

ELECTROCHEMICAL TESTS AS ALTERNATIVES TO CURRENT METHODS FOR ASSESSING EFFECTS OF EXHIBITION MATERIALS ON METAL ARTIFACTS

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Summary—Many materials used in the storage and display of museum objects are potentially corrosive and should not be used in the vicinity of specific metals. The 'Oddy test' is the procedure used in most museums to assess the suitability of such materials. However, there have been reports in the literature of problems with reproducibility and time constraints. In manufacturing industries, where similar problems have been encountered, electrochemical testing has replaced tests based on visual assessment. Experiments with electrochemical testing are reported here, using typical exhibition and storage materials in conjunction with lead, copper and silver coupons. The results suggest that the polarization resistance test has potential for rapidly and objectively identifying possibly damaging materials. This method provides a quantitative measure of corrosion rate in milli-inches per year for a specific metal used in conjunction with a particular material.

Introduction

It is well known that materials used in the construction of exhibition and storage cases may emit volatile components that can cause corrosion of metal artifacts. Many researchers have characterized the most destructive volatiles and identified their sources so they can be eliminated as far as possible in display and storage construction [1–5]. However, given the wide variety of materials used, including plastics, textiles, paints, inks, adhesives and wood products, many museums maintain a program of materials testing to identify potentially damaging case components.

The 'Oddy test' is the standard test in most museums for assessing the effects of materials on metal artifacts. It has certain advantages, but also certain limitations. Similar visual tests have been tried in many industrial laboratories, but rejected due to problems with reproducibility and time requirements. Several other tests are also currently employed by conservators and museum scientists, but none of them has proved ideal.

Therefore we decided to explore the electrochemical testing methods that are currently the standard approach in industry when the compatibility of materials used in conjunction with metals must be assessed. Our primary goal was to assess the suitability of such test methods for conservation applications.

Test methods currently used for artifacts

The Oddy test

The most common test procedure for exhibition materials carried out in museum laboratories today

is a variation of that originally described by Andrew Oddy of the British Museum Research Laboratory [6, 7]. The test involves placing a small metal test specimen in a glass vessel along with a sample of the prospective case material. A small vial containing moistened cotton is added, producing a relative humidity (RH) of 100% in the flask, and the flask is sealed. It is then placed in an oven held at an elevated temperature, typically 60°C. The elevated temperature and humidity accelerate the tarnishing process that might be caused by the release of vapors from case components. After oven exposure, the metal test specimens are assessed qualitatively for degree of alteration in comparison with controls placed in jars with no test materials and subjected to the same test regime.

An advantage of the Oddy test is that month-long artificial aging might be expected to give a reliable indication of what can be expected to occur in a storage or display case over a period of several months to several years. Also, the test requires little in the way of equipment and expertise, so it can be carried out in essentially any museum laboratory.

One major disadvantage is that, due to construction and purchasing schedules, curators and designers often need to know the suitability of materials in much less than a month. Thus the tests are often severely abridged (to two weeks, for example) or eliminated altogether. There are other difficulties: judging degree of tarnish is a subjective procedure; the elevated temperature may produce effects not seen under real conditions; variations in the size and grade of metal specimens and in the type of flask or the amount of water added can severely

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affect test results; and even slight accidental variations in conditions within the glass flasks can affect reproducibility [8–12]. In addition, the results of the Oddy test have never been comprehensively compared with long-term field trials of exhibition materials to assess how accurately the test predicts material performance.

The elevated temperature and humidity of the Oddy test may sometimes cause chemical reactions which result in the release of vapors that would not ordinarily be a problem in exhibition cases. For example, Schniewind [13] has proposed that in atmospheres of 100% RH (as used in the Oddy test) small variations in temperature could cause condensation of water inside wood, resulting in biodeterioration which might lead to unpredictable changes in the acid release behavior of wood materials.

Although the British Museum Department of Conservation recently emphasized the importance of following a standard protocol developed through extensive testing and experimentation, and noted problems with reliability that may result from failure to adhere to those protocols [12], we found that there are many variations in how the Oddy test is conducted [9–11, 14–17]. These include the type of glass container used, the size and thickness of metal test specimens, the method of placing or suspending test specimens within the glass container, the amount of distilled water added to the container, the length and temperature of oven exposure, whether or not the glass containers are opened for interim observations or to add more water, and the number of categories used for grading the results.

Replication issues

To examine issues of reproducibility and replicability, we conducted a series of experiments using the Oddy test according to a procedure widely employed in conservation laboratories in the United States [9, 17]. Thirty-four common exhibit case construction materials were chosen for testing, including a variety of wood products, metals, plastics, composites, gasket/caulking and interior exhibit-mounting materials.

Metal test specimens were cut to a uniform size (2 × 2cm for lead and copper, 1 × 1cm for silver). Test specimens were polished and degreased, stamped with a number on one side, then sealed in a nitrogen atmosphere until use. Thereafter they were handled only with tweezers or gloves.

The 34 products were each tested with three replicates for each metal. Three control specimens for each metal were also prepared. Replication in the Oddy test is very seldom reported in the conservation literature, since most museum laboratories

operate under constraints of time and materials. Two published accounts in the conservation literature [9, 18] indicated that variability could seriously affect the interpretation of results for some products; and industrial literature has frequently reported the need for replication of both test specimens and controls [19, 20].

The numbered test specimens were matched with product samples using a random number table. The object was to ensure that grading of results would be done 'blind' in regard to the identification of products and replicates, to avoid bias.

Test specimens and product samples were placed in 120ml glass jars with Teflon-lined lids (to improve sealing and thus help prevent moisture escape). The bottoms of the jars were lined with Mylar to prevent water that might condense in the somewhat curved bottom of the jar from coming into contact with the test specimens. Since we are interested in both contact and non-contact effects of materials, a portion of one side of each test specimen was placed in contact with the test material [9, 17]. A glass vial containing 2ml of distilled water was added to each jar; cotton was placed inside the vials to help prevent the water from spilling if the vial were to be knocked over while in the jar. The lids were then shut as tightly as possible and the jars were placed in a 60°C oven. Order of oven placement was also randomized, to reduce the chance that all three replicates of any material would reside in one oven location and be similarly affected by any temperature differentials. Jars were then left undisturbed for 30 days.

When the jars were removed, most had some visible water condensation on the inside of the glass, indicating that 2ml of water was sufficient to maintain the elevated humidity needed to accelerate corrosion. The test specimens were removed from jars using tweezers, then lined up in numerical order so that the identity of products and replicates was not known during the grading procedure. Grading for each metal was conducted by two analysts, in comparison with the three control specimens. The scale used was:

- (1) essentially equal to the controls, or negligible corrosion;
- (2) observably more corroded than the controls, with slight or minor corrosion;
- (3) much worse than the controls, with moderate to severe corrosion.

The lead specimens showed much more variation in degree of corrosion than the other two metals. The controls were relatively unaffected; test specimens with product samples ranged from no corrosion to moderate-to-heavy. For most products there

was little or no difference between the contact and non-contact exposures, although some of the non-contact test specimens were less corroded. Very few of the silver test specimens became corroded, especially on the non-contact side, and the degree of corrosion was seldom very high.

The range of degree of corrosion was narrower for copper test specimens than for lead; variation on the non-contact side was even less. The range was greater than for silver, however. Most importantly, the three control specimens for copper showed so much variation in appearance that interpretation of level of corrosion caused by product samples was hindered. The problem of change in appearance of copper control specimens has been noted in the literature [16], but no one has reported using replicates of control test specimens, so the grading difficulties caused by variation in appearance have not been addressed. In addition, there was a wide range of corrosion oxide colors ('patina') and appearances for copper, and it was difficult to correlate these with degree of corrosion.

Agreement between the two assessors was generally close. Lead, where there was the most visible corrosion, gave the least variation: four differences of opinion out of 102 for the contact side, and eight differences for the non-contact side. These assessments were completely independent. Copper gave more variation (24 differences), even though there was some consultation between the two assessors, due to the interpretation difficulties referred to above. The silver gave 25 differences. The greater variation in assessments for copper and silver is probably due to the fact that the differences between test specimens of these two metals were more subtle.

Although replicates for some of the products gave consistent results, the results for other replicate jars sometimes varied extensively, ranging over two adjacent grades, even when differences of opinion between the assessors were averaged out. In a few cases the results for the replicate jars ranged from 1 to 3. This degree of variation indicates that it may be very difficult to reproduce the test conditions in every jar, even when great care is taken. In industry, the difficulties involved in reproducibility and visual assessment of similar exposure tests have been recognized [21].

The frequency distribution (Table 1) of maximum difference between replicates (with differences between assessors averaged out) illustrates that it is common for replicates to vary; there may be at least one Oddy test-unit difference between replicate jars that contain the same test material. The variability is especially severe for lead. The problem is exacerbated because two graders will often assess the same coupon differently.

Table 1 Oddy tests: frequency distribution, maximum difference between replicates*

	0	0.5	1.0	1.5	2.0
Lead (contact)	9	2	19	1	3
Lead (non-contact)	11	1	17	3	2
Copper	7	8	12	3	4
Silver	12	9	9	3	1

*One unit of difference refers to a full grade difference on visual assessment scale.

1 equal to controls

2 visually more corroded than controls

3 much worse than the controls

The British Museum Department of Conservation has recently proposed ways of reducing the variability of these tests by advocating a standard protocol [12]. Our results, using the variation of the test method currently practised in many United States conservation laboratories, support the British Museum's suggestions regarding the need for conformity in procedures. These suggestions include photographic standards for each grade of corrosion for different metals. If these new procedures are strictly adhered to by all laboratories, reproducibility should be improved. In industry, however, such evaluations have never proved satisfactory, even after application of better experimental design principles [19-22].

The fact that grading is done 'blind' with respect to materials and replicates almost certainly increases the variation observed both among replicates and between assessors. Knowledge of which test specimens are replicates of which product does subtly bias assessment, however hard one tries to be careful and fair.

Similar problems with reproducibility have been reported in the conservation literature. For example, Beale [23] mentions that his Oddy tests of a particular product showed it to be one of the safest tested in his laboratory, whereas another laboratory had reported poor results with the same product. This situation was hypothesized to be due to differences in age of the product samples tested. While this may indeed be the case, it would be interesting to determine to what extent the difference might be due to lack of replicability of the tests themselves.

Greater standardization of protocols and improved documentation for visual assessment will not resolve another major problem of the Oddy test procedure: the time required for adequate exposure. The 28 days needed to perform the test will continue to cause difficulties for many conservation laboratories.

Other test methods in current use

Various alternatives to the Oddy test have been proposed in the conservation literature. These include a test using pH indicator papers to detect volatile acids, intended to supplement rather than replace the Oddy test [24, 25]; a microchemical test using sodium azide to identify the presence of sulphur, a common cause of tarnish in museum cases [26, 27]; a flame test to detect the presence of chlorine, which might corrode copper [28]; a test to determine the corrosive effects of organic acids emitted from wood products, by monitoring the weight of corrosion products formed [29]; and tests for detecting volatile organic acids and formaldehyde (methanal) [30]. However, none of these is completely satisfactory, for reasons which include the length of time required to perform the tests, subjectivity of assessments, and a limit to the number of corrosive agents that can be detected. We therefore decided to explore the electrochemical testing alternatives that are standard in industry.

Exposure tests in industry

Vapor-phase corrosion, where volatile contaminants from materials used in close proximity to an industrial product may accelerate corrosion of metal components, has been of as much concern in industry as it has in museums. Corrosive vapors may be encountered during manufacture, transport, storage or use. Direct contact between metals and materials used for storage, transport or use may also accelerate corrosion, and has long been recognized as a potential problem.

Exposure tests very similar to the Oddy test were developed in industry [2]. One such test involved suspending metal specimens in a glass vessel, enclosing the material of interest along with a vial of water to maintain 100% RH, then subjecting the vessel to an elevated temperature for several weeks. However, it has been more common to use weight change as a quantitative measure of corrosion, rather than visual assessment.

More than a decade ago, researchers studied a variety of test methods to assess corrosivity of thermal insulation materials used near metals in residential structures [21]. They were looking for a test that would be quantitative, reproducible, and could give rapid results, so that it could be incorporated into quality control operations during manufacture. They experimented with exposure tests that were assessed by visual observation and by measurement of coupon weight loss, as well as electrochemical testing. They compared the results of the different tests, measured in terms of corrosiveness compared

to distilled water, and found good agreement between results from the coupon weight-loss measurements and from electrochemical testing. By contrast, the agreement of appearance ratings with the other tests was only fair. It was concluded that visual assessment was inherently subjective and likely to be unreliable.

Crume [19] reports on visual exposure tests developed to test insulation materials, and describes the innovations in experimental design and data analysis that were necessary to obtain an acceptable level of reproducibility. Samples of insulation material were placed in contact with the metal of interest, as were cotton controls. After a period of exposure at elevated temperature and relative humidity, a visual assessment of degree of corrosion was made. It was found that simply comparing coupons to a control and assigning a grade did not provide reproducible results.

The protocol that was eventually developed included the use of an adequate number of replicates for the specimens exposed to an insulation material and for control specimens—for example, five test specimens per insulation material and five control specimens exposed only to clean cotton. More consistent results were obtained by ranking specimens (including both test and control specimens) from 'best' to 'worst', rather than trying to grade each specimen. It was also found desirable to use a panel of trained judges or observers, rather than a single individual. It was important that the judges did not know the identity of any of the test specimens, in order to prevent bias. Finally, a non-parametric statistical test was applied to the data, to determine whether there was any statistical difference between controls and test specimens. It was concluded that this more rigorous procedure was less subjective, and gave clearly defined statistical criteria for 'pass' or 'fail'; as a result, it was more reproducible. These methods and findings parallel those of similar experiments in conservation research [18].

A survey more than a decade ago showed that many organizations in the chemical process industry were already using electrochemical testing methods for materials selection [22]. As in the museum world, time was a critical factor: materials often had to be selected after only short-term laboratory simulations. Silverman [20] reports that a wide variety of electrochemical tests have been used successfully. These are sometimes combined with specimen exposure at the vapor/liquid interface or in the vapor phase. However, he noted that the biggest problem with such exposure tests is that, if specimens are to provide valid information on corrosion rate, a long exposure time or a large number of separate tests may be required.

Electrochemical testing

Corrosion is an electrochemical reaction; the rate is determined by the current flowing between anodic and cathodic areas. Monitoring that electron flow to measure the corrosion process is the basis of electrochemical testing. The corrosion current measurement can then be used to calculate rate of metal loss. Measurements of current-potential-resistance relationships following Ohm's law, $E = IR$ (voltage equals current times resistance), under carefully controlled conditions, can yield information on pitting and crevice corrosion tendency, as well as on corrosion rate. Many different test methods have been developed, each providing specific information and having both advantages and disadvantages for certain applications.

In industrial testing laboratories, electrochemical methods are a standard way of rapidly and quantitatively assessing the possible corrosion behavior of metals under exposure to specific environmental conditions. Electrochemical testing is widely considered to be a reliable approach to accelerated testing for assessing the possibility of corrosion under natural conditions, without changing the basic failure mechanisms involved. Much research and discussion of these methods is available in the literature, and the American Society for Testing and Materials (ASTM) has been active in the development and revision of standards for such tests [22, 31-44].

When a metal (such as a silver specimen) is in contact with a corrosive electrolyte, it assumes a potential (relative to a reference electrode) which is termed the corrosion potential, or E_{corr} . A specimen at E_{corr} has anodic and cathodic currents which are equal in magnitude: the corrosion current flowing from anode to cathode is exactly balanced by that flowing from cathode to anode. In order to measure the net current (I_{corr}), that equilibrium needs to be disrupted.

To study the anodic reaction of a specimen in a particular environment, a voltage is applied to change the freely corroding potential of the metal in a more electropositive direction with respect to the inert electrode (acting as a cathode). This process is known as anodic polarization. The opposite process, cathodic polarization, can also be carried out. Polarization of the specimen makes it possible to measure a resulting net current because, as the potential is shifted in, for example, the negative direction, the anodic (positive) current becomes negligible in comparison to the cathodic (negative) component. The net corrosion current can be measured from the interaction of the corrosion potential and either the anodic or the cathodic current.

The test specimen has a polarization resistance relationship (R_p) with the surrounding environment.

Polarization resistance refers to the degree to which a metal resists corrosion attack in a certain environment. When the external power source supplies voltage to displace the equilibrium of the metal relative to a reference electrode (to change the E_{corr} value to a polarized potential value, E_{pol} , with either a positive or a negative charge), the net current flowing between the metal and a counter electrode can be measured.

The most important parameter derived from an electrochemical test is the corrosion current. Using appropriate formulae, this measured current is easily translated into anticipated rates of corrosion for a specific metal in relation to the materials under consideration.

Experimentally, polarization characteristics are measured by plotting the current response as a function of the applied potential. To do this, the potential is applied to a value about 20mV below the equilibrium value and is then swept at a low scan-rate up to a potential about 20mV above the equilibrium value, the current response being recorded. The resulting plot is termed a polarization spectrum (Figure 1). A relationship exists between the slope of a polarization curve and the corrosion rate. However, such curves are not fully linear because of various interference reactions. In what is called the Tafel region, the measured curves better match the theoretical curves, their (Tafel) slopes being constant.

The corrosion rate can be calculated in several different ways using the polarization spectrum. For example, computer programs can be used for automatic curve-fitting with default calculation methods. Alternatively, an operator can examine the spectrum on-screen and perform the curve-fitting manually. The value obtained refers to an average, uniform corrosion rate, and provides no information about susceptibility to localized pitting or cor-

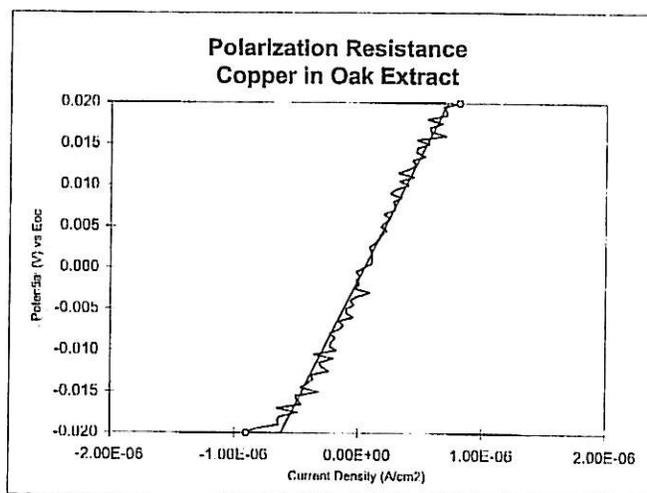


Figure 1 Polarization resistance scan.

rosion attack. Therefore, as a modification of a polarization spectrum, a cyclic polarization spectrum is often employed (Figure 2). This technique consists of a scan starting at a certain cathodic potential and ending at a preselected anodic potential at which the scan is reversed in polarity and the final potential value coincides with the starting point. Even if no corrosion is visible, this cyclic scan can detect the propensity of localized areas to microscopic pitting, which means that corrosion can develop over time. If corrosion is indicated, the scan can be held at a set potential allowing corrosion product to amass for subsequent identification.

Our experiments included trials using both the polarization resistance technique (using two curve-fitting methods) and the cyclic potentiodynamic polarization technique. The results are discussed below.

Polarization resistance

This technique is used to measure the resistance of a test specimen to oxidation during the application of an external potential. The corrosion rate is directly related to the polarization resistance (R_p), and can be calculated from it by the formula:

$$R_p = \frac{\beta_A \beta_B}{2.3(i_{\text{corr}})(\beta_A + \beta_B)}$$

where β_A = anodic Tafel constant expressed in volts per decade of current, β_B = cathodic Tafel constant expressed in volts per decade of current, 2.3 = the natural log of 10; i_{corr} = corrosion current (in μA).

From this equation the corrosion current can be calculated as:

$$i_{\text{corr}} = \frac{\beta_A \beta_B}{2.3(R_p)(\beta_A + \beta_B)}$$

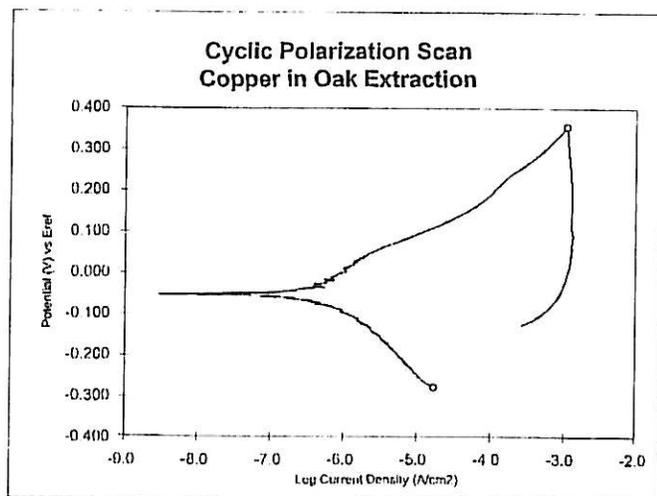


Figure 2 Cyclic polarization scan.

Once i_{corr} is determined, the corrosion rate (in milli-inches per year, mpy) can be calculated from the following equation:

$$\text{corrosion rate (mpy)} = \frac{0.13 i_{\text{corr}} (\text{E.W.})}{(A) (d)}$$

where E.W. = equivalent weight (in $\text{G}\cdot\text{eq}^{-1}$), A = area (in cm^2), d = density (in $\text{g}\cdot\text{cm}^{-3}$), 0.13 = metric and time conversion factor.

During the polarization resistance experiment, the data are obtained by scanning a range of $\pm 20\text{mV}$ about the open circuit potential (E_{corr}). A typical scan starts at -20mV versus E_{corr} and ends at $+20\text{mV}$ versus E_{corr} . The scan rate is typically $0.25\text{mV}/\text{sec}$. The resulting curve plots the applied potential versus the measured current (see Figure 1).

Cyclic potentiodynamic polarization

This technique is used to determine the active/passive characteristics and pitting tendencies of a test specimen in a given metal/solution system. The technique first applies a potential scan beginning at a cathodic potential of E_{corr} and continuing in the positive (anodic) direction until a large increase in current occurs. When the scan reaches a user-programmed current density value, it reverses and begins scanning in a negative (cathodic) direction. The scan rate is typically $1.7\text{mV}/\text{sec}$. The resulting curve is a plot of the applied potential versus the logarithm of the measured current (see Figure 2).

The potential at which the current increases sharply is defined as the pitting potential. When pitting occurs on the forward scan, the reverse scan will trace a hysteresis loop. The potential where the loop closes on the reverse scan is the protection (or repassivation) potential.

Materials and methods

Electrochemical tests are intended to simulate the corrosion behavior of a metal under the influence of a specific environment. To simulate the corrosive medium, an initial set of experiments using aqueous extractions of 34 materials was prepared following an in-house laboratory standard which is a modification of ASTM C871-84 [45].

To prepare the sample for extraction, it must be of a size that maximizes the surface area. For free-flowing particulate solids, or cloth-like materials, no further reduction in size is required. For non-free-flowing particulate solids, each sample is pulverized, cut, or broken into smaller pieces so that no piece is larger than 6.3mm in diameter.

Fifty (50 ± 0.1 g) grams of the sample are placed in a borosilicate glass flask to which 200ml of solvent (water, methanol, etc.) are added. An aqueous mixture is heated rapidly on a hotplate to boiling, and maintained at boiling temperature for 30 minutes. In the case of methanol solvent, the mixture is held at ambient conditions for 24 hours. A reflux condenser is required to avoid excess evaporation of the solution.

After 30 minutes, the solution is removed from the heat and allowed to cool for 15 minutes before filtering through a Buchner funnel containing a pre-washed, ashless, rapid-filtering paper (Whatman 41 or equivalent). The entire sample/solution is transferred and filtering continues until the filtrate stops flowing. The flask is washed thoroughly, using a minimum of 10 small washes and keeping the total filtrate within 250ml. The filtrate is transferred to a volumetric flask, allowed to cool below 38°C and then diluted to exactly 250ml. The extracted solution is now ready for testing.

Testing was conducted using a Gamry Instruments corrosion measurement system. The system consists of two potentiostatic boards that are installed within a personal computer, and associated software (electrochemical control software, and polarization software controlling the potentiostat); this computer is connected to a test cell containing the specimen to be tested, an inert counter electrode (graphite or platinum) and a reference electrode. The potentiostat we used is a PC3 Potentiostat/Galvanostat/ZRA, a research grade electrochemical instrument that is installed in an AT-compatible computer. This provides the measurement electronics of the Gamry Instruments computer-controlled electrochemical testing system; a single laboratory computer can drive up to four potentiostats performing four different experiments. The electrochemical control software used was the CMS100 electrochemical measurement system, a Microsoft Windows-based program that can run standard electrochemical applications. The polarization software used to control the potentiostat is the CMS105 DC corrosion measurement system, which is an add-on application to the above software package. It uses standard electrochemical techniques to investigate corrosion problems, allowing prediction of long-term corrosion rates and extraction of mechanistic information from an array of experiments. For our experiments, a corrosion rate (in milli-inches per year, or mpy*) was calculated through polarization resistance, using two different methods of calculating rates, and through cyclic potentiodynamic polarization.

*Software available in the United States usually uses mpy as the unit of measurement for corrosion rate. Conversion to the equivalent metric corrosion rate can be calculated as $1\text{ mpy} = 0.0254\text{ mm per year, or }25.4\mu\text{m per year}$ [46].

For these initial tests, the electrochemical testing procedure was replicated for each material/environment and metal type (lead, copper and silver). However, small sample sizes for each of the materials collected for these initial experiments precluded replication of the full extraction procedure (for example, a single extraction was used to repeat the electrochemical procedures) so the results cannot be considered representative of the true reproducibility of the tests. We therefore decided that larger specimens of test materials must be obtained for our final experiments.

These initial tests confirmed what has been found for the Oddy test: lead will corrode to a greater degree than copper, which will corrode more than silver. These tests also confirmed that a wide range of corrosion rates will be found within a single metal for various materials, which means that it should be possible to differentiate clearly between 'good' and 'bad' materials. Reproducibility was generally good, and we found that an extraction could be completed in about 90 minutes, with a further hour needed to run the electrochemical test.

For the final experiments we replicated the full extraction procedure, twice for each material, since that would better duplicate the real-world variability between different laboratories. We also decided to experiment with more than one extraction protocol, hypothesizing that different extraction procedures might lead to the release of different levels of corrosive agents. Thus the new protocol included some experiments with three extraction methods (water, methanol, and a 50/50 solution of water and methanol). The goal was to see which extraction method gave the highest corrosion rates for different types of material and different metals (lead, copper and silver). We were also interested in the comparative reproducibility of the various methods.

Seven materials were selected for testing using all three extraction techniques. There were two replicates for each of the three metals, giving a total of 126 analyses. The test materials were cardboard, wood (oak), Medex particle board, liquid hide glue, a paint (Glidden Dulux soft matt latex paint in white tint), vulcanized rubber and Plexiglas. Six other materials were tested using only the aqueous extraction protocol. There were two replicates of each of these tests for each of the three metals, giving an additional 36 tests. These test materials were Jade 403N adhesive, PVA adhesive, an enamel (Glidden water-based white Spred enamel), a sealer/primer (Parks), Marvel-seal (aluminized), and 3M spray adhesive 77. Materials were selected with the goal of finding some that would be very corrosive for one or more material, some that would be relatively non-reactive, and some that were intermediate. We hoped in this way to obtain a wide range of corrosion rates.

In the final experiments we chose the polarization resistance method since this has two advantages over the cyclic potentiodynamic polarization technique: (1) the R_p measurement requires a much shorter time to complete; and (2) the R_p technique exposes the test specimen to smaller voltages, and thus does not significantly change the surface of the specimen. We were also more interested in obtaining information on corrosion rate than on tendency towards localized pitting corrosion. Therefore this final group of experiments included only calculation of polarization resistance corrosion rates. Our two primary goals for the experiments were:

- (1) to see if any one extraction method consistently extracts more corrosive agents than other methods, and if the reproducibility of the three methods is comparable; and
- (2) to identify the range of corrosion rates for the three metals.

Results and discussion

Table 2 gives the data for polarization resistance corrosion rates from the final experiments. These data represent true replicates of the entire extraction process, as well as of the electrochemical testing procedure. The table also provides comparative data for the alternative extraction procedures.

Effects of extraction method

We had originally hypothesized that, for some of the test materials, the aqueous extraction procedures used for electrochemical tests might not extract all of the problematic compounds. However, looking at the low, mid and high values for each extraction method and each metal type (Tables 3, 4 and 5), it is clear that water extraction consistently produces the highest corrosion rates, indicating that it generally does extract most corrosive agents. Therefore water extraction is the best way to identify the worst possible corrosion that might be encountered. Analysis of variance showed that relative error differs for extraction method but not for metal type, and there is no interaction effect between extraction method and metal type.

In further work we shall continue to use the standard water-extraction method, since it consistently provides the worst-case scenario, which is needed if we are to eliminate the potentially worst materials. We might sometimes exclude from use materials that would not cause problems, given the period of exposure, amount used, or distance from the object; but that is better than accepting a material that in some circumstances could cause corrosion.

Ranges of corrosion rates

In industry there is a sliding scale for 'acceptable' corrosion rates, depending on the metal alloy and its intended use. For example, carbon steel is often considered unacceptable if its rate exceeds 10mil per year, stainless steel if its rate exceeds 2mil per year, a nickel-based alloy if its rate exceeds 0.1mil per year, and titanium if its rate exceeds 0.01mil per year. Since industry rates are based on specific uses and needs, acceptable rate limits will have to be determined for the conservation field by experimentation.

In some industries, much research has gone into identifying the expected ranges of corrosion rates for specific types of material. For example, the corrosion of steel encased within concrete structures has been well studied by the polarization resistance method. Through measurement of hundreds of values in laboratory experiments, it has been possible to map the expected values of corrosion rates for various environments. Similar tests have been done using real structures in field exposures; these values were found to agree very well with those obtained from laboratory experiments. Thus ranges have been identified that are considered valid and reliable for the types of environment typically encountered [47].

In our experiments, the corrosion rates for lead ranged from a low of 0.016mpy up to 8.252. The range for copper was much lower, 0.003 to 3.618; and the range for silver was even lower, zero to 0.145. This represents the same general conclusions as found with the Oddy test regarding the relative corrosion rates of these metals. In industry, where long-term exposure tests have been conducted, the same results have been found: lead is susceptible to rapid attack by organic acid vapors, copper to moderate attack, and silver is relatively immune [2].

We have found that the computed rates vary depending not only on the extraction method but also on the type of electrochemical test being used, and according to the method of curve-fitting employed. Thus, specifying exactly what was done is a crucial part of data reporting and interpretation for electrochemical testing. We concluded that because some of the curves may be non-linear, it is currently necessary to fit the curves manually, because our particular computer program and its default calculation method are deficient when applied automatically to all curves.

How well do the results of each test correspond with the known performance of materials? We know, for example, that the wood product giving the highest corrosion rate in electrochemical testing—oak—is well known to cause major problems in conjunction with museum objects [29]. It has

*Table 2 Polarization resistance corrosion rates (mpy)**

Solution †	Corrosion rates					
	Lead		Copper		Silver	
	A	B	A	B	A	B ‡
Cardboard, water	0.075	0.076	0.332	0.142	0.098	0.132
Cardboard, 50/50	0.410	0.366	0.144	0.098	0.008	0.081
Cardboard, MeOH	0.303	0.128	0.085	0.025	0.009	0.012
Oak, water	2.512	3.234	0.733	0.647	0.003	0.002
Oak, 50/50	0.818	1.659	0.165	0.121	0.003	0.001
Oak, MeOH	1.522	3.012	0.045	0.143	0.006	0.002
Medex, water	0.152	0.105	0.225	0.134	0.045	0.055
Medex, 50/50	0.129	0.201	0.035	0.055	0.005	0.000
Medex, MeOH	0.131	0.080	0.016	0.020	0.000	0.003
Hide glue, water	8.068	8.252	0.406	0.411	0.057	0.054
Hide glue, 50/50	6.151	3.494	0.132	0.260	0.145	0.067
Hide glue, MeOH	1.871	2.516	0.285	0.410	0.082	0.032
Paint, water	1.212	1.070	0.059	0.180	0.029	0.031
Paint, 50/50	0.622	0.252	0.452	0.552	0.013	0.010
Paint, MeOH	0.626	1.392	0.029	0.071	0.006	0.010
Rubber, water	0.034	0.064	0.009	0.005	0.026	0.000
Rubber, 50/50	0.917	0.511	0.012	0.018	0.008	0.012
Rubber, MeOH	0.171	0.016	0.184	0.041	0.020	0.000
Plexiglas, water	0.537	2.392	0.892	0.525	0.004	0.002
Plexiglas, 50/50	1.507	0.476	0.567	1.424	0.005	0.000
Plexiglas, MeOH	4.463	1.313	0.045	0.016	0.002	0.019
Jade glue, water	1.189	1.274	0.804	0.371	0.000	0.000
PVA glue, water	0.884	0.509	1.610	3.618	0.012	0.021
Enamel, water	2.030	5.013	0.165	0.218	0.030	0.084
Primer, water	0.165	0.172	0.139	0.025	0.010	0.003
Marvel-seal, water	0.815	0.702	0.019	0.062	0.000	0.000
3M spray 77, water	4.565	2.267	0.003	0.025	0.003	0.000

*Corrosion rate in mils per year (mpy) is the standard unit of measurement in the United States (1 mil equals 0.001 inch). Conversion to an equivalent metric corrosion rate is calculated as: 1 mpy = 0.0254 millimeter per year or 25.4 micrometers per year [46].

†Solutions used are water only; 50/50, half water and half MeOH; and MeOH only.

‡A arc B arc replicates.

been suggested that these problems are due to the emission of acetic (ethanoic) acid and other organic acids [15], sometimes exacerbated, as with all wood products, by the presence of insecticides.

Field tests to compare electrochemical testing methods with the actual performance of materials have been discussed in the corrosion literature. For example, Berke and Hicks found good correlation between polarization resistance tests of steel used in

conjunction with concrete and the visual appearance of the steel in field tests of three years duration [48]. Berke and Friel found good agreement in their comparison of electrochemical test results with long-term exposure tests on metal-coated steels used in marine and industrial atmospheres [49].

The only way we can be sure of the full range of corrosion rates for various metals, and how those ranges relate to long-term use of a material for

Table 3 *Electrochemical experiments: lead corrosion rates in mpy*

	Water	50/50	Methanol
Low	0.034	0.129	0.016
Mid	1.070	0.622	1.313
High	8.252	6.151	4.463
Water	n = 26		
50/50	n = 14		
Methanol	n = 14		

Table 4 *Electrochemical experiments: copper corrosion rates in mpy*

	Water	50/50	Methanol
Low	0.003	0.012	0.016
Mid	0.218	0.144	0.071
High	3.618	1.424	0.410
Water	n = 26		
50/50	n = 14		
Methanol	n = 14		

Table 5 *Electrochemical experiments: silver corrosion rates in mpy*

	Water	50/50	Methanol
Low	0.000	0.000	0.000
Mid	0.030	0.010	0.010
High	0.132	0.145	0.082
Water	n = 26		
50/50	n = 14		
Methanol	n = 14		

storage or exhibition cases, is to conduct the same type of experiments that have been done in industry. Thus a large number of materials will need to be tested, with replicate samples, to identify the full range of variability for each type of metal. Then, long-term exposure tests using the same materials will enable us to compare these numerical rates for each metal with the appearance of unacceptable levels of corrosion.

Our initial results point to the need for further research on specific questions. For example, some of the replicates for rubber in conjunction with silver did not show any measurable corrosion, which is unexpected. Further research should examine the sensitivity of the electrochemical test method to sulphur compounds and, if a problem is found, we may want to experiment with different extraction methods for certain materials.

Similarly, the corrosion rate for lead in conjunction with Plexiglas was not what we expected: it was much higher than with Medex particle board, and close to the results obtained for oak. The Plexiglas material we tested was new, and came with a protective Kraft paper glued to the surface, which was removed just prior to extraction. It is possible that chemicals in the adhesive remained on the Plexiglas surface and were subsequently dissolved. Certain adhesives are known to be corrosive. If further tests reveal this to be a problem, then electrochemical tests must be conducted using Plexiglas materials that have been cleaned by various methods and/or 'aged', to minimize contamination from protective coatings.

Conclusions

The advantages of the Oddy test which have caused it to gain widespread acceptance in the conservation field include ease of use and no special apparatus requirements. Thus it can be done by virtually any laboratory. However, our experiments confirm those of others who have found problems with reproducibility of results, due largely to variations in how the tests are conducted. Even if greater uniformity in test methods can be shown to improve reproducibility, the 28-day test period is longer than many laboratories are given to make a decision about the suitability of materials for storage or display. Therefore, in practice, many institutions conduct these tests for only two weeks, which may not be long enough to identify problematic materials; some materials are not tested at all, because of time constraints.

In industry, problems with exposure tests similar to the Oddy test have been recognized for many years. Electrochemical tests are now standard in industrial laboratories where materials must be tested for corrosivity in contact with metal components. Our experiments indicate that such tests could also be useful for the conservation field, and merit further investigation. One advantage of electrochemical tests is that results can be obtained relatively quickly (in a few hours). They also provide a quantitative result, which allows materials to be ranked and categorized clearly and objectively. The equipment needed is relatively inexpensive. Tests can be done with (re-usable) coupons of any alloy, so that the objects of interest can be closely matched. Once corrosion rate scales for various metals are established and correlated with a specific level of visual change, it could become routine to determine whether or not a test material will be damaging for a certain metal.

There are important factors to be considered. To assess true reproducibility, replication must be at

the level of the extraction process and not just the electrochemical testing procedure; water extraction will usually provide a worst-case result by extracting the majority of components that can cause corrosion; the polarization resistance test is the most appropriate for standard situations in which overall corrosion is of primary concern; at least two replicates are necessary for a reliable test, and more than two for materials with greater variability; and the accuracy of computer-generated curve-fitting procedures must be verified.

We hope to continue to analyze representative exhibit and storage materials, in order to build up a database of corrosion rates for different metal compositions. For certain types of material, if reproducibility is poor, it may be necessary to use more than two replicates as a matter of routine. Ideally, we would also like to corroborate our experimental results by checking them against results from controlled long-term exposure tests with some of the same test materials.

Although the Oddy test and the electrochemical test are different in approach, the fundamental process of corrosion testing is essentially the same for both. The corrosion process requires an electrolyte (water). The Oddy test puts a metal in a sealed container with 100% relative humidity, which allows volatile water-soluble contaminants to condense on a metal test sample and corrode the surface. It is the reaction with water that brings about corrosion; if water is absent, the emitted volatiles will not cause corrosion. The electrochemical test extracts the water-soluble contaminants from the material under test, achieving the same corrosion conditions as in the Oddy test, but producing a quantitative measure of the resulting degree of corrosion.

It is possible that the long-term heating which a material undergoes in the Oddy test may produce additional degradation products not detected in the electrochemical testing procedures. However, since exhibition and storage environments are not heated to the temperatures involved in the Oddy test, this should not be problematic. It is also possible that electrochemical tests may extract some components not volatile enough to be damaging under normal museum conditions. If so, some materials with unfavorable electrochemical test results might in fact be acceptable for some field applications. All of these issues require further trials of electrochemical testing methods, and comparison of results to real experience with the same materials after long-term exposure in an enclosed environment.

Electrochemical testing promises to provide the conservation field with a fast, quantitative, reliable and relatively inexpensive approach to ensuring the compatibility of case materials with metal artifacts, once corrosion rate scales for various metals are

established and correlated with a specific level of visual change. The next step in evaluating the potential of this approach will include experiments to elucidate the range and degree of reproducibility of results for a wide variety of products used in conjunction with the metals of interest, and to correlate electrochemical test results with the actual performance of materials in the field.

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Suppliers

Gamry Instruments corrosion measurement system:
Gamry Instruments, 734 Louis Drive,
Warminster, PA 18974, USA.

Test protocols: Corrosion Testing Laboratories, Inc.,
60 Blue Hen Drive, Newark, DE 19713, USA.
Protocols include: (1) CTL Standard Method
for Preparing Solid Samples for Chemical
Analysis or Corrosion Testing of Leachable
Ions; (2) Procedure for Performing Rapid/Slow
Potentiodynamic Polarization Scans.

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Résumé—*De nombreux matériaux utilisés dans le stockage et l'exposition d'objets de musée sont potentiellement corrosifs et ne devraient pas être utilisés à proximité de certains métaux. La méthodologie utilisée dans de nombreux musées pour vérifier la fiabilité de ces matériaux est le 'test Oddy'. Cependant, on a pu voir des rapports dans la littérature mentionnant des problèmes relatifs à la reproductibilité et aux contraintes de temps. Dans les industries de fabrication, où des problèmes analogues se sont posés, les tests électrochimiques ont remplacé les tests visuels. L'article relate des expériences mettant en jeu les matériaux de stockage en présence de plaques de plomb, cuivre, et argent. Les résultats laissent penser que le test de résistance de polarisation peut permettre une identification objective des matériaux dégradés. Cette méthode fournit une mesure qualitative de la corrosion relative à un matériau donné.*

Zusammenfassung—*Viele Materialien, die im Ausstellungs- und Depotbereich von Museen Verwendung finden, sind möglicherweise korrosiv und sollten nicht in der Gegenwart bestimmter Metalle verarbeitet werden. In vielen Museen wird für die Beurteilung der Eignung solcher Materialien der sog. 'Oddy-Test' herangezogen. Allerdings finden sich in der Literatur Berichte über Schwierigkeiten bei der Auswertung. Im industriellen Bereich, wo vergleichbare Probleme aufgetreten sind, wurden die auf einer visuellen Beurteilung basierenden Tests durch elektrochemische Versuche ersetzt. Im hier beschriebenen Artikel wird über Versuche mit solchen elektrochemischen Tests berichtet; es wurden typische im Museumsbereich verwendete Materialien in ihrer Wirkung auf Blei, Kupfer und Silber bewertet. Der Polarisationswiderstandstest etwa ist ein geeignetes Verfahren zur raschen und objektiven Erkennung möglicherweise schädlicher Materialien. Dieses Verfahren erlaubt eine quantitative Angabe der Korrosionsrate in milliinch pro Jahr für ein spezifisches Metall, das im Verbund mit einem anderen Material verwendet wird.*