Oddy testing protocol based on procedures at the
Indianapolis Museum of Art/Winterthur Museum (IMA/W)

Dates of Use: 2019 to present

I. Introduction

This Oddy testing protocol is based on procedures that were initially developed at the Indianapolis Museum of Art by Dr. Gregory Dale Smith and Kathleen Kiefer. In 2015, the Winterthur Museum began using the IMA protocol but integrated minor modifications that were influenced by research and testing conducted by Elena Torok, Joelle Wickens, and Samantha Owens in 2014-2015. In 2018, after discussion at the Materials Working Group annual meeting in NY, additional minor modifications were made in advance of the 2019 and 2020 MWG Round Robin Testing programs to ensure all participating groups could obtain comparable results. Since 2020, additional minor edits have been made, and are described in Appendix II.

This document is currently maintained by the Materials Working Group, Testing and Standards committee, Oddy Testing Focus Group. Contributors include: Beth Holford, Sarah Freshnock, Rachael Heyse, Julia Sybalsky, and Elena Torok.

II. General Overview of Test

a. The time estimate for execution of this test protocol as written is 118 minutes per test, including sample tests and control tests, photography and publishing of results in the

online Test Results Table maintained on the AIC Wiki. Procedural choices, such as the use of different washing methodologies, could increase or decrease this time.

Cleaning:	10 minutes
Test Preparation:	29 minutes
Test Assembly:	22 minutes
Test Disassembly:	10 minutes
Recordkeeping:	12 minutes
Coupon Photography:	19 minutes
Uploading Results:	17 minutes

b. Specifications for equipment and supplies needed to run this test are listed in **Appendix 1**. Unless necessary, these materials should not be substituted. In the case that a substitution must occur (a vendor no longer exists, a product is discontinued, etc.), this substitution should be documented in test results.
c. All tests are run in duplicate, i.e. a test consists of two stoppered test tubes containing sample material with associated coupons. Each test is assigned an identifying number that is written on an adhesive label on the outside of the tube. Replicates are labeled “A” and “B”.
d. Negative controls, also in duplicate, are included with every group of materials tested. A unique identifying number is also assigned to each control, and replicates are also labeled “A” and “B”.
e. Samples should be prepared for testing according to the Material Sample Preparation Guide presented on the AIC Wiki. If samples cannot be prepared according to these guidelines, this information should be documented in test results.
f. All testing details and results should be recorded in a database or spreadsheet such as the Template for Recording Results provided on the AIC Wiki.
g. Evaluation of metal coupons and the assignment of Permanent, Temporary, and Unsuitable ratings to sample materials is guided by the Standardized Corrosion Vocabularies.

III. Personal Protection Equipment (PPE)

a. Required PPE includes nitrile gloves, safety glasses, and a laboratory coat. A particle mask or respirator is also required for metal coupon preparation if access to a fume hood or some kind of modified enclosure is not possible.
b. No testing materials should be touched with ungloved hands, as skin oils can cause contamination of results.

IV. Materials and Equipment

A list with more detailed specifications can be found in **Appendix 1**.

a. Forced air oven
b. Precision Balance
c. Weighing paper/dishes

d. Borosilicate glass test tube, 75 mL, 200 mm (outer glassware)
e. Borosilicate glass culture tube, 0.75 mL, 50 mm (inner glassware)
f. Versilic peroxide-cured silicone stopper, size 27D
g. Parafilm ® M
h. Poly-temp PTFE tape
i. Metal coupons (silver, lead, and copper), each 8 mm x 2.5 mm
j. 3200 grit Micromesh
k. Pointed scoring tool
l. Scissors
m. Tweezers
n. Specimen forceps
o. Glass pipette
p. Templating Material (Archival Cardstock, Coroplast)
q. PCC-54 Enzymatic Detergent Concentrate
r. Glass beakers (1L, 150mL)
s. Test tube cleaning brush
t. Deionized water
u. Acetone
v. Kim wipes
w. Nitrile gloves
x. Glass plate
y. Test tube holders
z. Sample (approximately 2 g)

V. Washing Method for Glassware and Stoppers

a. Glass test tubes should be cleaned before testing using 5% PCC-54 Enzymatic Detergent Concentrate in water. Test tubes should be soaked in this solution for at least 20 minutes, rinsed thoroughly with water until suds are gone, and then triple rinsed with deionized water. Test tubes should be left to air dry, and must be completely dry before coupons, sample, etc. are inserted.
b. Glass culture tubes should be cleaned before testing using acetone. Submerge culture tubes in a glass jar with acetone for 5 minutes. Swish periodically. Remove culture tubes using tweezers, pour out excess acetone and place in a clean glass jar to dry.
c. Stoppers should be submerged/rinsed with deionized water before testing, and then wiped down using acetone on a Kimwipe.

VI. Sample Preparation

a. Prepare 2.0 +/- 0.1 g of sample material.
b. Sample preparation will vary by sample type. Samples should be prepared according to established MMA procedures found here.

VII. Metal Coupon Preparation

a. Immediately after receipt, metals should be removed from original packaging and moved to Corrosion Intercept bags. If a styrene box was supplied with the lead, it may be retained and used to provide support inside the Corrosion Intercept bags.
b. Cover the working space with blotter paper for ease of capturing metallic particulates.
c. Coupons may be cut to size (0.8 mm x 2.5 mm) and then polished singly, or polished as a larger strip (e.g. 2.4 mm x 100 mm) and then cut. Cut metal foils using scissors or a scalpel (lead only) that have been cleaned with acetone. If prepared singly, coupons may be cut to size ahead of time and stored in Corrosion Intercept bags until polishing can be done.
d. Polish both sides of each metal coupon or partial sheet evenly using 3200 grit Micromesh. Polishing should be done lengthwise (i.e. along the long dimension of the strip). While holding one end of the strip against the glass plate, polish from the center of the strip towards the end, and then rotate to repeat this action to the other end. Flip the strip over and repeat again. A video of the polishing process is available here.
e. The same piece of Micromesh can be used to polish coupons of the same metal but should never be used to polish coupons of a different metal.
f. The glass plate should be cleaned with acetone between polishings of different types of metals.
g. If necessary, for large sample runs, the silver and copper coupons can be polished and sealed in Corrosion Intercept and left overnight. However, the lead coupons must be polished on the day the test is to be set up and begun.
h. Use tweezers to dip each coupon in an acetone bath. Remove the coupon from the bath and immediately wipe/dab dry using a Kim wipe. Coupons should not be allowed to air dry or left to soak in the acetone bath for prolonged periods of time or because this has been observed to cause corrosion, particularly on lead.
i. Materials (e.g. blotter) with lead particulates or used to clean equipment with lead particulate should be disposed of according to your institution's hazardous materials policy.

VIII. Stopper Preparation

a. Cut three parallel 10 mm long x ~5 mm deep slits in the small end of each stopper using a scalpel blade that has been cleaned with acetone.
b. In order to streamline the stopper preparation, it may be helpful to create a template. Archival cardstock or Coroplast can be cut out to match the diameter of the stopper's smaller end. Draw and cut out a 10x10mm square in the center. The sides of this cut square will provide a template for cutting the outermost slits. Cut the third slit in the middle between the first two (center of the square). Rinse the cut stopper surface with acetone applied by KimWipe when finished.

IX. Test Tube Preparation and Assembly

a. Place the sample in the base of the 75 mL test tube.

b. Fill the culture tube with ~0.65 mL of deionized water using a pipette. Place the culture tube in the base of the test tube, next to the sample, using the specimen forceps. Place the prepared test tube into the test tube holder. From this point forward, care must be taken to limit rapid movement of the test tube, as water can easily spill from the culture tube.
c. Use tweezers that have been cleaned with acetone to insert each of the three coupons (Cu, Ag, Pb) into a slit in the small end of the stopper. Slits can be opened by pressing the curved sides of the stopper. Place the Pb coupon in the middle slit.
d. Insert the small end of the stopper (with coupons) into the top of the test tube. Make sure the metal coupons are not touching the sides of the glass or one another. Push the stopper in tightly.
e. Wrap approximately 10 inches of Poly-temp PTFE tape around the top of the test tube and stopper. The wrapping needs to be tight, because as the test tube heats up, the stopper will be pushed out of the tube. Wrapping the tape tightly will help keep the stopper in place.
f. Wrap Parafilm over and around the PTFE tape-covered stopper and test tube top. Again, ensure wrapping is tight and secure.
g. Label the exterior of each test tube with an adhesive white label to identify the sample inside, note A or B, and the testing start date.
h. Place test tubes in a test tube holder or large glass beaker so they can remain as upright and vertical as possible during testing.

X. Testing

a. Place test tubes in a forced air oven that has been pre-heated to 60^{o}C.
b. Leave test tubes in the oven for 28 days.
    - In the first few hours of testing, check to make sure no stoppers have popped open. If a stopper has popped but is still generally resting in its appropriate position at the top of the test tube, push it back in and re-secure the area with more PTFE tape and Parafilm. If the stopper has popped out of the test tube entirely and is resting on the shelf or floor of the oven, the test tube should be re-prepared. Any type of stopper popping event should be noted in results.
    - Every few days, a quick visual inspection of test tubes should be performed to confirm that approximately the same level of water is still present in the culture tubes. If water levels are low or non-existent, there is likely a leak between the stopper and test tube. In this event, remove the test tube from the oven, remove the stopper, add more water to the culture tube, re-seal the stopper back in its appropriate position, and then place the test tube back in the oven. Any type of water loss event should be noted in results.

XI. Coupon Assessment

a. After 28 days in the oven, remove test tubes and allow them to cool to room temperature.
b. Identify a clean, flat surface of the lab that has access to strong and consistent indoor lighting. Gather an Optivisor or magnifying loop and the standardized Corrosion Vocabularies produced by The Metropolitan Museum of Art. These illustrated glossaries describe commonly-observed corrosion phenomena, and will be used as reference to rate test results. They can be downloaded using the links below:

    Copper Corrosion Library
    Silver Corrosion Library
    Lead Corrosion Library

c. Assessment involves rating all non-control coupons as compared to the control coupons. Ratings are assigned to each metal as summarized below. In general, all coupons will receive one of three of the following ratings:

    "Permanent" rating: The material tested may be used indefinitely in the presence of art.

    - Coupons look similar to the controls.
    - Copper: Very slight reddening
    - Silver: Light white haze. Remnants of polishing compounds from some manufacturers can develop or appear as white splotches. This stock is generally returned to the manufacturer, however, if it makes it into a test, the white splotches are ignored.
    - Lead: Very slight darkening

    "Temporary" rating: The material is safe for use near but not in contact with art for up to six months.

    - Copper: Slight-to-extreme reddening, rainbow-like color change, formation of up to 20 black spots.
    - Silver: Development of a heavy white haze, yellow tarnish or orange haze, or very slight purpling.
    - Lead: Slight-to-extreme darkening, blue or rainbow tarnish, or thin yellow, orange, blue, white compacted corrosion, haze from slight crystal formation over the entire coupon, or heavier crystal formation at the interface with the stopper.

    "Unsuitable" rating: The material should not be used in contact with or near art and another material should be found.

    - Copper: Severe blackening or formation of a heavy haze.
    - Silver: Slight-to-extreme purple, rainbow, or black tarnish.
    - Lead: thick yellow, orange, blue, or white compacted corrosion, or white fluffy crystal formation.

d. To remove coupons from a test tube, first remove the Parafilm and PTFE tape and then carefully pull the stopper out. Flip the stopper over (large side down, with coupons pointing vertically upward) onto a clean, flat surface. Squeeze the sides of the stopper to open the slits, and use clean tweezers to carefully remove Cu, Ag, and Pb coupons. Place coupons on a white piece of paper.
e. Open control test tubes and examine control coupons first. If coupons in both test tubes are minimally corroded, meaning that the corrosion phenomena observed are at or below the threshold of those rated "P" in the Standardized Corrosion Vocabularies the control test is considered valid. If corrosion phenomena observed on any metal coupon in one or both control jars are rated "T", the control test is not valid. The corrosion phenomena present on the controls is still recorded in the test record, and individual metals for each associated sample test are described and rated according to the corrosion phenomena observed, but overall ratings for all sample tests in the batch must be retested.
f. Examine coupons from sample test tubes next. The corrosion phenomena observed on each coupon below the area that was inserted into the stopper are compared to the Standardized Corrosion Vocabularies and described separately for replicates "A" and "B" in the test record. Corrosion phenomena not present in the glossary should nevertheless be described and may be added to the glossary. The lowest of the ratings assigned to the three metals in the test is also the overall rating assigned to the material. In the event that there are moderate differences in the same corrosion phenomenon on "A" and "B" replicates, as long as the rating for both levels is still the same, the result is valid. However, if the more extreme corrosion pushes one coupon into the next rating category, or if the replicates exhibit different types of corrosion phenomena, then the sample should be retested.
g. Record test results using the supplied Template for Recording Results.

XII. Disassembly and Reuse of Test Materials

a. After testing is complete, disassemble tests and clean according to instructions in Section V above.
b. Samples should be discarded. Lead coupons should be disposed of according to your institution's hazardous waste policy.
c. Glassware should be reused in future testing, so long as it does not have breaks, cracks, or other signs of deterioration.
d. If reusing stoppers in future testing, it is recommended to do so only if they were associated with tests that yielded Permanent results. Stoppers should not be reused if associated with a test that yielded Unsuitable results.
e. It is unknown to what extent reuse of stoppers from Temporary tests will introduce contaminants into the test; more research is needed in this area. Until this data is available, any reuse of stoppers from Temporary tests should be tracked and limited (for example, up to a maximum of three "temporary" tests), and each test should include one replicate that is run with a new stopper.
f. All reuse of stoppers is clearly recorded in the test record.

g. Stoppers that begin to show any evidence of material breakdown (discoloration, shedding, powdering, etc.) should also be discarded.

XIII. Photography/Documentation of Coupons

a. After evaluation, Oddy test coupons with matching duplicate results and passing control jars are photographed with a color reference using two different types of lighting: diffuse lighting and glancing-angle lighting. Guidelines for Coupon Photography are provided on the AIC Wiki, here.
b. Test results and images are uploaded to the AIC Wiki Test Results Table. Instructions are published here.

Appendix I. Materials and Supplies

ITEM	VENDOR	QUANTITY	COST
Forced Air Oven, 104 L (3.7 cubic feet)	Varies. Examples: VWR 89511-412 Thermo Scientific: Heratherm	1	$4545
Veritas Precision Balance S303	Hogentogler S303	1	$429.00
VWR Weighing Paper 10.2×10.2 cm (4×4") pack of 500	VWR 12578-165	1	$89.92
Acetone, high purity (≥99.8%), 1L	Sigma Aldrich 34850	1	$107
PCC-54 Enzymatic Detergent Concentrate, 3L bottle	Thermo Scientific 72288	1	$144.54
Micromesh 3200 grit	Conservation Resources MM-3200	Varies according to number of tests needed	$7.00 per 6” x 6” sheet
Silver foil at least 99.9% purity and 0.1mm thick, annealed, 100cm^2	Fisher Scientific AA42317GH	Varies according to number of tests needed	$189
Copper foil at least 99.9% purity and 0.1mm thick, annealed, 100cm^2	Fisher Scientific AA42189GH	Varies according to number of tests needed	$322
Lead foil at least 99.998% purity and 0.1mm thick, 100cm^2	Fisher Scientific AA12051GH	Varies according to number of tests needed	$370
General Tools Scratch Awl tool 8.75"L x 3.75"W x 3.25"H (or any fine needle-pointed tool)	Amazon General Tools 818	1	$6.99

Fiskars All-purpose Scissors 8.25"L x 3"W	Amazon 116000-1005	1	$6.48
Scalpel handle (#3) with Surgical Grade Blades #11 20pcs	Amazon B091SQZWT3	1	$11.99
Cole-Parmer Essentials Stainless Steel Tweezers, Mini, Blunt Round Tips, Serrated Grip, 76mm	Cole Parmer UX-07398-30	1	$5.20
1ml Graduated 12/pack Glass Dropper Pipette with Rubber Cap	Walmart A2ZSCILAB	1	$9.99
Heritage Archival Bristol Board 8x10" 6pt. (Stopper cutting template)	Talas TPB570122	1	$7.00/ea
Cole Parmer Pyrex 1000-PACK Brand 1000 Low-Form Glass Beaker Starter Set; 50 to 1000 mL, 5/pk	Cole Parmer UX-34502-90	1	$53.00
Eisco™ Nylon Labware Brushes 120 x 50 mm brush head	Fisher Scientific Eisco CH0220E	1	$5.15
Kimberly-Clark Professional™ Kimtech Science™ Kimwipes™ Delicate Task Wipers, 1-Ply box of 286 wipes	Kimberly-Clark Professional™ 34120	1	$12.54
Kimberly-Clark® Safeskin Purple Nitrile Exam Gloves, Medium, Purple, Box Of 100	Kimberly Clark 55082	1	$17.00
10 in. x 12 in. x 0.09375 in. Clear Glass Plate	Home Depot 599019	1	$4.78
Cotton Blotter Paper 19" x 24"	Talas TPB139003	1 per round of tests; continual stock needed	$5.95
VWR Aluminum test tube holder 24 x 26mm (fits 24 tubes)	VWR 89259-898	1	$42.61/ea
White Laser Mailing Labels (30 labels, 1 in. x 2 5/8 in. per sheet) - 750	Avery 5260	1	$9
Stainless steel specimen forceps, 305 mm, 12" long	Varies. Example: VWR 82027-382	1	$40.28
Kimax test tubes, Type 1 Class A borosilicate glass, 75 mL, 25 x 200 mm OR VWR test tubes, Type 1 Class A borosilicate glass, 75 mL, 25 x 200 mm	VWR 89001-432 OR VWR 10545-930	1 pack of 24 1 pack of 48	$85.70 $81.91
Saint Gobain - Versilic silicone stoppers, size 27D	US Plastics Corp. 76132	1 pack of 10	$27.12
Culture tubes, disposable, borosilicate glass, 0.75 mL, 6 x 50 mm	Varies. Examples: VWR 47729-566 OR	case of 2,000 Pack of 72	$266.20 $60

	VWR 89001-454		

Appendix II. Changes to this Protocol

In March 2024 this protocol was updated by the Materials Working Group to:

- Add Appendix II.
- Add the estimated time needed to complete the protocol.
- Replace several appendices with links to resources outside the protocol: Material Sample Preparation Guidelines, standardized Corrosion Libraries, Template for Recording Results, and Guidelines for Coupon Photography.
- Extend the list of Supplies and Materials to be more inclusive of items referenced in the protocol.
- Clarify steps in the washing procedure.
- Add a +/- range to the sample size specified.
- Change the coupon size to 0.8 mm x 2.5 mm to
- Include an option to prepare larger pieces of metal, then cut them down to coupon size.
- Clarify steps in the polishing process.
- Add options to safely store partially-prepared coupons for short periods of time.
- Describe the use of a template in preparing stoppers.
- Adjust the coupon assessment language to more closely match the Corrosion Libraries.
- Add language describing reuse of stoppers and associated risk of contamination.
- Add language describing photography of coupons and upload of results to the AIC Wiki.
- Update Appendix I, Materials and Supplies to:
    - Generalize the metal specification, setting lower limits for purity and thickness based on the previous protocol, while updating the product numbers listed to match what is currently in use at the Metropolitan Museum. This allows users the option of choosing a purer/thinner metal that is consistent in its thickness and size, metal to metal. At the time of writing, a modest savings was also associated with this change.
    - Provide alternate vendors for specific items;
    - Include 2024 pricing.