

A CORROSION INHIBITOR TEST FOR COPPER-BASED ARTIFACTS

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Summary—A new quantitative corrosion testing procedure for copper and copper alloy corrosion inhibitors was developed, with the aim of calculating corrosion protection in percent for each inhibitor on trial. 2-amino-pyrimidine, 2-amino-5-mercapto-1,3,4-thiadiazole, benzotriazole, 5,6-dimethylbenzimidazole, 2-mercapto-benzimidazole, 2-mercaptobenzoxazole, 2-mercaptopyrimidine and 2-mercaptobenzo-thiazole were tested for their effectiveness in preventing the conversion of nantokite (copper(I) chloride, CuCl) into paratacamite (basic copper(II) chloride, $\text{CuC}_2\text{3Cu(OH)}_2$) which causes major damage in archaeological copper artifacts.

Introduction

The aim of this work was to evaluate industrial copper corrosion inhibitors using a new quantitative corrosion testing procedure. The effectiveness of 2-aminopyrimidine (AP), 2-amino-5-mercapto-1,3,4-thiadiazole (AMT), benzotriazole (BTA), 5,6-dimethylbenzimidazole (DB), 2-mercaptobenzimidazole (MBI), 2-mercaptobenzoxazole (MBO), 2-mercaptopyrimidine (MP) and 2-mercaptobenzo-thiazole (MBT) was established.

Copper and copper alloy archaeological artifacts can frequently be damaged or destroyed by cyclic copper chloride corrosion. Artifacts stored at a relative humidity (RH) between 42 and 46% are usually stable [11] but, if the RH is higher, further corrosion can occur. The aim of applying corrosion inhibiting compounds to material containing copper chloride is to prevent copper(I) chloride (nantokite) converting into basic copper(II) chloride (atacamite, paratacamite, botallackite).

In the past, new conservation treatments were applied directly to archaeological material. This is now considered inappropriate, so a standardised testing procedure must be established.

Corrosion testing using archaeological copper and copper alloys, and the interpretation of results, have several disadvantages:

- the treatment might damage the object
 - the treatment is probably irreversible
 - the long-term effects are unknown
 - the corrosion products vary
 - the amount of corrosion varies greatly
 - difference in porosity of corrosion products alloying constituents might influence the treatment
 - the size and shape of the artifact are different
- The new corrosion test for copper and copper

alloys should give conservators a tool to evaluate new treatments keeping variables to a minimum and without the use of expensive equipment, usually not available in small conservation laboratories. The test also focuses on the reproducibility of the results, allowing comparison between experimental work.

A corrosion test procedure was developed to simulate the stratigraphy of copper chloride corrosion frequently encountered on artifacts containing copper chloride corrosion. Pre-corroded test coupons are the basis for assessing the performance of corrosion inhibiting compounds in an elevated RH at ambient temperature. The test applies, wherever possible, existing standards that can be found in the International Standards Organisation (ISO), British Standards Institution (BSI), Deutsche Industrie Norm (DIN) and American Society for testing Materials (ASTM).

The choice of new corrosion inhibitors

Selection mechanism

Only benzotriazole (BTA) has been widely accepted in the stabilisation of copper and copper alloy artifacts. Madsen [21] proposed BTA for archaeological conservation based on the vast research done for industrial purposes. Due to its perceived advantages and satisfactory results, BTA has been the predominant corrosion inhibitor used in the conservation of copper and copper alloys. This has seemingly led to the exclusion of research on other compounds for copper in archaeological conservation. Recently, another corrosion inhibitor was suggested for copper corrosion inhibition in conservation: 2-amino-5-mercapto-1,3,4-thiadiazole (AMT) [31] however, AMT has not been accepted generally for use in

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archaeological conservation, due to lack of information available, both on industrial applications and in the field of archaeological conservation.

To search for other copper corrosion inhibitors, a survey of current chemical and engineering literature was undertaken. Copper corrosion inhibitors tested in industry were recommended for specific tasks in either an industrial or a household environment. Inhibitors were usually tested on uncorroded metallic copper and copper alloys, used in modern installations. Inhibitors were applied to metallic, polished and, in many cases, oxide-free copper surfaces. No study published dealt with samples comparable to the heterogeneous corrosion layers found on archaeological artifacts from burial environments. In no case was a ternary alloy such as copper-tin-lead tested, one of the various copper alloys found in archaeological sites. These findings made it obvious that published test results given in the industrial literature were of value only for preliminary selection of compounds for future use in archaeological conservation. The published data gave an indication of what to expect in further trials in conservation.

A re-evaluation of published literature from a conservation point of view was necessary to identify new inhibitors specifically related to objectives in archaeological conservation. There are several industry-based comparative studies in the area of copper corrosion inhibitors, such as Musiani et al. [4], Lewis [5], Horner et al. [6-10], and others. Many publications cover corrosion protection in water flow systems such as boilers, radiators, chemical containers and seawater systems. Industrial inhibitors are all subject to much harsher conditions than those to which an archaeological artifact would be exposed. Such industrial environments have little direct relevance to conditions a treated archaeological artifact would generally experience in a storage area or a showcase.

The studies consulted were screened for specific features such as applications and types of compound potentially suitable for archaeological metallic objects. To be appropriate as a copper corrosion inhibitor for conservation, the compound should satisfy various requirements arising from the object, the environment and the conservator. These requirements include the following:

- (1) The corrosion inhibitor should form a complex with copper, resulting in a polymeric layer. These polymer formations are considered to have the most suitable structure, because they develop a barrier between the metal substrate and its environment. The polymer structure should be thick, dense and without voids in order to stop further oxidation of metal.
- (2) The inhibitor should adsorb or chemically bond to the substrate.
- (3) The inhibitor-copper polymer film formed should not be soluble in water or organic solvents, since an object is often rinsed with alcohol or acetone (propanone) after treatment to remove excess inhibitor.
- (4) The effective range of a corrosion inhibitor should be as low as pH 2. The surface of corroded copper has areas, such as corrosion pits, where significantly low pH was detected. Copper(I) chloride can have a pH 3.5-4 [11]. It is suggested that the acidity in chloride pits is even lower. Therefore the inhibitor should be effective over the pH range between 2 and 8.
- (5) The compound should preferably act as an anodic inhibitor. During pitting corrosion the anodic areas in the central area of the pits are actively corroding, while the surrounding surface is cathodic [11]. The best inhibitors would react in anodic and cathodic areas. Considering the case of an actively corroding copper artifact, the metallic parts act as an anode and the corrosion layers mainly act as a cathode. The insides of corrosion pits are also found to be anodic. Due to the heterogeneity of an alloy and its corrosion products, the boundaries of anodic and cathodic parts in a corroded object are usually not clear or distinct [12].
- (6) The visual appearance of the patina should not be altered by the application of the inhibitor. As mentioned above, some of the previous treatments applied in conservation caused the disfiguration or discoloration of the outer corrosion layers. Benzotriazole, for example, was found to darken the appearance of the corrosion products, especially in repeated application, as is sometimes required. AMT was shown to cause some darkening of metallic copper when applied in industrial methylated spirits [13].
- (7) The inhibitor should not be hazardous or harmful to the user. In many instances laboratories are not equipped with air extraction systems, or the inhibitor has to be applied immediately post excavation in the field. Benzotriazole is suspected of being carcinogenic [14].
- (8) Metallic based inhibitors should be avoided. In many instances they are powerful inhibitors. Titanates have been used on iron and chromates, and silver oxide on copper. The latter is frequently used as a treatment for 'bronze disease' in conservation.

The metallic traces left in the corrosion products and on the surface may interfere with the analytical results of trace element studies.

- (9) The solubility of the compound in water or alcohol is another important factor. The solvent in which it is applied should be inexpensive, non-toxic and not hazardous. It would be ideal to be able to control the viscosity of the inhibitor formulation in order to influence penetration and spread of solutions.
- (10) The inhibitor should be readily available in a pure form through chemical suppliers. However, it is apparent that in many cases inhibitors are produced on a trial basis. Many conservation laboratories do not have the facilities to refine mixtures of compounds.
- (11) The price of the inhibitor is a relative measure, but it should still be economical for large-scale applications. Some inhibitors are active in the low concentration of 10-IM.

The industrial literature search identified three main types of inhibitor which were found to be very effective in the inhibition of copper corrosion. These inhibitors contain sulphur, nitrogen, or a combination of both. Corrosion inhibition efficiency was found to be as follows: sulphur-containing > nitrogen-containing > oxygen-containing [15].

Copper corrosion inhibitors chosen for further testing

The literature cited covered a wide range of inhibitors. The research into these publications aimed at finding inhibitors suitable for museum environments. The following inconsistencies with conservation aims were encountered:

- Industrial tests were performed on reagent grade copper or alloys rarely comparable to

- those expected to occur in an archaeological context.
- Archaeological artifacts are covered in oxide layers whereas the metal surfaces used in industrial experiments are free of contamination; the surfaces were cleaned since oxides would have interfered with test results.
- In many cases, the pH range of inhibitor solutions was not indicated or the application was in a narrow pH range. Frequently, the pH was buffered to allow evaluation in near neutral solutions, In the case of archaeological copper and copper alloys, the pH range over which the inhibitor is effective should be as low as 2 since it is thought that chloride corrosion pits develop at a pH between 3-5 and 4 [1].
- Only in a few instances was it mentioned if an inhibitor formed an anodic, cathodic, or both types of corrosion protection.
- Inhibitors obviously not complying with the requirements listed above for archaeological metals were not considered.

As a result of the literature survey, a list of inhibitors was compiled. According to the conservation requirements outlined earlier, the six most closely matching corrosion inhibitors were selected (see Table 1) and compared in the following experimental work against BTA and AMT, the two inhibitors known to conservation.

Experimental procedures

Corrosion testing procedure

To test corrosion inhibiting treatments, such as corrosion inhibitors, for copper and copper alloy artifacts containing active copper chloride corrosion, the following procedure is recommended:

Table I

	<i>Formula</i>	<i>Short name</i>
Nitrogen-based inhibitors		
benzotriazole	C ₆ H ₅ N ₃	BTA
2-aminopyrimidine	C ₄ H ₅ N ₃	AP
5,6-dimethylbenzimidazole	C ₉ H ₁₀ N ₂	DB
Sulphur-based inhibitors		
2-amino-5-mercapto- 1,3,4-thiadiazole	C ₂ H ₃ N ₃ S ₂	AMT
2-mercaptopyrimidine	C ₄ H ₄ N ₂ S	MP
2-mercaptobenzoxazole	C ₇ H ₅ NOS	MBO
2-mercaptobenzothiazole	C ₇ H ₅ NS ₂	MBT
2-mercaptobenzimidazole	C ₇ H ₆ N ₂ S	MBI

- (1) Air-abrading of copper (99.9%) coupons (20 x 50 x 1mm) with 47 μ m glass beads, or sanding with 600 Grade sandpaper (ASTM G1:90), five coupons for each inhibitor.
- (2) Degreasing in an ultrasonic bath in 100ml acetone.
- (3) Drying for 5 min. at 50°C under infrared lamp, and 10 min. cooled in a silica-gel buffered polyethene box.
- (4) Weighing to ± 0.01 mg.
- (5) Immersion in 25ml of a 1M reagent grade solution of cupric chloride in deionized water, one day at ambient temperature.
- (6) Rinsing in deionized water, 3 x 20 min. in 100ml.
- (7) Quick drying in 200ml ethanol.
- (8) Drying for 5 min. at 50°C under infrared lamp.
- (9) Exposure to 105°C in an oven for 60 min.
- (10) 10 min. cooling in a silica-gel buffered polyethene box.
- (11) Weighing to ± 0.01 mg.
- (12) Immersion in 25ml ethanol containing 0.1M corrosion inhibitor, 24 hours at ambient temperature, only partially covered so oxygen can enter,
- (13) Drying for 5 min. at 50°C under infrared lamp.
- (14) 24 hours desiccation in a silica-gel buffered polyethene box.
- (15) Exposure to ~100% RH for 24 hours, at ambient temperature.
- (16) Drying for 5 min. at 50°C under infrared lamp and 60 min. at ambient RH and temperature.
- (17) Weighing to ± 0.01 mg.

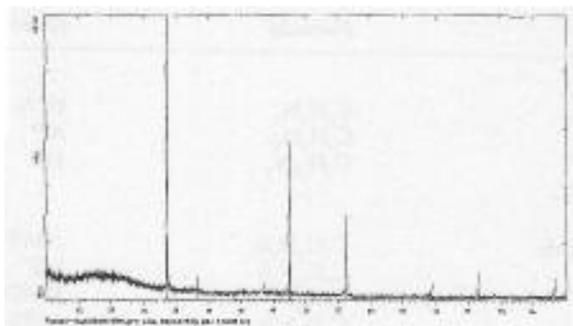


Figure 1 Nantokite.

Sample preparation

For the test listed above, 99.9% pure copper sheet was used. The sheet as provided was rolled to a thickness of 1mm. A BSI (British Standards Institution) standard proposes a sample size not smaller than 25cm² [161], but this sample size was considered too large. Samples of this size could not be accommodated in the humidity chamber available during the experiment.

The material was cut on a guillotine to the size of 20 x 50 x 1mm. This is the coupon size Angelucci *et al.* [17] used in their experiments. This sample size also enables the treatment of greater numbers of coupons in one experiment, helping the assessment of reproducibility of experimental results.

Steps used for the preparation of the coupons must be monitored carefully to minimize variations in the corrosion of the surface. Each step of the surface preparation had to be chosen and observed carefully as slight changes could affect the resulting corrosion data. Establishing reasons for changes in corrosion rates is difficult once corrosion has occurred, since so many complex interacting factors have to be taken into consideration.

Analysis of the corrosion products on the test samples

To check the compositions and the structure of the corrosion layers present on the coupons, X-ray powder diffraction was used. One sample was taken after corrosion in the 1M solution of copper(II) chloride, drying for 5 min. at 50°C under infrared lamp, exposure to 105°C in an oven for 30 min. and one hour cooling in a silica-gel buffered polyethene box. Another sample was taken from the same specimen after the surface was exposed for 24 hours to 95% relative humidity. The XRD analysis was done on a Siemens X-ray diffractometer D5000 in the Chemistry Department of University College London. The sample taken after the copper(II) chloride corrosion clearly shows the presence of nantokite (Figure 1). The sample taken after the exposure of the nantokite covered surface to ~100% RH for 24 hours was clearly converted to and identified as paratacamite (Figure 2).

A metallographic section was taken to investigate the corrosion layers, before and after exposure of the nantokite layer to ~100% RH. The section taken of the pre-corroded coupon showed a layer of 20-25 μ m of nantokite on top of the copper substrate (Figure 3). This was covered with a 5-10 μ m layer of what seemed to be red cuprite (copper(I) oxide, Cu₂O). The coupon covered in paratacamite showed a 10-25 μ m layer of what seemed to be cuprite on top of the copper substrate, and a

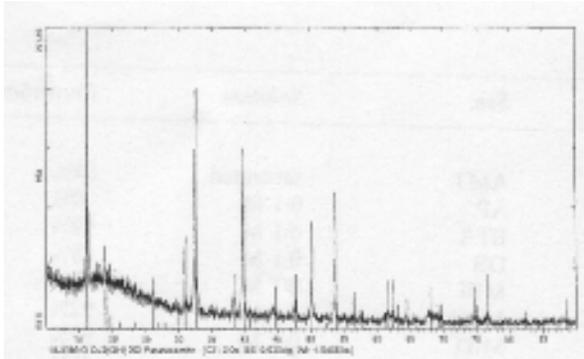


Figure 2 Paratacamite.

160-175µm layer of paratacamite covering the cuprite (Figure 4). The XRD analysis previously undertaken did not detect the cuprite visible in the nantokite- or the paratacamite-covered coupon samples. This could be due to the much smaller amount of cuprite.

Calculation of the corrosion inhibition

To evaluate the effectiveness of a corrosion inhibitor, Trabanelli [18] proposes that the percentage of inhibition (P.I.) can be calculated from the formula below:

$$P.I. = \frac{\text{uninhibited corrosion rate} - \text{inhibited corrosion rate}}{\text{uninhibited corrosion rate}} \times 100$$

The uninhibited corrosion rate is the weight of corrosion product after a unit time on a blank specimen (a nantokite-covered sample not treated with a corrosion inhibiting compound exposed for 24 hours to ~100% RH at ambient temperature). The inhibited corrosion rate is the comparable ratio for the corrosion inhibited coupon (after a 24 hour exposure to 95% RH at ambient temperature). This formula was used to establish the corrosion protection rate of inhibited

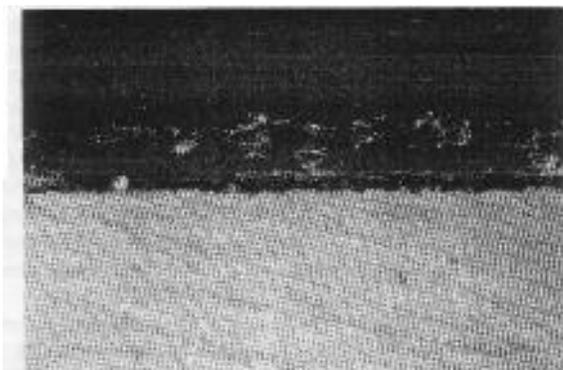


Figure 3 Cross-section of coupon after 24 hours in copper(II) chloride, covered in nantokite.

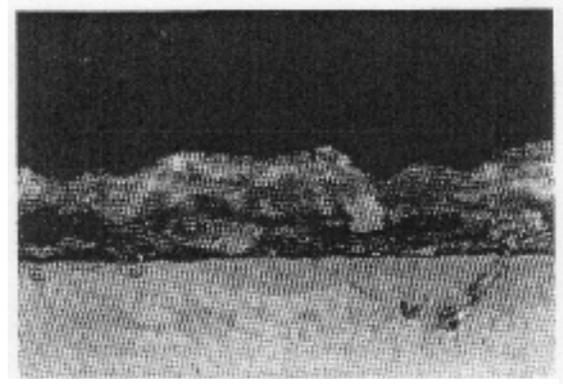


Figure 4 Cross-section of coupon after 24 hours at ~100% RH, covered in paratacamite.

coupons in the following experiment. Zero inhibition equates with complete corrosion equal to the blank specimen.

The selected corrosion inhibitors were tested for their corrosion inhibiting effectiveness according to the procedure outlined previously, and the corrosion rate was calculated based on the formula stated above.

Results and discussion

Visual appearance

After 24 hours immersion in BTA and DB the inhibitor solutions were discoloured due to a complexing of the corrosion products in the inhibitor solution. Both solutions had a dark green to black appearance. The other inhibitors did not cause a dramatic discoloration of the inhibitor solutions after a 24 hour treatment of the corroded coupons. The complexing of nantokite in the BTA and DB solution could be an indication for probable use as a chloride-removing agent on artifacts; however, it is not clear if there is a preferential complexing of copper chlorides. There is also a possibility that other copper corrosion products such as malachite or cuprite might be complexed into the inhibitor solution, damaging non-chloride-containing parts of the corroded surface. The coupons treated with MBI were covered in a white coating, probably a MBI-Cu complex. The MBI treatment is considered a failure since artifacts would be disfigured by such a white coating.

The coupons thought to have the smallest changes to their surface appearance were AP > MBO > AMT > MBT > MP. Overall, it was apparent that the mercapto compounds caused a slight yellow discoloration, probably due to the sulphur present. BTA, DB and MBI were considered to be visually

Table 2

<i>Corrosion inhibitor</i>	<i>Syn.</i>	<i>Solution</i>	<i>Corrosion inhibition</i>
2-amino-5-mercapto- 1,3,4 thiadiazole	AMT	saturated	84%
2-aminopyrimidine	AP	0.1 M	0%
benzotriazole	BTA	0.1 M	99%
5,6-dimethylbenzimidazole	DB	0.1 M	88%
2-mercaptobenzimidazole	MBI	0.1 M	98%
2-mercaptobenzoxazole	MBO	0.1 M	92%
2-mercaptobenzothiazole	MBT	0.1 M	97%
2-mercaptopyrimidine	MP	saturated	38%

unacceptable due to the discoloration of the surface after the 24 hour inhibitor treatment.

Corrosion inhibitor effectiveness in percent

According to the formula outlined above, the corrosion protection for the chosen inhibitors in percent was calculated (see Table 2).

AP was not effective in preventing the conversion of nantokite to paratacamite. AMT and MP had a low solubility in ethanol a 0.1M solution could not be prepared-so they were applied as a saturated solution. It was determined that AMT has a solubility of $\sim 9\text{g.l}^{-1}$ in ethanol at room temperature and MP had a lower solubility of $\sim 1\text{g.l}^{-1}$ in ethanol at room temperature. Even in a saturated solution, MP was only 38% effective and AMT only 94%. The BTA (99%) treated coupon had a dark black-green surface after the inhibitor treatment. This might suggest a darkening of artifacts after a BTA treatment. The DB treatment removed most of the primary chloride-containing corrosion layer and left large areas of the surface etched down to the copper substrate, and was only 88% effective. MBI (98%) was slightly less effective than BTA, despite the coverage with an unsightly white complex layer. MBO yellowed and darkened the surface of the coupons slightly after corrosion inhibition and was only 92% effective. MBT was a good corrosion inhibitor by protecting the coupons effectively to 97%; however, it yellowed the surface. MP was only 38% effective and was considered a failure.

The experiment indicates that sulphur-containing compounds are not necessarily more effective than nitrogen-containing corrosion inhibitors. BTA has shown to be the most effective corrosion inhibitor in comparison with the newly selected inhibitors. In conservation BTA is normally applied as a 3% by weight solution as recommended by Madsen [191] this is equivalent to $\sim 0.25\text{M.l}^{-1}$. This experiment indicates that a 0.1M BTA solution is more effective than AMT and

other inhibitors. Only MBI (98%) and MBT (97%) were considered as having good corrosion-inhibiting properties, but the white surface finish of MBI makes it unsuitable for conservation of archaeological artifacts. The yellowing of the coupons due to MBT is also unacceptable.

Conclusion

The testing procedure using copper coupons covered with a layer of nantokite and cuprite to assess copper corrosion inhibitors was found to be a successful way to evaluate quantitatively newly selected compounds for arresting the cycle of copper chloride corrosion. The corrosion inhibitor effectiveness of the selected compounds was rated BTA > MBI > MBT > MBO > DB > AMT > MP > AP. The only new inhibitor having a relatively good performance was MBT, only slightly less effective than BTA. MBT did, however, cause a yellowing of the coupons, altering the appearance of the corroded surface dramatically. None of the new corrosion inhibitors can be recommended, due to either ineffectiveness, poor visual appearance, and/or causing damage to the chloride-corroded coupons. These drawbacks of the treatment could be observed on artificially corroded copper coupons, rendering it unnecessary to apply new compounds initially to batches of archaeological material. This accelerated corrosion test is a preliminary screening device for prospective conservation treatments on archaeological copper and copper alloys. It also enables researchers to compare quantitatively the effectiveness of inhibitor treatments or coating systems.

In the future, further compounds should be tested with this corrosion test to be able to substitute BTA with an inhibitor that causes less colour change and is more effective on artifacts containing large amounts of copper chloride corrosion.

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Commercial product suppliers

Copper sheet 99-9%:

Smiths Metal Centres,
42-56 Tottenham Road,
London NI 4BZ, UK.

2-amino-5-mercapto-1,3,4-thiadiazole, 2-amino-pyrimidine, benzotriazole, 5,6-dimethylbenzimidazole, 2-mercaptobenzimidazole, 2-mercaptobenzoxazole, 2-mercaptobenzothiazole, 2-mercaptopyrimidine: BDH, Hunter Boulevard, Magna Park, Lutterworth, Leicestershire LE17 4XN, UK.

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