

## Performance of copper corrosion inhibitors in a museum environment - a comparative study using FTIR spectroscopy

W.A. Mohamed<sup>a</sup>, N.M. Rateb<sup>b</sup>, A.A. Shakour<sup>c</sup>

<sup>a</sup> Conservation Department, Faculty of Archaeology, Cairo University, Giza, Egypt.

<sup>b</sup> Chemistry department, Faculty of Science, Cairo University, Giza, Egypt.

<sup>c</sup> Air Pollution Department, National Research Center, Dokki, Giza, Egypt.

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### Abstract

The study aims to evaluate the ability of corrosion inhibitors to protect archaeological copper from corrosion in a museum environment. This test required a five years test period. For this test coupons were prepared and treated with the inhibitors and placed in the display cabinet together with the exhibited metal objects in the Old Egyptian Department of the museum of the Faculty of Archaeology in Cairo University.

Indoor air quality was evaluated; the concentrations of pollutants such as sulfur dioxide, nitrogen dioxide, ammonium, hydrogen sulfide, formaldehyde and suspended particulates and their water soluble constituents were measured. During the test period temperature and relative humidity were recorded. Corrosion inhibition of each inhibitor after five years exposure to museum environment was estimated by weight gain measurements. Inhibitors complexes formed on the surfaces of the test coupons were studied with Fourier transform infrared spectroscopy (FTIR). The spectra of such complexes were taken before and after five years exposure to the museum environment and then they were compared.

*Keywords:* copper, corrosion inhibitors, museum, environment, FTIR.

Corresponding author: TEL:+02 3841297 e-mail: [wafaaanw@hotmail.com](mailto:wafaaanw@hotmail.com)

### 1. Introduction:

It is well known that after the treatment of corroded metal objects in the laboratory it is important to apply a protective material on the metal surface. Corrosion can readily start unless the metal surface is properly protected with a suitable material such as a corrosion inhibitor. Some inhibitors, referred to in published literature, were tested in the laboratory by accelerated corrosion tests. Their performance has been evaluated against artificial corrosive conditions of high relative humidity and high temperature (Brusic, et al., 1991, Bastidas and Otero, 1996, Zucchi et al., 1995, Zucchi et al., 1996, Faltermeier, 1999 and Mohamed, 2000), but only a few of them gave satisfactory results (Borea, et al., 1971, Stambolov, 1978, Turgoose, 1985 and Skerry, 1985). However, the performance of such inhibitors in long term exposure to the museum environment has not been evaluated. The tested inhibitors are 1H-benzotriazole (BTA) ( $C_6H_5N_3$ ), 2-amino-5-mercapto-1,3,4-thiadiazole (AMT) ( $C_2H_3N_3S_2$ ) and 2-mercaptobenzothiazole (MBT) ( $C_7H_5NS_2$ ). The concentrations of sulphur dioxide, nitrogen dioxide, ammonia, hydrogen sulphide, formaldehyde and suspended particulates were measured in this research. These air pollutants are expected to affect metal objects in display cases and consequently are supposed to affect the inhibition efficiency of the tested inhibitors.

Sulphur dioxide ( $SO_2$ ) catalyzes the formation of cuprous oxide in damp atmosphere (Wiederholt, 1964), while hydrogen sulphide ( $H_2S$ ) is extremely corrosive to copper, and more than one volume of the gas in 600 million volumes of air can tarnish copper.  $H_2S$  is able to promote lattice imperfections, High concentrations of the gas produces black copper (I) sulphide on the copper surface (Stambolov, 1985).

Nitrogen dioxide NO<sub>2</sub> produces three reaction processes in an indoor atmosphere; production of nitric acid, rapid equilibration with ozone and nitric oxide (NO), and generation of nitrous acid (HONO) (Brimblecombe, et al., 2001). Nitric acid is known to be a strong oxidizing agent, but, it is volatile, so the free acid formed from NO<sub>2</sub> polluted air cannot be entrained on dry surfaces. This makes it of lesser harm if compared with SO<sub>2</sub>.

Chlorides are the most dangerous contaminants for metals; (Thomson, 1986). High concentrations of HCl gas in any kind of environment catalyzes the formation of copper (I) oxide (Feitknecht, 1952) In the presence of even a small amount of chloride cuprous oxide film is broken up causing further anodic dissolution of the metal (Stambolov, 1985). Formic acid vapor is usually released in display cabinets made of wood hence; it surrounds the museum objects in the case's atmosphere. High emission of the vapor while the case is airtight makes the carboxylic acid concentration extremely high (Ryhl-Svendsen, 2001). Wood-related materials, adhesives, varnishes and paints, all off-gas carbonyl pollutants (primarily aldehydes and organic acid vapors) (Gibson, 1998). Formaldehyde converts to formic acid in the presence of water. Formic acid formed from formaldehyde is much more dangerous than formaldehyde itself (Moller, 1984). Copper, bronze and brass can be attacked after 100 days in the presence of only 1,200 ppb of formaldehyde at (50% RH and 20°C ± 3°C) (Stulik and Grzywacz, 1992).

## 2. Experimental Procedure

### 2.1 Weight gain measurements

Copper coupons 50 x 25 x 1mm were cut from 99.997% copper sheets. Five coupons were used for each treatment. The coupons were polished by 600-800 grit SC papers, washed with running tap water, distilled water then degreased. The coupons were weighed then immersed in inhibitor solutions. The inhibitors were dissolved in 96% pure methanol. The inhibitors concentrations were 25 x 10<sup>-2</sup> mol, 1 x 10<sup>-2</sup> mol and 1 x 10<sup>-2</sup> mol for BTA, AMT and MBT respectively. The immersion durations in the inhibitor solutions were six hours, six hours and 24 hours respectively. After the coupons were removed from the inhibitor solutions they were dried and weighed. The coupons were placed in the museum inside a display cabinet together with a collection of metal objects in the exhibition hall. The percentage of inhibition (P.I.) was calculated according to the formula: 
$$P. I. = \frac{UCR - ICR}{UCR} \times 100.$$

(Trabanelli and Carassiti, 1970). Where UCR (uninhibited corrosion rate) is the mean weight of the corrosion products after exposure to test conditions; and ICR (inhibited corrosion rate) is the corresponding mean value of the inhibited coupons.

### 2.2 Air Quality Measurements

Atmospheric contaminants in both forms (particulates and gases) were measured inside the main show hall of the Old Egyptian Department of the museum. The museum is a concrete construction, located in the fourth floor of the main building of the faculty of archaeology. It is naturally ventilated and illuminated. Artificial light is rarely used. Windows are the sources of light during museum open hours and ventilation is supplied by the door and some side windows. Curtains are used to prevent direct sunlight from falling on the museum objects and they are always closed when the museum is closed. A (HVAC) facility is added but it usually works from 9am. to 2 pm. on very hot days in summer. It rarely works in winter for reasons of economy.

The measuring apparatus was placed in the exhibition hall as close as possible to the display case which contains the test coupons. It was not permitted to put the measurement apparatus which contains a vibrating vacuum pump inside the display cases. Sulphur dioxide (SO<sub>2</sub>), nitrogen dioxide (NO<sub>2</sub>), ammonium (NH<sub>3</sub>), hydrogen sulphide (H<sub>2</sub>S), formaldehyde (HCHO) and suspended particulates were measured through four consecutive months (from September 2002 to January 2003).

SO<sub>2</sub> was determined colorimetrically by the West and Geak method. Nessler's reagent was used for ammonia determination. NO<sub>2</sub> was determined by a modified Harrison and Jacobs method (Stern, 1976). The filtration technique was used in order to collect the total suspended particulate. The apparatus consisted of a vacuum pump, a calibrated dry gas meter to determine the sample volume and a filter holder. The used membrane filter was weighed before and after measurements. The weight gain is attributed to the weight of the particulate matter. Sulphate, chlorine and ammonium ions were determined in the water-soluble portion of suspended particulate matter.

### 2.3 FTIR spectroscopy:

Reference spectra of solid powders of the inhibitors were produced with FTIR spectroscopy. FTIR spectra of copper /inhibitors complexes formed on the surface of pure copper coupons treated with the tested inhibitors were also produced. The resulting spectra were compared with the spectra of the same copper /inhibitors complexes after exposure to the museum environment for five years. Samples were scratched off the surfaces of the coupons using a sharp hand engraver and they were compressed as pellets with KBr,

## 3. Results

### 3.1 Results of air quality measurements

It was found that the concentration of gaseous pollutants changed significantly during the test period. The results of air pollution measurements are given in Figures 1 & 2.

In Figure 1 the measured concentrations of gaseous pollutants are shown. The value of the mean concentration of SO<sub>2</sub> measured in the museum atmosphere (19.25 µg/m<sup>3</sup>) greatly exceeded the limit given by the National Bureau of Standards (NBS) which is 1 µg/m<sup>3</sup> (Stern, 1986). The concentrations of (NO<sub>2</sub>) ranged from 6 to 36 µg/m<sup>3</sup>, the mean of which was 18.75. This value exceeded the acceptable concentration for archaeological objects (10 µg/m<sup>3</sup>) given by Thomson, 1986.

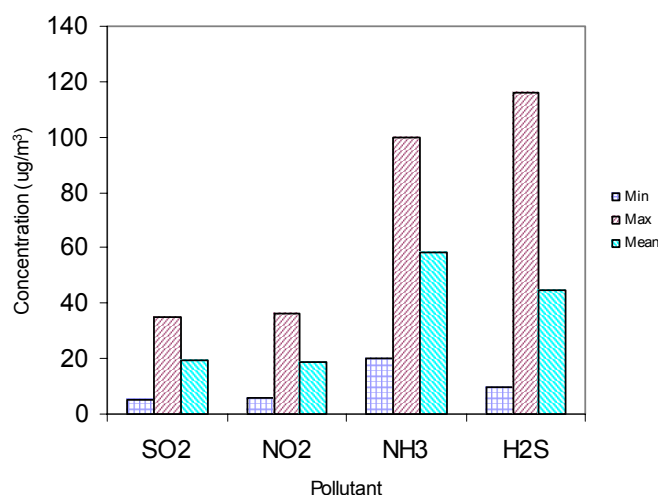


Figure 1. Concentrations of measured pollutants (µg/m<sup>3</sup>) in the museum environment.

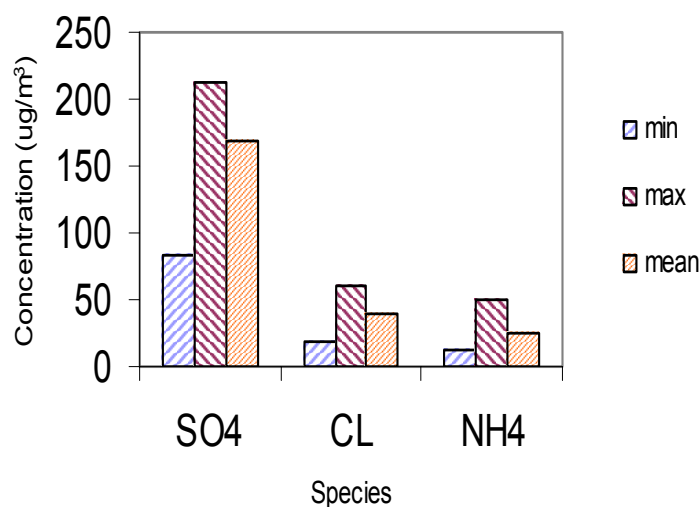


Figure 2. Concentrations of water-soluble constituents ( $\mu\text{g}/\text{m}^3$ ) of suspended particulates.

The mean concentration of ammonium was  $58.25\mu\text{g}/\text{m}^3$  while the mean concentration of hydrogen sulphide was  $45\mu\text{g}/\text{m}^3$ . The concentration of formaldehyde was  $32\mu\text{g}/\text{m}^3$ . The mean concentration of suspended particulates was  $123.50\text{mg}/\text{m}^3$ , while the maximum and minimum values were  $201.70$  and  $90.0\text{mg}/\text{m}^3$  respectively. The size of particulate was found to be less than  $10\mu\text{m}$ . The concentration of suspended particulates ranged from  $90$  to  $201.7\text{mg}/\text{m}^3$ , the mean concentration of which is  $123.5\text{mg}/\text{m}^3$ .

Water-soluble constituents of the suspended particulates  $\text{NH}_4$ ,  $\text{Cl}$  and  $\text{SO}_4$  were measured, results are given in Figure 2. The mean concentration of chlorine was as high as  $39.54\mu\text{g}/\text{m}^3$ , the mean concentration of ammonia was  $24.71\mu\text{g}/\text{m}^3$ , the mean concentration of sulphate was  $167.85\mu\text{g}/\text{m}^3$ . It was found that temperature values fluctuated inside the show-case from  $16^\circ\text{C}$  to  $35^\circ\text{C}$  and humidity fluctuated from  $60\%$  to  $75\%$ . These are very high values if compared with the acceptable temperature values for museum display. The acceptable value is  $19 \pm 1^\circ\text{C}$  in winter and  $24 \pm 1^\circ\text{C}$  in summer; however it should be reasonably constant to stabilize RH value, which must be limited to  $50$  or  $55 \pm 5\%$  (Thomson, 1986).

### 3.2 Results of weight gain measurements

The assessment of inhibition efficiency is based on weight gain measurements; results are represented in Figure 3. It has been found that after six months of exposing the inhibitor treated copper coupons to the museum environment the weight of the coupons treated with AMT and MBT increased, while the copper coupons treated with BTA showed no increase in weight after six months. The inhibition efficiency of BTA treatment was  $100\%$ , while for AMT treatment it was  $28\%$ . MBT treatment gave  $5\%$  inhibition efficiency.

After five years an increase in weight was measured in BTA, AMT and MBT treated coupons, but generally all the inhibited coupons gained less weight than the untreated coupons. The value of the inhibition efficiency of MBT treatment did not change after five years while the inhibition efficiency of BTA treatment decreased to  $83\%$ , but AMT inhibition efficiency increased to  $30\%$ . It can be concluded that after five years all the values of inhibition efficiency changed except the value of MBT inhibition efficiency.

Table (1): Gravimetric data of weight gain measurements.

Inhibitor	Wt. before Exp. (mean)	Wt. after 6 M. (mean)	Average change of Wt.	Wt. after 5 Y. (mean)	Average change of Wt.
Non	11.46800	11.46956	0.001560	11.47160	0.00360
BTA	11.29684	11.29684	0.000000	11.29744	0.00060
AMT	11.41464	11.41576	0.001122	11.41716	0.00252
MBT	11.12720	11.12868	0.001480	11.13062	0.00342

Wt. = weight in grams.  
 Exp. = exposure to museum environment.  
 M. = months.  
 Y. = years.

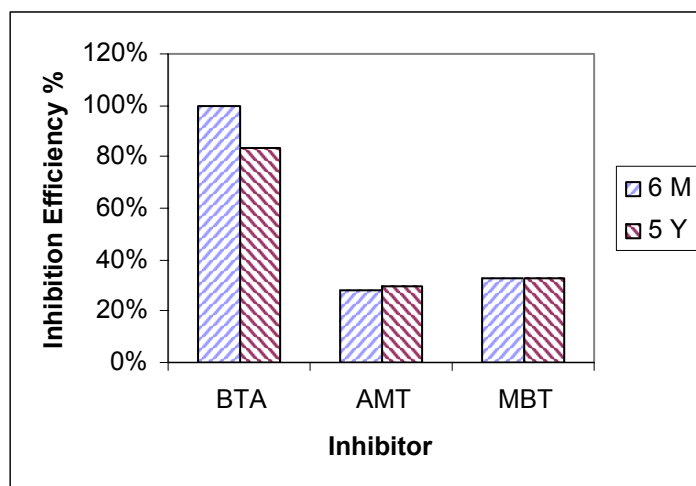


Figure 3. the inhibition efficiency of tested inhibitors after six months (6M) and five years (5Y).

### 3.3 Results of FTIR Spectroscopy

Figure 4a shows a reference transmission spectrum of solid BTA on KBr pellet. The spectrum shows a multi peak band at 2516-3247  $\text{cm}^{-1}$  which indicates the presence of the NH group, also it shows a peak at 1209  $\text{cm}^{-1}$  due to the presence of the N=N group. Figure 4b represents the transmission spectrum of Cu (I)BTA complex in which the band characteristic to the N-H group has disappeared. This indicates that Cu treated with BTA was bonded to the amino nitrogen in the BTA structure.

The noticeable shift in the intensity of the band N=N at 1209  $\text{cm}^{-1}$  in Figure 4b illustrates that Cu was also coordinated to the unsaturated ring nitrogen. This coordination was first suggested by Poling 1970. Figure 4c represents the transmission spectrum of Cu (I) BTA complex after the exposure of the sample to the museum environment for five years. In this spectrum a band appeared at 3267  $\text{cm}^{-1}$  indicating the presence of the N-H group and a band at 1210  $\text{cm}^{-1}$  with higher intensity, the latter indicating the presence of the N=N group. It can be concluded that after five years exposure to the museum environment the inhibitor complex lost the bonding with the copper surface. The band which appeared at 1622  $\text{cm}^{-1}$  representing the C=N group was shifted to 1644  $\text{cm}^{-1}$  as shown in Figure 4c.

Figure 5a represents the transmission spectrum of AMT on KBr pellet. In this spectrum there are bands at 3340  $\text{cm}^{-1}$ , 3251  $\text{cm}^{-1}$  and 3127  $\text{cm}^{-1}$  indicating the presence of NH<sub>2</sub> and S-H groups. Figure 5b shows the spectrum of Cu (I) AMT complex. The sharp band in Figure 5a at 3127  $\text{cm}^{-1}$  is assigned to the S-H group. This band disappeared in the spectrum in Figure 5b as the complex was formed through S-H and NH<sub>2</sub> groups. The disappearance of the band indicated the bonding of sulphur to the metal ions. This is in agreement with the IR results of studies undertaken by Gajendragad and Agrawala 1975 and Ganorkar et al., 1988. The

multiple band at 2925-3400  $\text{cm}^{-1}$  of the AMT spectrum in Figure 5a is due to stretching of the NH bond. This was shifted to 2923-3434  $\text{cm}^{-1}$  in the spectrum of the AMT complex Figure 5b. There is a significant band appearing at 443  $\text{cm}^{-1}$  which is attributed to the M-N bond. This indicates the participation of the nitrogen of the  $\text{NH}_2$  group in active coordination. The spectrum of the complex after five years exposure to a museum environment shown in Figure 5c shows a shift in the band representing the M-N bond from 443  $\text{cm}^{-1}$  (as shown in Figure 5b) to 449  $\text{cm}^{-1}$ . This shift was accompanied by an increase in the intensity, this indicated that the M-N bond became a little stronger with time.

Figure 6a shows the transmission spectrum of solid MBT on KBr pellet. In Figure 6a the spectrum shows a band at 3111  $\text{cm}^{-1}$  which indicates the presence of the N-H group and the presence of a band at 1153  $\text{cm}^{-1}$  which indicates the presence of the C=S group. Figure 6b shows the spectrum of the Cu(I)MBT complex, in which the N-H group disappeared. Hence it is suggested that the complex was formed via a bonding between Cu(I) and nitrogen atom as explained by Chadwick and Hashmi, 1979 - as elimination of H(-N) atoms. The complex was reanalyzed with FTIR spectroscopy after five years in the environment specified above. If the spectra of the MBT complex before and after five years (Figures 6a and b) are compared, it will be found that there is no evidence of the presence of the N-H group but there is a new band that appeared in Figure 6c at 1115  $\text{cm}^{-1}$  indicating the presence of the C=S group. Another band appeared at 443  $\text{cm}^{-1}$  and it is attributed to the M-N group which indicates the participation of the nitrogen in the coordination. This band shifted to 440  $\text{cm}^{-1}$ , as shown in Figure 6b, with a sharp decrease in the intensity. It can be concluded that in time the MBT complex lost bonding with the copper, although this loss of bonding is not as sharp as it is in case of the BTA complex.

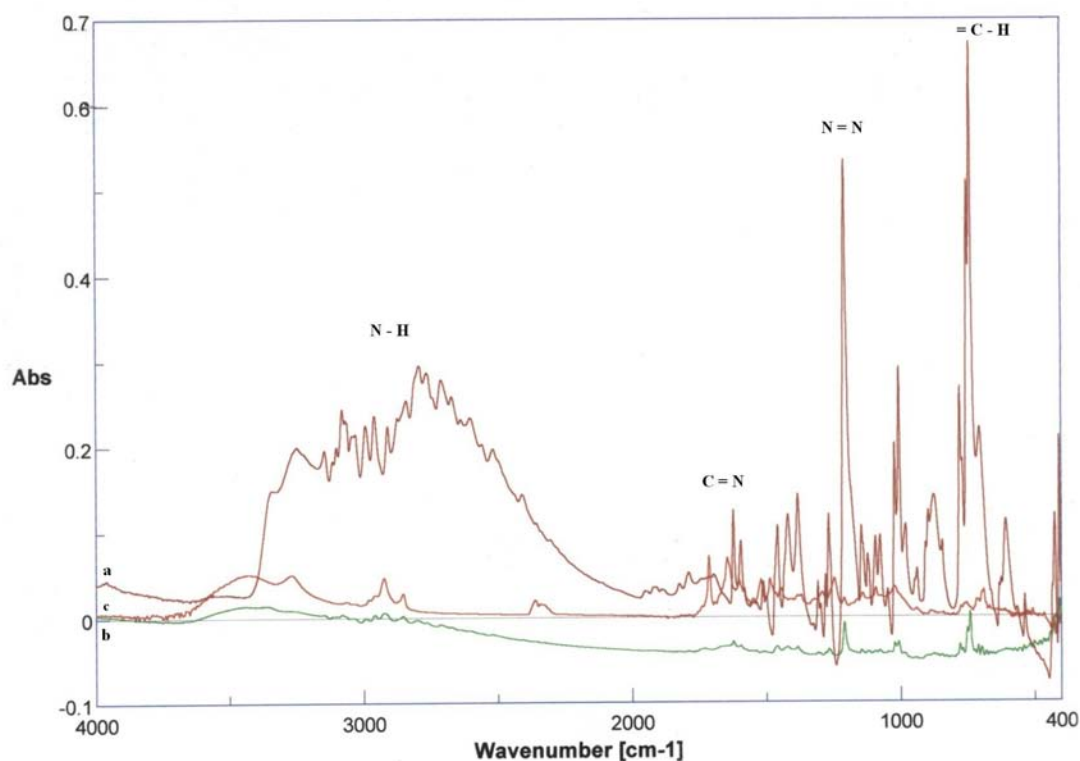


Figure 4. FTIR spectra of BTA (a) BTA on KBr pellet, (b) BTA complex and (c) BTA complex after 5 years.

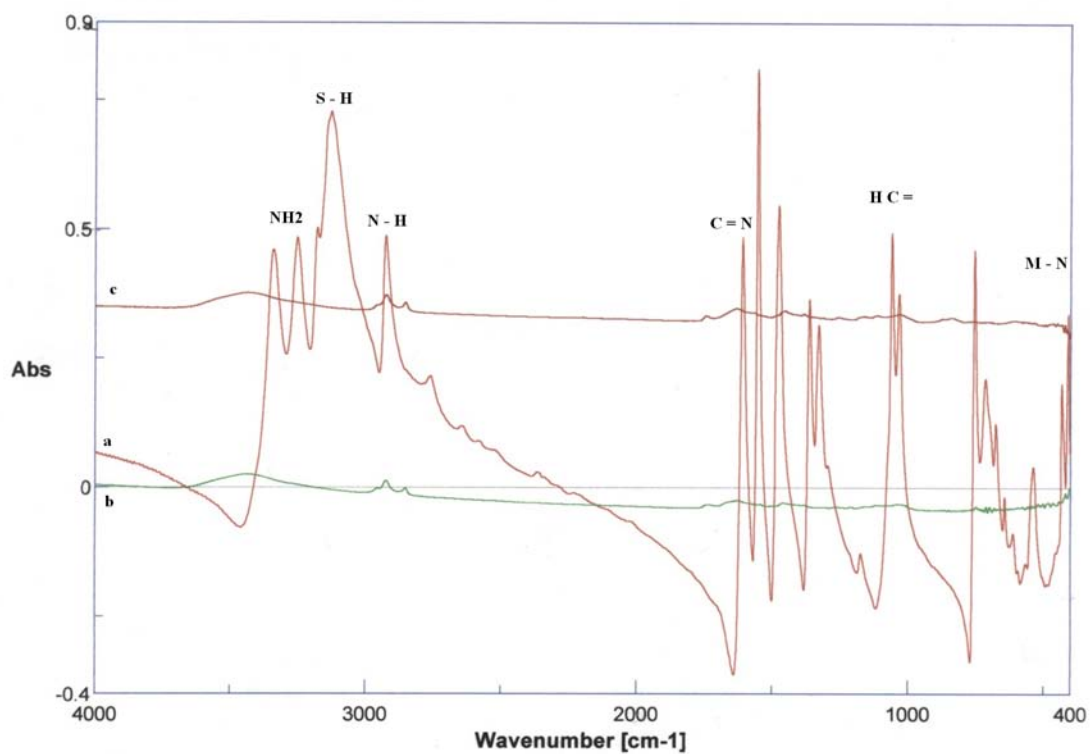


Figure 5. FTIR spectra of AMT (a) AMT on KBr pellet, (b) AMT complex and (c) AMT complex after five years.

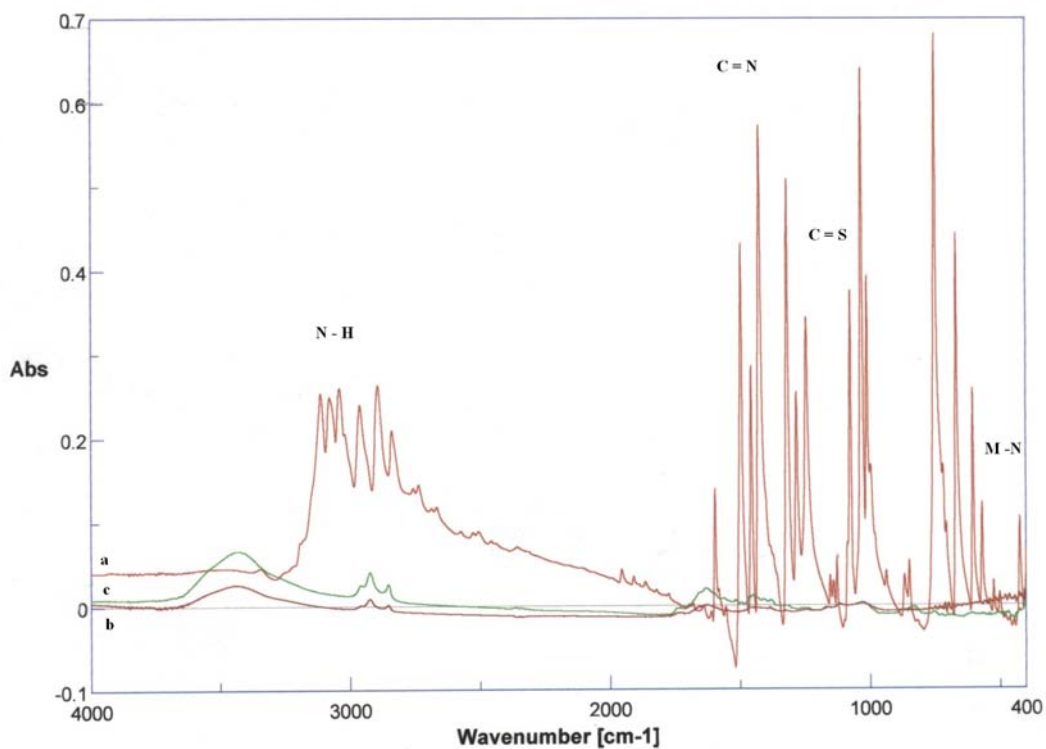


Figure 6. FTIR spectra of MBT (a) MBT on KBr pellet, (b) MBT complex and (c) MBT complex after five years.

#### 4. Discussion

The evaluation of the durability of corrosion inhibitors in an actual museum environment is of great importance, as it gives a more realistic picture of the performance of the inhibitor after treatment of the metal object is completed and the object is returned to the display-case. The experiments proved that BTA was the best of the tested inhibitors to protect copper from corrosion in an uncontrolled museum environment. However its inhibition efficiency decreased to 83% after five years.

The results indicated that polluted atmosphere, high and uncontrolled RH and temperature negatively affected the inhibition efficiency of the tested inhibitors. Although it was found in the literature that these inhibitors gave good inhibition efficiencies when tested in the laboratory against accelerated corrosion tests, the current research proved that the long term service test undertaken in the actual environment (uncontrolled museum environment) gave comparatively low inhibition efficiencies.

The concentrations of the measured pollutants in the museum of Cairo University were very high compared with the acceptable limits given by Thomson and the National Bureau of standards (NBS). The natural ventilation through windows made the situation even worse. Standards for the acceptable concentrations of  $\text{NH}_3$  and  $\text{H}_2\text{S}$  in a museum environment have not been found in the literature. This may suggest further areas of study to establish the ideal limits for metals display and the effects of the combination of the different pollutants on the metal surfaces

Although the weight gain and weight loss measurements are of the most commonly used procedures in corrosion studies, they must be accompanied by another procedure to explain the results. FTIR spectroscopy was very helpful in giving an explanation about the alterations that have taken place in the complex film. The decrease in inhibition efficiency was due to dislocation of bonding with the metal surface.

#### 5. Conclusion

From the study it can be concluded that after six months exposure to a museum environment BTA gave 100% inhibition. AMT and MBT after six months gave low inhibition efficiencies 28% and 5% respectively. The value of BTA inhibition decreased to 83% after five years because of the partially breaking up of bonding with the metal surface. MBT lost bonding with copper to a lesser extent, although this loss of bonding is not as sharp as it was in case of BTA inhibition and did not affect inhibition efficiency after long term exposure. Surprisingly AMT inhibition efficiency increased to 30% after five years as the M-N bond became stronger. This may suggest a further area for study.

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