub>] and vice versa (dehydration / hydration) which is controlled by the surrounding climate (temperature and relative humidity). Understanding the behaviour of soluble salts in relation to the existing climate an appropriate conservation program can be developed.

**Keywords:** Salt weathering, solubility, hygroscopicity, climate monitoring, environmental control

### Klimaabhängige Salzverwitterung poröser Strukturen

#### Zusammenfassung

*Die Kristallisation und Auflösung von bauschädlichen Salzen in Abhängigkeit von den Umweltbedingungen wird anhand dreier Beispiele beschrieben. Die Herangehensweise „Monitoring von Salzkristallisationen in Kombination mit Klimamessungen“ wurde als Methode entwickelt, um das Zusammenspiel zwischen Salzaktivitäten und den umgebenden Klimafaktoren an einem Bauwerk zu erfassen. In der Krypta von St. Maria im Kapitol in Köln verursacht zyklisches, durch Heizung initiiertes Kristallisieren der Salze Halit [NaCl] und Nitronatrit [NaNO<sub>3</sub>] die Schädigung von Baustein und Wandmalereien. Im Chor von Mussbach werden Wandmalereien durch Temperatur-bedingtes Kristallisieren des Salzes Nitrokalit [KNO<sub>3</sub>] beeinträchtigt. Im Grotzensaal des Neuen Palais in Potsdam verwittern einzelne Oberflächen durch klimagesteuerte Umwandlung (Hydratbildung) des Salzsystems Thenardit [Na<sub>2</sub>SO<sub>4</sub>] und Mirabilit [Na<sub>2</sub>SO<sub>4</sub>·10 H<sub>2</sub>O]. Das Wissen um die an einem Bauwerk vor sich gehenden Verwitterungsprozesse ist die Grundvoraussetzung für die Entwicklung eines angemessenen Konservierungskonzeptes.*

**Stichwörter:** Salz, Salzkristallisation, Salzverwitterung, Löslichkeit, Hygroskopizität, Klima, Monitoring

## 1 Introduction

Salt weathering on monuments plays a dominant role in the desintegration of cultural heritage. In general, salts in monuments originate from ions that have been leached out of the ground, the building material, the depositions from a polluted atmosphere and from biological metabolism. These ions circulate in aqueous solutions within the walls and porous materials. Where water evaporates the salts accumulate and the solutes concentrate. Where and when supersaturation is reached, different phases precipitate and fractionate from multicomponent salt solutions [1,2]. If salts crystallize or hydrate within a porous structure this process often leads to a disruption of the internal structure, which can cause powdering and/or the loss of surface material.

The growth and dissolution of salt species depends on the environmental conditions of the specific monument [2]. If the relative humidity is lower than that corresponding to the saturated solution of the salt in question, crystallization will occur. If, on the other hand, the relative humidity exceeds the equilibrium relative humidity of the salt, it will remain in solution. Should the equilibrium relative humidity of a salt fall within the range of variation of the ambient relative humidity, cyclic dissolution and crystallization will accelerate the deterioration of the building materials that contain this salt. Therefore, the processes which are causing the damage to the monument have to be determined to develop an appropriate conservation approach.

This paper describes a non-destructive method developed to investigate this issue and its application to three case studies.

## 2 Monitoring of Salt Crystallization in Combination with Climate Measurements

To preserve a monument it is important to find out and understand the chemical and physical processes damaging the building materials and the conditions under which this decay occurs. Crystallization and hydration/dehydration of salts occur at a given temperature and at particular relative humidity. If the climatic conditions under which a salt will effloresce or hydrate could be predicted, it would be possible to know what climatic conditions have to be maintained to avoid crystallization cycles.

Hence, the first investigation step is mapping and describing the damage on a monument indicating where damage appears and where samples for analyses can be taken. Then, salt efflorescences are sampled from the surface of the building materials and analysed – e.g. microscopically and microchemically following established procedures [3] – or determined using X-ray diffraction analysis.

When it is well-known which salts are producing the damage, it is necessary to investigate under which climate conditions the crystallization takes places. Therefore, we have to define areas (e.g. a size of ca. 10 cm<sup>2</sup>) for each analysed salt species on the surface of the building materials where newly crystallized salts can be regularly observed. On these fields new efflorescences are periodically removed and analysed to allow predicting when the salt will crystallize.

Simultaneously, measurements of the room climate are taken. Air temperature and relative humidity as well as the surface temperature at the coldest point in the monument are measured. The absolute humidity and the dew point temperature can be calculated. The climate measurements and calculations indicate the degree of temperature and humidity changes and show if condensation on surfaces takes place.

The climate measurements combined with the periodic observation of the crystallizing salts permit to determine under which climatic situation the different salts will crystallize and what kind of damage they produce.

This method has been called *Monitoring of salt crystallization in combination with climate measurements* and has been applied to sections of some monuments, as described below.

## 3 Crypt of St. Maria im Kapitol, Cologne

### 3.1 History

It is important to know the history of the church of St. Maria im Kapitol in Cologne to understand the salt weathering processes in the crypt. A brief review is given below.

The church was constructed between 1015 and 1065. The crypt is located under the choir in the eastern part of the church. It lies about one third to one half of its height under ground. The walls of the crypt are built from different kind of building stones and the vaults are adorned with Romanesque

wallpaintings. During their eventful history these paintings have been covered for an unknown period of time, to be uncovered around the turn of the 19<sup>th</sup> century. Their condition at that time is documented by aquarelle copies. Later in that century, between 1838 and 1851, the crypt was utilized as a salt depot. During the Second World War, the crypt was partially destroyed. Thus today, the only original building materials of the vault carrying Romanesque wall paintings are in the eastern part of the crypt. In 1969, a floor heating system was installed, and this was replaced by a hot-air heating in 1987. Since 1977, the walls have been partly covered by a sacrificial lime plaster, leaving only the original building materials and wallpaintings visible.

### 3.2 Salts and Damages

Extreme damage to the various stones and plasters is apparent and can be attributed to the salt concentration within the walls. The degree in salinity, as well as the deterioration, decreases from bottom to top of the walls. At a height of 2,5 m no actual salt efflorescences can be observed, but by regular collection of the material that falls down from the vaults, it was proven that weathering processes are also occurring in the vaults and that these result in the loss of wall painting material [4].

On the surface of the plaster covered walls, fluffy efflorescences and crusts exist, and subflorescences

lead occasionally to the bursting of the plaster. Fluffy efflorescences can often be observed in the vicinity of fissures and cracks of the plaster. Crusts are partly 2 mm thick and their occurrence is probably linked to a high salt content in the wall.

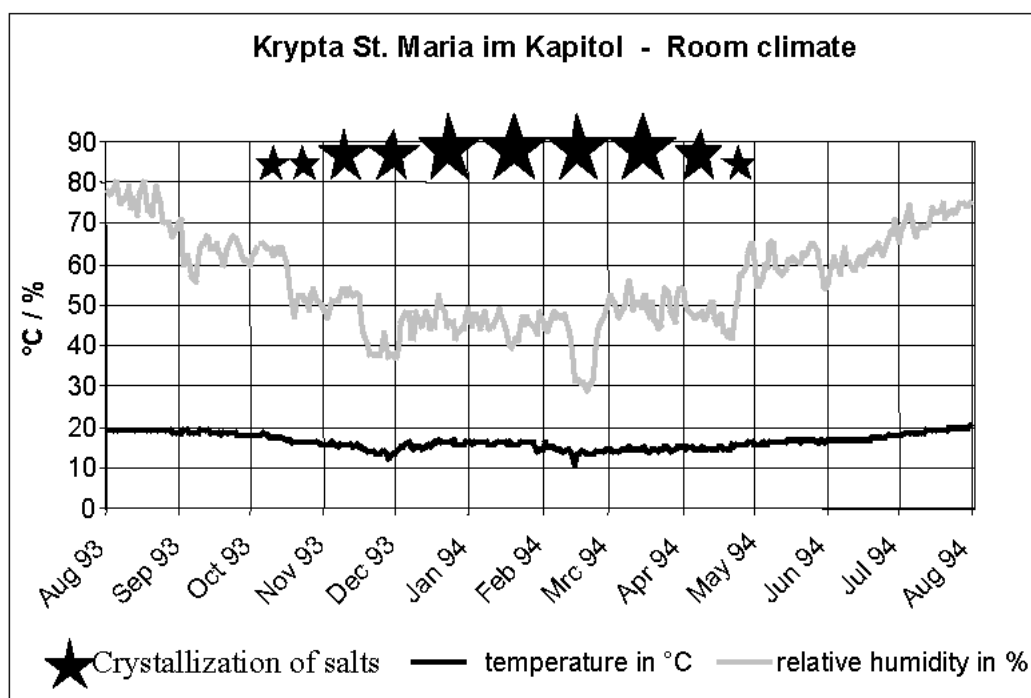
Efflorescing salts and crusts were sampled for analysis. They are most frequently formed by a mixture of halite [NaCl] and nitratine [NaNO<sub>3</sub>]. Only to a small degree thenardite [Na<sub>2</sub>SO<sub>4</sub>], mirabilite [Na<sub>2</sub>SO<sub>4</sub>·10H<sub>2</sub>O], epsomite [MgSO<sub>4</sub>·7H<sub>2</sub>O], gypsum [CaSO<sub>4</sub>·2H<sub>2</sub>O] and trona [Na<sub>3</sub>H(CO<sub>3</sub>)<sub>2</sub>·2 H<sub>2</sub>O] are present in the crypt.

The dominance of chlorides can be attributed to the use of the crypt as a salt depot. Nitrates have probably migrated into the walls from ground moisture. The source for sodium ions can be attributed to the Portland cement used in repairs.

### 3.3 Room Climate

The climate in the crypt was measured by a recording thermohygrograph which was controlled and calibrated by an aspirated psychrometer. The wall surface temperature was determined with an electrical Ni-Cr-Ni contact.

The averaged daily values of temperature and relative humidities curves for one year are shown in Fig. 1. In general, the room temperature varies from about 20 °C in the summer to about 13 °C during



**Figure 1:** Evolution of the average daily values of temperature and relative humidity in the crypt of St. Maria im Kapitol, Cologne, related to the observed salt crystallization

winter months. During the summer months of July, August and September, the crypt is quite moist with relative humidities within the 70 to 80 % range. By starting the heating in October the relative humidity decreases slowly and, during winter, it oscillates around 50 %, while in January or February, it may drop to around 35 %. The dry period continues until April, when the relative humidity rises, so that the crypt becomes quite moist again by July.

From the measurement of the wall surface temperatures the dew-points could be calculated. It was shown that the dew-point is not reached in the crypt at any time during the year.

### 3.4 Interaction of Salts and Room Climate

After two years of measuring the climate and observing the behaviour of the salts in the crypt, it is possible to predict the main salts that will effloresce under given environmental conditions [5,6].

In the moist summer months, July, August and September, the fluffy efflorescences of halite and nitratine disappear. By starting the heating system in October small salt crystals of halite and nitratine begin to grow. More and more salts crystallize until April, at which point the highest damage and decay in the crypt can be observed (see Fig. 1). In the transitional period, around May and June almost no crystallization or dissolution of halite and nitratine is perceptible until July, when the dissolution of the main salts becomes visible again.

The critical relative humidity in the crypt is around 65-70 %: above this range halite and nitratine dissolve and below it they crystallize. However, the exact point could not be determined with the applied measurement equipment.

From laboratory experiments with saturated salt solutions the equilibrium relative humidities of single salts are known [7] indicating the dissolution or crystallisation of the respective salt. The equilibrium relative humidity of halite is 75,5 % and that for nitratine is 75,4 % at 20 °C. Variations in temperature do not influence these values particularly. Therefore in the crypt the dissolution or crystallisation of halite and nitratine should take place around 75 % relative humidity. But the results of the observation and measurements demonstrate that the equilibrium relative humidity of these salts is reduced to values around 65-70 % in contrast to that of the single salts. The reason for this is probably the different thermodynamic properties of salt

mixtures in comparison to the thermodynamic properties of single salts.

Current research is attempting to calculate how mixed salt solutions under particular thermodynamic properties will behave [8]. These theoretical calculations show that the dissolution or crystallisation of the halite/nitratine system occurs between 65-70 % relative humidity thus verifying the observations and measurements from the crypt.

### 3.5 Conclusions

In the crypt of St. Maria im Kapitol the damage and decay of the stones and plasters has been caused mainly by cyclic crystallization and dissolution of the predominant halite and nitratine salt-mixture.

The critical relative humidity in the crypt is around 65-70 %, when either crystallization or dissolution of halite and nitratine appear. Thus, to prevent salt damages in the crypt, it is suggested as a preliminary measure to avoid heating for one winter period and observe which relative humidity will be reached and how the salt system will react to the changed environmental condition.

## 4 St. Johannes in Neustadt / Mußbach

### 4.1 History

The Gothic church St. Johannes in Neustadt / Mußbach (Palatinate, southwestern Germany) was constructed in 1370 and decorated with wallpaintings. At the beginning of the 18<sup>th</sup> century, following the Reformation, the church was subdivided by a wall between the choir and the nave. The choir was used by the Catholics, and the nave by the Protestants, to celebrate their services. Meanwhile, the Gothic wallpaintings have been covered for an unknown period of time. In the 19<sup>th</sup> century the choir was painted again. To preserve the walls from rising damp a concrete wash was applied in 1930.

In 1959 the catholic parish of Mußbach received a new church. Thus, the choir of St. Johannes was never used again for services and it was no longer maintained. Consequently, the weathering of the building materials has accelerated. In 1967, during an investigation of the choir, the Gothic wallpaintings were rediscovered and uncovered where they are still to be seen.

## 4.2 Salts and Damages

The extreme damage to the building materials and wall paintings is apparent and related to moisture influx and salt crystallization. The degree of deterioration decreases from the ground up to a height of ca. 1,50 m of the walls. Severe damage can be observed near the windows, which were not water-tight for several years.

According to the observations and mapping of salt efflorescences their related damage it can be established that at the outer walls, surface and ground water rise probably by capillarity into the walls. Where water evaporates, salts lead to the decay of the building materials, e.g. fluffy efflorescence can often be observed on the surface and lead to the bursting of wallpainting fragments.

In the lower part of the choir, niter [ $\text{KNO}_3$ ] crystallises most frequently producing the damage. Nitrates have probably migrated into the walls with the ground moisture, whereas potassium is related to the Portland cement wash (alkaline building material), which was used to prevent this rising damp.

## 4.3 Room Climate

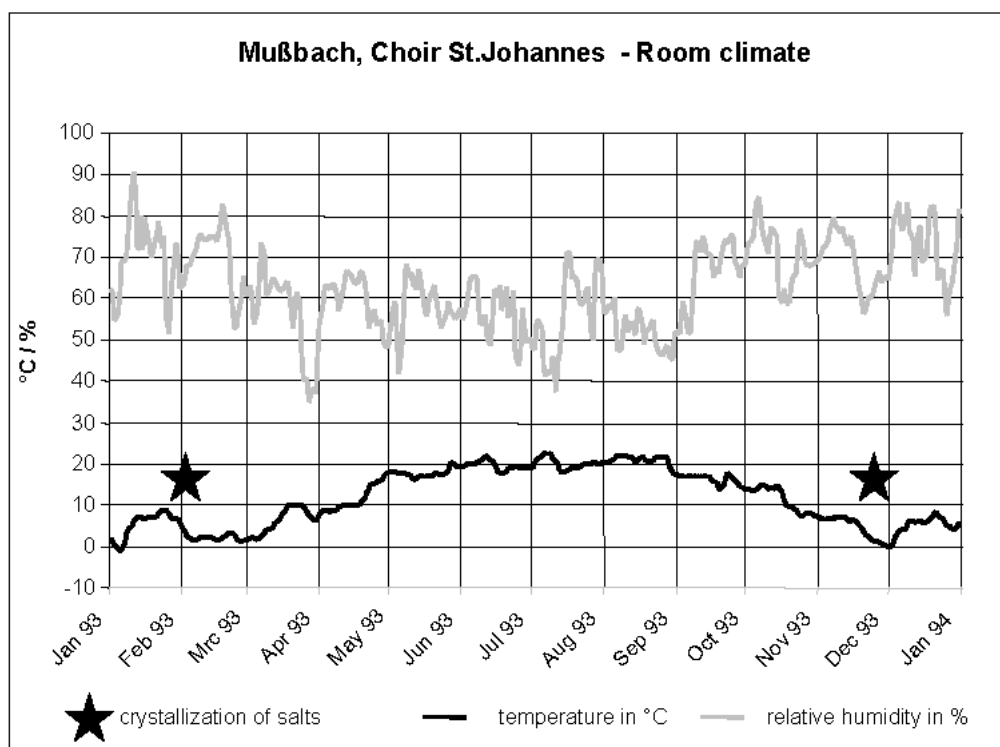
The evolution of the room climate in the choir of St. Johannes is shown in Fig. 2 where the average

daily values for relative humidity and temperature are plotted. The choir is not heated and, hence it is strongly influenced by the outdoor climate. In general, the room temperature varies from about 22 °C to about -2 °C and the relative humidity oscillates between the range of 35 % and 90 % RH. However, changes in temperature and relative humidity can develop rather quickly depending on the outdoor climate situation.

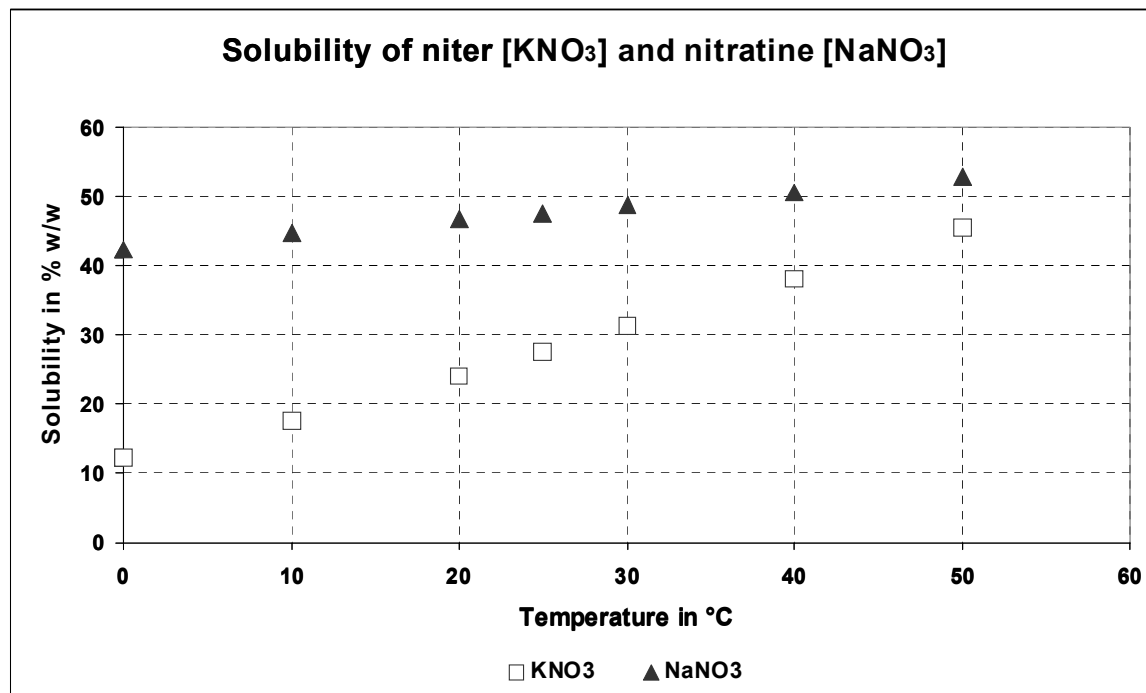
## 4.4 Interaction of Salts and Room Climate

After nearly two years of monitoring the crystallization of niter [ $\text{KNO}_3$ ] in combination with climate measurements, it is possible to predict under which environmental conditions niter will effloresce [5]. New crystallizations of niter can be observed at low temperatures—in Fig. 2 this corresponds to February and December of the year 1993. If the temperature is below 10 °C and drops again several degrees, new crystallizations of niter can be found at the defined areas.

In order to understand this weathering process, it is essential to examine the solubility properties of niter. Fig. 3 shows the solubilities of both niter [ $\text{KNO}_3$ ] and nitratine [ $\text{NaNO}_3$ ] as a function of



**Figure 2:** Evolution of the average daily values of temperature and relative humidity in the choir of St. Johannes, Neustadt / Mußbach, related to the observed salt crystallization



**Figure 3:** Solubility of niter [KNO<sub>3</sub>] and nitratine [NaNO<sub>3</sub>] in H<sub>2</sub>O, data from [9]

temperature. As can be seen, the solubility of niter is strongly dependant on temperature as compared to that of nitratine. As an example, the solubility of niter is around 24 % w/w at 20 °C, while it is around 12 % w/w at 0 °C. This means that the solubility is by half by this change in temperature. Consequently, at low temperatures a solution containing potassium and nitrate ions reaches the saturation much earlier than at higher ones. The result is the supersaturation of the solution and the crystallization of niter at low temperatures.

Based on the investigations in the choir of Mußbach the following weathering processes can be assumed: nitrates and soluble alkalis (K<sup>+</sup>) migrate together with other ions in a solution which is fed by ground water through the building materials. Near the surface of the walls the solution becomes enriched NO<sub>3</sub><sup>-</sup> and K<sup>+</sup>, but during spring, summer and autumn supersaturation of the solution is not reached because the temperature is high. In winter the temperature is generally low resulting in a high concentration of nitrates and potassium in the solution of the wall due to the solubility of niter (see Fig. 3). Then, following a drop in temperature to below 10 °C the supersaturation of the solution is reached and niter crystallizes out.

Therefore, in the choir of Mußbach the critical temperature is around 10 °C: if the temperature is below it by several degrees, crystallization of niter

takes place and produces the consequent damage to the pore structures.

#### 4.5 Conclusions

According to the observations and measurements in the choir of Mussbach, salt weathering is apparent in the building materials and wall paintings, especially near the windows. Rising damp is the origin of the presence of ions such as K<sup>+</sup> and NO<sub>3</sub><sup>-</sup>. During the spring, summer and autumn the salt solution migrates within the pore system of the wall but does not precipitate. The salt solution reaches the saturation and niter crystallizes visibly if the room temperature is below 10 °C, and then falls again by several degrees. Thus, the wall paintings at the choir in Mußbach are affected by temperature controlled crystallization of niter [KNO<sub>3</sub>].

## 5 Grottensaal, New Palace, Potsdam

### 5.1 History

The New Palace was constructed between 1763 and 1769 under the reign of King Friederich II. from Prussia and is situated in the park of *Sanssouci* in Potsdam, Germany. The palace consists of more than 500 rooms and was built first and foremost for guests of the monarch. The show pieces of the palace are 4 halls for festivals, one of them being the *Grottensaal*, situated on the ground floor

in the centre of the palace towards the park. During the Baroque period it was quite fashionable to decorate halls with different decorative pieces from nature, such as minerals, rocks, fossils or shells, to make them look like a grotto. The *Grotten-saal* of the „New Palace“ in Potsdam is decorated in this manner and it contains an enormous collection (around 20.000 pieces) of minerals and rocks from all over the world plastered with a gypsum-mortar.

The restoration history of the *Grotten-saal* started immediately after the palace was finished: due to static problems, a new ceiling had to be introduced between the *Grotten-saal* and the *Marmorsaal* above it. Thus, the actual ceiling of the *Grotten-saal* had to be lowered.

After World Wars I and II further interventions were carried out to replace the loss of decorative pieces. During the several restoration phases that took place between 1960 and 1980, the New Palace was treated with alkaline products to consolidate the building and to isolate it from rising damp through the injections of cement materials.

## 5.2 Salts and Damages

The extreme damage suffered by various decorative pieces is apparent and has already been described elsewhere [10]. In the *Grotten-saal*, damage is predominantly related to salt crystallization within and on the walls. On the surface of the attached minerals and shells, fluffy efflorescences form, while subflorescences sometimes lead to the bursting of decorative pieces and the mortar that holds them in place. In general, the degree of the salinity and, consequently the deterioration, decreases from bottom to top of the walls and horizontally from the windows to the interior of the hall.

Because of the highly valuable decoration of the hall, it was not possible to take many samples for analysis, however, it was possible to determine that the most pervasive salt was sodium sulphate, both present as thenardite [ $\text{Na}_2\text{SO}_4$ ] and as mirabilite [ $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ ]. Gypsum [ $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ] was also present but only to a lower degree, although it is a component of the original mortar.

At the defined fields used for monitoring, where the salts were observed regularly and removed periodically, salt efflorescences occur all year round. When salts crystallize as whiskers, mirabilite could be identified microscopically. When they fell down as a powder, thenardite was identified. In many cases, both minerals were found to be present together.

## 5.3 Room Climate

The evolution of the room climate in the *Grotten-saal* is shown in Fig. 4 where the average daily values for the relative humidity and the temperature are plotted. The *Grotten-saal* is not heated and, therefore, the climate curve is typical for a non-heated room and is strongly influenced by the outdoor climate.

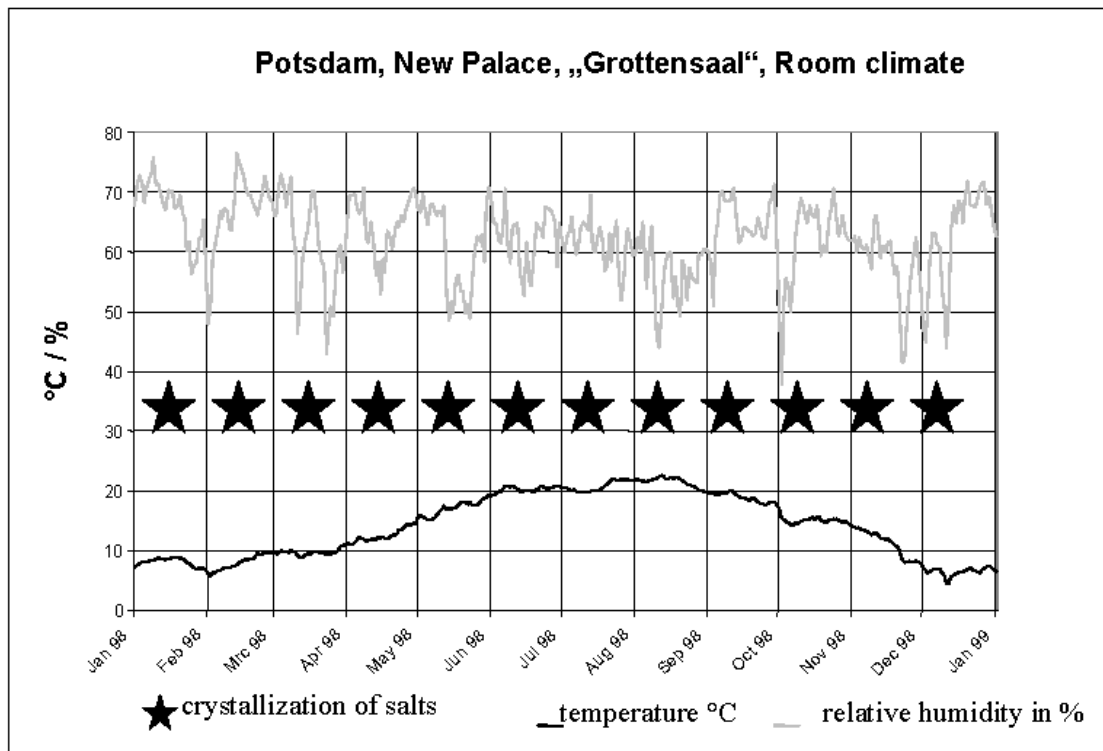
In general, the room temperature varies from about 22 °C to about 6 °C and the relative humidity oscillates between about 30 % and 75 % RH. Changes in temperature and relative humidity develop rather quickly depending on the outdoor climate situation. For example, in Fig. 4 it can be seen that during October 1997 and April 1998 the relative humidity has exceeded or fallen below 65 % about 25 times per month, showing the high oscillation of relative humidity between 60 % and 70 % RH in the *Grotten-saal*.

## 5.4 Interaction of Salts and Room Climate

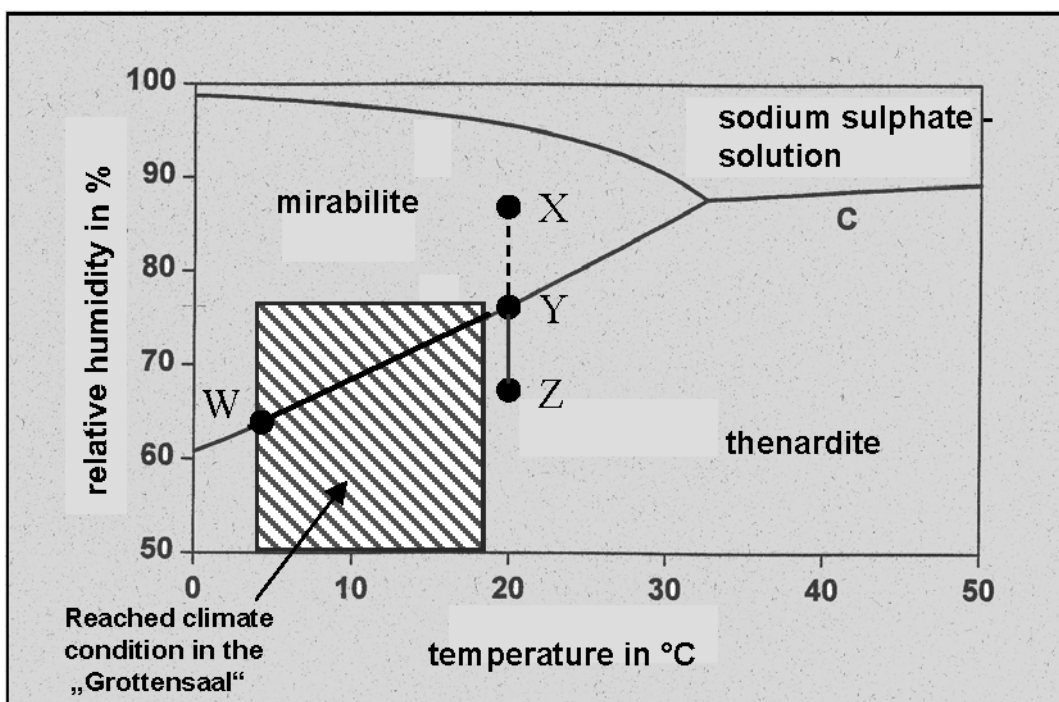
The salts responsible for the damage in the *Grotten-saal* are mirabilite [ $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ ] and thenardite [ $\text{Na}_2\text{SO}_4$ ] and probably originate from the alkaline cement materials which were used for consolidation of the monument. Various studies have shown that sodium sulphate is a reaction product from alkaline building materials [11].

Based on the present investigations, the following weathering processes can be assumed to occur in the „Grotten-saal“: water, from the ground and surface, migrates through the building materials becoming enriched with autochthonous salt-ions, such as  $\text{SO}_4^{2-}$  from the gypsum mortar or  $\text{Na}^+$  from the cement used for consolidation during the 20<sup>th</sup> century. When supersaturation of the solution is reached, mirabilite crystallizes. But mirabilite is affected by the surrounding atmosphere and can dehydrate [12]. In order to further understand the further weathering processes in the „Grotten-saal“, it is essential to examine the system thenardite – mirabilite –  $\text{H}_2\text{O}$  as a function of temperature and relative humidity as shown in Fig. 5.

Sodium sulphate is able to exist in both anhydrous (thenardite) and hydrated (mirabilite) forms. For example, at 20 °C, as an example, there are two stable forms of sodium sulphate: the anhydrous form, thenardite, which is stable below ca. 71 % relative humidity (e.g. in point Z in Fig. 5) and the decahydrate, mirabilite, which is stable between ca. 71 % and 93 % (e.g. in point X in Fig. 5). Above the equilibrium relative humidity of 93 %, however, mira-



**Figure 4:** Evolution of daily average values for temperature and relative humidity in the *Grottensaal* of the New Palace, Potsdam, related to the observed crystallization of salt



**Figure 5:** The system thenardite  $[\text{Na}_2\text{SO}_4]$  – mirabilite  $[\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}]$  –  $\text{H}_2\text{O}$  as a function of temperature and relative humidity. The climate conditions in the *Grottensaal* are marked by those enclosed within the square.



bilite becomes desliquescent absorbing moisture from the air to dissolve into a solution.

As can be seen from Fig. 5, the transformation of mirabilite to thenardite and vice versa takes place at defined relative humidities and temperatures. For example, at 20 °C the transformation takes place at humidity conditions around 76 % RH (point Y in Fig. 5), but at 5 °C the transformation is carried out around 64 % RH (point W in Fig. 5).

The cyclic transformation between the hydrated and anhydrous forms has been proven to be significant for weathering processes. In Fig. 5, where the climate conditions in the *Grottensaal* are also marked, it can be seen that the oscillating temperature and humidity curves frequently pass through the critical values causing the transformation of thenardite and mirabilite, resulting in the observed deterioration.

Thus, in the *Grottensaal* salt weathering is dominated by both newly crystallized salt (mirabilite) as well as by the fact that this salt is capable of de- and re-hydrate in response to the oscillation of the air humidity and temperature. This implies that the climate contributes essentially to the weathering activity of the salts in the „Grottensaal“.

### 5.5 Conclusion

The *Grottensaal* of the New Palace suffers under extreme salt deterioration in particular near the outer walls, where water can enter both from the ground and the outside. The main source of salt ions present can be found in the cement materials used for consolidating the monument. Alkaline salts and the autochthonous sulphates result in the formation of sodium sulphate. Weathering can be attributed to the crystallization of mirabilite and by transformation of mirabilite to thenardite and vice versa which is controlled by the surrounding climate.

The high content of salts and the presence of cement in the walls make it very difficult to minimize damage processes unless it is possible to remove the water influx from the ground and the exterior as soon as possible.

## 6 General Conclusions

The weathering of monuments by salt systems is defined by the compositions of the salt solutions that migrate through the specific building and its climate conditions.

Therefore it is essential to investigate the salt behaviour in relation to the climate it is in, to be able to determine a means of preventing salt damage to monuments. The knowledge about the history of the monument, the materials which were used for consolidation and restoration together with monitoring of damages and salts in combination with climate measurements can lead to the development of an appropriate conservation approach for the monument. It is recommended that before any salt extraction method is undertaken, i.e., poulticing, the option of preventing salt damage through environmental control should be explored. For this purpose, the described method of *salt crystallization monitoring in combination with climate measurements* is recommended.

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