Are you lead historical artefacts safely stored?

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Indoor conditions have the greatest influence on the long-term storage of historical artefacts. The following text summarizes essential knowledge regarding the evaluation of the corrosivity of indoor atmospheres with an emphasis on the effect of the presence of volatile organic acids on corrosion of lead. It provides an overview of the information and resources necessary to decide whether lead storage conditions are safe and concludes by outlining a path to resolution if they are not.

INTRODUCTION

Museums, libraries, galleries, archives, depositories, and other heritage institutions exhibit and preserve many objects of cultural heritage in order to prevent their degradation. From this point of view, a wide range of materials in different conditions meet in them. Determining all possible variants and combinations of materials and conditions that can lead to degradation, and thus damage to stored objects, is a challenging task, requiring a careful evaluation of specific cases [1-3].

A number of factors affect the degradation of historical materials, which can often be at least partially eliminated in the indoor environment. For example, heating can reduce relative humidity, air filtration can limit the penetration of pollutants from the surrounding environment. The type of building, the method of air quality management and the climatic conditions of the given locality nevertheless define to a significant extent the conditions to which collection objects are exposed in the long term. The combination of these factors can create very specific conditions in indoor environments. Stored objects can, for example, be exposed to volatile organic compounds (VOC), which include formaldehyde and volatile organic acids (VOA), commonly represented by acetic and formic acid. In the case of historical and cultural objects, there is a long-term effect of these factors on the materials. Even though the degradation processes can be very slow, after tens to hundreds of years irreversible changes in the materials will occur, leading to the deterioration and loss of the object. This is where preventative conservation comes into play, which aims to minimize degradation either by modifying conditions and the environment, or by protection of the artefact [1-6].

In terms of metallic materials from which objects of historical and cultural heritage can be made, the metal most sensitive to VOC air pollution is **lead** [1, 7, 8, 9].

Lead

Lead has been used since ancient times because its ores were relatively readily available, it has a low melting point, and it is very malleable, which makes it easy to cast and shape. The oldest surviving object dates from between 3000 BC and 2000 BC and was found in Asia Minor. Lead is a relatively abundant material in objects of cultural heritage. It has a large presence in the Czech Republic due to the mining of this metal on our territory - in the Middle Ages, the territory of the Czech Kingdom was one of the main producers of gold and silver in Europe, and lead was often an accompanying metal of silver [7, 10, 11].



Fig. 1. Corrosion damage of the lead seal (Petr d'Aubusson, 27 October 1492, Rhodes)

In terms of corrosion resistance, lead corrodes very slowly under normal atmospheric conditions: the surface of lead gradually loses its luster and a grey-white layer of corrosion products – oxides, hydroxides, and carbonates – forms on it (Fig. 1). Especially in the case of lead oxide, it is a layer of corrosion products with protective properties that slows down further corrosion reactions [1, 7, 9, 12-14].

Because of that, many lead objects have been preserved, often dating back several hundred years [1, 7, 9, 12-14]:

- Stained glass windows – in the Middle Ages, windows were made from glass sheets bound together with molten lead (Fig. 2).
- Objects of daily use – lead was used in the production of containers, furniture fittings, figurines, toys, etc.
- Construction – lead was used in the production of pipes, garden and architectural elements (roofs, etc.).
- Lead was used to make seals (bullae), medals, coins and jewellery.
- The invention of letterpress brought about the preparation of alloys of lead and antimony, which excel in good casting properties, and thanks to this, very precise die castings can be done.
- Organ pipes were made from lead alloys (Fig. 3).
- Technical monuments – lead was used, for example, as a seal and for the production of thermal fuses.

In the presence of vapors of organic acids, the most common of which are formic acid (HCOOH) and acetic acid (CH_3COOH) in the environment of depositories and museums, active corrosion of lead occurs. Bulky incoherent soluble salts, such as lead formate and lead acetate, are formed on its surface, which then gradually change to more stable carbonates, cerussite and hydrocerussite in the presence of atmospheric carbon dioxide [1, 7, 9, 10, 15-18].

Indoor atmospheres and VOC

Indoor atmospheres generally present milder conditions in terms of corrosivity compared to outdoor conditions (Fig. 4-6). This is due to more stable levels of humidity and temperature without significant fluctuations, a lower concentration of dust particles and a lower concentration of typical gaseous pollutants, i.e. nitrogen oxides and sulfur dioxide. On the other hand, the content of some specific pollutants can be higher indoors, which poses a potential risk in terms of corrosion damage. Typical examples are the aforementioned volatile organic compounds, especially acids, which cause corrosion not only of lead objects, but also of copper, zinc, nickel or iron [1-6 19, 20].

There can be many sources of volatile organic compounds indoors:

- furniture made of plywood and hard wood, especially oak [3, 12, 21-25],
- cleaning agents [12, 21, 24],
- adhesives and sealants [12, 21, 24],
- degradation products, e. g. paper [2, 4],
- unsuitable packaging material, cardboard [26].

The rate of chemical interaction of VOCs with metals is dependent on other conditions, especially temperature and relative humidity. These parameters influence not only the rate of corrosion reactions, but also the course of degradation of VOC-releasing materials, and thus the concentration of VOCs in the environment. As a result, the corrosivity of the indoor environment can change over the course of the year. Another specific situation is the crypto climate conditions, i.e. a space that, in terms of atmospheric conditions, has difficulty communicating with the surroundings. These are mainly archive boxes, display cases and storage spaces. The composition of atmosphere and climatic conditions in a crypto climate can be significantly different from



Fig. 2. Detail of a stained glass window from the monastery in Osek, ca 1350



Fig. 3. Organ with lead pipes in the church of All Saints in Heřmánkovice

the surrounding indoor atmosphere [1-3, 20, 25, 27]. The effects of climatic parameters (temperature and relative humidity) and atmospheric pollutants are interconnected, and it can be misleading to rely on each of them individually when determining the corrosivity of the atmosphere. Therefore, it is more appropriate to determine the corrosivity directly by exposure of metal coupons and to use their corrosion rate for evaluation [1-3, 20, 25, 27-30].

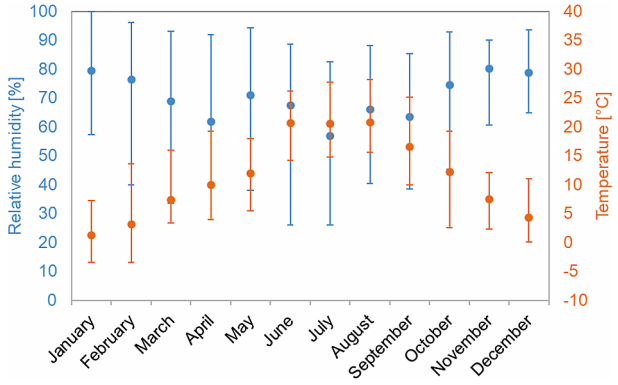


Fig. 4. Annual record of outdoor temperature and relative humidity

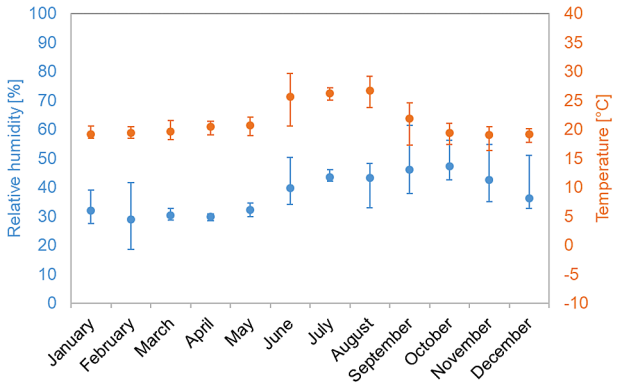


Fig. 5. Annual record of temperature and relative humidity in a depository without climate control

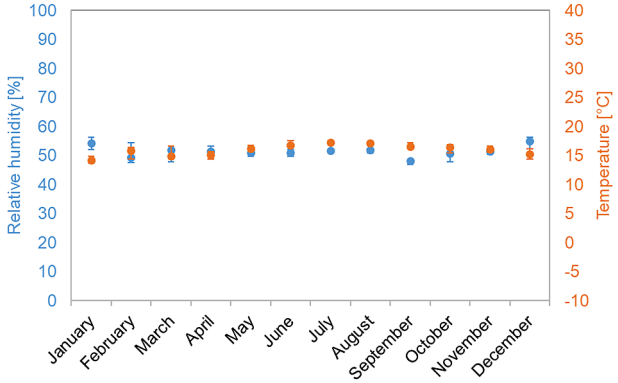


Fig. 6. Annual record of temperature and relative humidity in a climate-controlled depository

However, the measurement of climatic parameters, namely temperature, relative humidity and content of volatile organic acids, can indicate whether the internal conditions represent a potential danger for stored lead objects and it is necessary to determine the corrosivity of the internal atmosphere. Regular monitoring of climatic parameters also makes it possible to recognize in time whether there have been changes in conditions affecting corrosivity. Last but not least, the connection of the results of the measurement of climatic parameters with the determination of the corrosion rate by exposure of the metal coupons enables to understand the causes of possibly increased corrosivity [1-3, 20, 27-30].

Characterization of corrosivity of indoor atmospheres

Measurement of temperature and relative humidity

Information on temperature and relative humidity should be recorded regularly using electronic recorders. The recording interval depends on the purpose of the measurement – if it is a regular monitoring of internal conditions, it is sufficient to record the values every 4 hours, which enables the calculation of the daily average. If the measurement accompanies the determination of the corrosivity of the internal atmosphere, it is advisable to record at an interval of one hour for the entire exposure time of the corrosion coupons. The memory capacity of commercially available recorders is usually sufficient for this recording frequency. Relative humidity and temperature data logger should be calibrated before the measurement (see box comment). If such a data logger has an autonomous power supply (battery), care must be taken to ensure that the battery life is sufficient for the entire measurement period, and the functionality of the recorder must be checked once a month. When placing data loggers, it should be kept in mind that temperature and relative humidity can vary within the same room.

Verification of the humidity sensor:

Before monitoring, it is recommended to have the equipment used calibrated by the manufacturer, or at least to verify the correctness of the relative humidity measurement by exposure in a closed box above the level of a saturated salt solution, e.g., NaCl, above which the equilibrium relative humidity is 75% stably for common temperatures from 0 to 50 °C [31]. For verification, the relative humidity inside the box must be allowed to stabilize for approximately 24 hours.

Commonly available relative humidity data loggers, whose function is based on capacitive measurement, are usually reliable at relative humidities below 95%.

This graphs compare the annual development of relative humidity and temperature in the exterior (Fig. 4), in the interior (Fig. 5) and in the depository with maintained stable temperature and relative humidity (Fig. 6).

Both changes in the differences of monthly values and changes in the dispersion of values within individual months are evident. Generally, in addition to high relative humidity, large fluctuations in temperature and relative humidity are dangerous for stored objects – specifically, in the case of metal objects, condensation and the subsequent initiation of corrosion can occur in case of significant variations.

Measurement of the volatile organic acids (VOA) content

There is a wide range of tools for monitoring the concentration of pollutants in the atmosphere, which can affect the corrosivity of the environment – from simple indicating devices to complex and expensive instrumental analysis techniques. The preferred are affordable techniques that allow immediate and easy interpretation of results. When choosing them, an important aspect is the detection limit for the monitored pollutant.

For the basic classification of the presence of volatile organic acids, the so-called **A-D Strips®** are available. This system is based on the colour change of paper strips impregnated with an acid-base indicator and the evaluation of the colour change after a certain period of exposure in the monitored environment. The primary purpose of their use is to detect the acetate syndrome of film substrates made from cellulose acetate. The A-D Strips® application procedure is well described in the model experiment by Technical Museum in Brno [32]. A colour change after the recommended exposure time will occur if the VOA content in the atmosphere is greater than 1 ppm. Since corrosion damage to lead objects occurs at lower concentrations of VOA [9], the sensitivity of this detection system is insufficient in terms of assessing the corrosivity of the atmosphere.

Colour changes of the indicator are also used by **GASTEC detection tubes** (GASTEC Corp.). They are available in two variants:

Passive dosimetric tubes GASTEC 81D with detection range for acetic acid 5-100 ppm.hours. The exposure time recommended by the manufacturer is up to 10 hours, however, it has been verified that they can be used even after 30 days of exposure. This moves the detection limit for volatile organic acids up to ppb units. After 30 days, however, the border of the colour change of the indicator becomes less clear, so it is recommended to expose the passive dosimetric tube for about 7 days. This makes it possible to detect VOA concentrations in the order of tens of ppb, which is sufficient. Passive dosimetric tubes are suitable for longer-term determination of VOA content with higher sensitivity.

GASTEC 81L *gas pipettes* with a detection range for acid from 0.125 to 23 ppm in air drawn into the detection tube using a GASTEC pump. Gas pipettes are suitable for preliminary indicative determination of the immediate presence of VOA by sucking analysed air using a pump from a closed space (archive box, showcase, drawer, etc.). Because gas pipettes provide information on VOA content immediately, including relatively high concentrations, they can be used to optimize the exposure time of passive dosimetry tubes, so as not to exceed the detection limit of the tube or lose the clarity of the colour indication due to long exposure times.

Passive samplers described in the standard EN ISO 11844-3 [30] are suitable for reproducible measurement of VOA content with a sufficiently low detection limit. Volatile organic acids are captured by the sorption medium and after exposure the VOA content is calculated from the anion concentration determined by ion chromatography. For a successful measurement including correct evaluation, it is important that the passive samplers are installed within 7 days of their manufacture, the exposure time must be shorter than 40 days, and the evaluation must take place within 7 days of the end of the exposure. Exceeding these limits may result in underestimation of VOA content. Additionally, the passive samplers must be stored before and after the exposure in the refrigerator.

SPME (Solid phase microextraction) sorption fibers will provide detailed information about the presence of volatile compounds in the monitored area. It is a qualitative technique and requires the participation of a specialized workplace (e.g., UCT Prague) in the evaluation of exposed fibers with a gas chromatograph with a mass spectrometer.

Determination of corrosivity by corrosion rate measurement

The procedure to determine the corrosivity s of indoor atmospheres towards metals is defined by three parts of the standard EN ISO 11844 *Corrosion of metals and alloys — Classification of low corrosivity of indoor atmospheres* [28-30]. They introduce classes of corrosivity IC1 to IC5 and describe methods for determining the corrosion rate. These are based on the evaluation of mass gain or mass loss of corrosion coupons, evaluation of the amount of corrosion products by galvanostatic reduction, measurement of corrosion loss using a resistometric sensor, or measurement of weight gain of a quartz crystal with a thin metal coating. The resulting corrosion rate then determines the classification of the given environment into one of the five corrosivity classes defined by the standard for silver, copper, steel, zinc, and now also lead (see the text in the box).

Determination of corrosion rate by mass change measurement, unlike the other mentioned methods, does not require special laboratory equipment, therefore attention will be focused on this technique. At the same

time, however, it is necessary to keep in mind that the result of the measurement is an average corrosion rate during the exposure time of metal coupons. To monitor changes in corrosion rate during the measurement, it is necessary to choose a technique enabling continuous measurement, for example, electrical resistance measurement.

Lead as a VOA detector:

Lead is specifically sensitive to the presence of volatile organic acids, especially acetic acid. This feature can be advantageously used to indicate presence of VOA in the indoor environment. Therefore, in 2021, based on the results of the NAKI II project "*Methodology of corrosivity classification of indoor environments for lead collection objects*" (DG-18P02OVV050), lead was added as a reference metal in the standard EN ISO 11844-1 [28].

Corrosion rate by measuring changes in mass can be determined based on the mass increase of the corrosion coupon, which is caused by the conversion of the metal to corrosion products on its surface, or from the mass loss after the corrosion products have been removed from the surface. Undoubtedly, the simpler procedure for implementation is the determination of weight gain, but this procedure is more difficult for the interpretation of the results. To convert the mass gain into a corrosion rate, it is necessary to know the composition of the corrosion products, which is not easily available information.

The method of determining the corrosion rate by the mass loss measurement works with metal coupons of silver, copper, zinc, carbon steel and lead. The coupons must be of sufficient purity, of suitable dimensions regarding weighing, and the surface of the coupons must be prepared and cleaned according to a standardized procedure. The details of sample preparation are described in the standards EN ISO 11844-2, EN ISO 8407 and the already mentioned methodology, in which individual aspects are discussed, including the issue of weighing and evaluating exposed coupons [28-30, 33]. Most often, the coupons are displayed by hanging them in a vertical position on a stand adapted for that purpose (Fig. 7).

Corrosion coupons should be exposed for 12 months in such a way that all changes in parameters that occur during the year are covered (changes in temperature, relative humidity and atmospheric pollution during the seasons in external conditions, temperature fluctuations during the heating season, etc.). In justified cases, it is possible to shorten the exposure period to three months, e.g. if it is required to investigate the change in aggressiveness during the year or in specific periods, for example holding exhibitions and the resulting increased attendance. The exposure period must then be taken into account in the corrosion rate calculation and the exposure period must always be indicated if it is other than 12 months. A longer exposure time is not desirable, because, with the constant corrosivity of the environment, the corrosion rate gradually decreases, which can lead to an underestimation of the corrosivity class.

After the exposure, the final weight of the corrosion coupons will be determined after the corrosion products have been removed using a suitable pickling solution. Recommended solutions are for each metal are listed

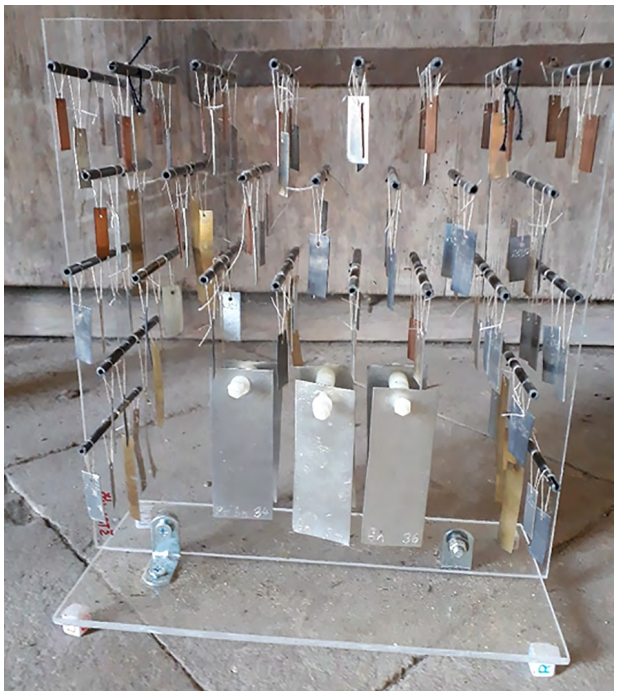


Fig. 7. Placement of the corrosion coupons

Tab. 1. Classification of corrosivity of indoor atmospheres for silver and copper [28]

Corrosivity category	Corrosion rate r_{corr} (mg m^{-2} a^{-1})	
	Ag	Cu
IC1	$r_{corr} \leq 170$	$r_{corr} \leq 50$
IC2	$170 < r_{corr} \leq 670$	$50 < r_{corr} \leq 200$
IC3	$670 < r_{corr} \leq 3000$	$200 < r_{corr} \leq 900$
IC4	$3000 < r_{corr} \leq 6700$	$900 < r_{corr} \leq 2000$
IC5	$6700 < r_{corr} \leq 16700$	$2000 < r_{corr} \leq 5000$

Tab. 2. Classification of corrosivity of indoor atmospheres containing VOA for lead

Corrosivity category		Corrosion rate r_{corr} of Pb (mg m^{-2} a^{-1})
Pb1	Very low indoor corrosivity	$r_{corr} \leq 1300$
Pb2	Low indoor corrosivity	$1300 < r_{corr} \leq 2000$
Pb3	Medium indoor corrosivity	$2000 < r_{corr} \leq 3000$
Pb4	High indoor corrosivity	$3000 < r_{corr} \leq 6000$
Pb5	Very high indoor corrosivity	$6000 < r_{corr} \leq 20000$

in the standard [33]. For lead either a 1% HCl solution or a Rochelle salt solution (15 g $KNaC_4H_4O_6 \cdot 4H_2O$ + + 5 g NaOH in 100 ml water) at laboratory temperature gives best results [34]. The recommended procedure for removing corrosion products is interval pickling according to the EN ISO 8407 standard.

Based on the corrosion rates, the indoor conditions can be classified into one of five classes of corrosivity. Tab. 1 lists the classification for silver and copper. In the case of lead, the current version of the standard defines classes for low corrosivity, which is unusual in archives, libraries, and depositories, that is, in environments with sources of volatile organic acids. For such indoor environments containing volatile organic acids, a scale with corrosion rate intervals listed in Tab. 2 was proposed in the methodology [20].

Are you lead historical artefacts safely stored?

The preceding paragraphs present a set of findings and procedures for answering the central question. But what do we do if we discover that our lead historical artefacts are not safely stored? The following diagram (Fig. 8) should provide a basic signpost in which directions to go when dealing with the high corrosivity of the indoor atmosphere.

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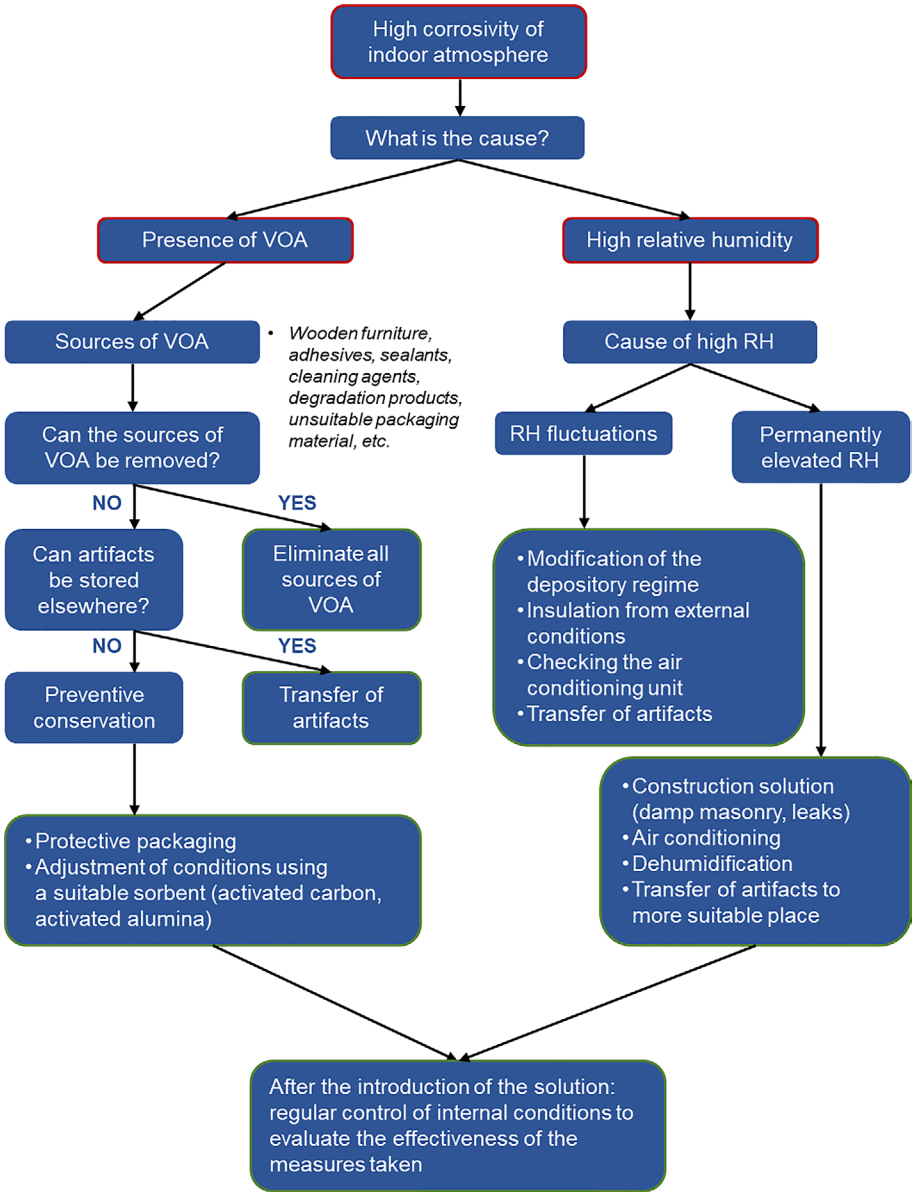


Fig. 8. How to deal with high corrosivity of the indoor atmosphere

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