Materials Working Group: Materials Testing & Standards
Round Robin 2020

Oddy Test Protocol
Modified Indianapolis Museum of Art (IMA)/Winterthur Variant

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 MWG Round Robin to ensure all participating groups could obtain comparable results. In 2020, the protocol was edited to provide more detail and clarity and replace materials that were no longer commercially available. This document is currently maintained by Elena Torok.

I. Test Overview

a. All specifications for equipment and supplies needed to run this test are listed in **Appendix 1**. 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.

b. 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".
c. 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".
d. Samples should be prepared for testing according to the Material Sample Preparation Guide prepared by the Metropolitan Museum of Art (**Appendix 2**). If samples cannot be prepared according to these guidelines, this information should be documented in test results.
e. All testing details and results should be recorded in a custom-built database or spreadsheet prepared by the Metropolitan Museum of Art (**Appendix 3**).
f. Evaluation of metal coupons and the assignment of Permanent, Temporary, and Unsuitable ratings to sample materials is guided by the Glossary of Corrosion Phenomena compiled by the Metropolitan Museum of Art (**Appendix 4**).

II. 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.

III. Materials and Equipment

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

a. Forced air oven
b. Borosilicate glass test tube, 75 mL, 200 mm (outer glassware)
c. Borosilicate glass culture tube, 0.75 mL, 50 mm (inner glassware)
d. Versilic peroxide-cured silicone stopper, size 27D
e. Parafilm ® M
f. Poly-temp PTFE tape
g. Metal coupons (silver, lead, and copper), each 10 mm x 25 mm
h. 3200 grit Micromesh
i. PCC-54 Enzymatic Detergent Concentrate
j. Deionized water
k. Acetone
l. Kim wipes
m. Nitrile gloves

n. Glass plate
o. Sample (approximately 2 g)

IV. Pre-Testing Washing and Cleaning Methods

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.
c. Stoppers should be rinsed before testing with deionized water, and then wiped down using acetone on a Kimwipe.

V. Metal Coupon Preparation

a. Cut metal foils to 10 mm x 35 mm coupons using scissors that have been cleaned with acetone.
b. Polish both sides of each coupon evenly using 3200 grit Micromesh. Polishing should be done horizontally across the coupon; while holding one end of the coupon against the glass plate, polish from the center of the coupon towards the end, and then rotate to repeat this action to the other end. Flip the coupon over and repeat again.
    - The same piece of Micromesh can be used to polish two coupons of the same metal but should never be used to polish coupons of a different metal.
    - The glass plate should be cleaned with acetone between polishings of different types of metals.
    - If necessary, for large sample runs, the silver and copper coupons can be polished and sealed in a clean glass jar and left overnight. However, the lead coupons must be polished on the day the test is to be set up and begun.
c. Use tweezers to dip each coupon in an acetone bath. Remove coupon from the bath and immediately pat dry using a Kim wipe. Coupons should not be left to soak in the acetone bath for prolonged periods of time or allowed to air dry.

VI. Stopper Preparation

a. Cut three parallel 10 mm x ~5 mm deep slits in the small end of each stopper using a scalpel blade that has been cleaned with acetone.
b. Stoppers may be reused in testing ONLY if their prior use was in a test tube that contained a sample that was evaluated to be Permanent or Temporary. Stoppers used with samples evaluated to be Unsuitable should be discarded. Stoppers that begin to show any evidence material breakdown (discoloration, shedding, etc.) should also be discarded.

VII. Sample Preparation

a. Prepare 2.0 g of sample material.
b. Sample preparation will vary by sample type and should be prepared according to established MMA procedures found in **Appendix 2**.

VIII. 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. 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. 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 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.

IX. 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 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.

X. Assessment at Completion of Testing

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 a printed copy of the MMA's Glossary of Corrosion Phenomena (**Appendix 4**), which will be used as reference while conducting coupon examination.
c. Assessment involves rating all non-control coupons as compared to the control coupons. Ratings are assigned to each metal as instructed in the MMA's Glossary of Corrosion Phenomena (**Appendix 4**). 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: Thin white haze
- Lead: Slight darkening
- For silver, 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.

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

- Copper: Slight reddening, yellowing, or rainbow-like color change, formation of up to 20 black spots
- Silver: Slight yellowing, purpling, or darkening.
- Lead: Darkening, yellow/olive tarnish, haze from slight crystal formation over the entire coupon, or heavier crystal formation at the interface with the coupon holder.

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

- Copper: Severe blackening or severe reddening or matte-textured surface.
- Silver: Severe color change to dark purple, yellow, or black.
- Lead: White fluffy crystal formation of any size.

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 MMA's Glossary of Corrosion Phenomena (**Appendix 4**), 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 MMA's Glossary of Corrosion Phenomena (**Appendix 4**) 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.

XI. Disassembly and Reuse of Test Materials

a. After testing is complete, disassemble tests and clean according to instructions in Section IV above.
b. Samples should be discarded.
c. Glassware should be reused in future testing, so long as it does not have breaks, cracks, or other signs of deterioration.
d. Stoppers can be reused in future testing, but only if they were associated with tests that yielded "P" or "T" results. Stoppers should not be reused if associated with a test that yielded "U" results OR are showing signs of deterioration (discoloration, shedding, etc.).

XII. Photographing Oddy Test Coupons

a. After evaluation, Oddy test coupons should be photographed according to established MMA standards. Test results and images should be uploaded to the AIC Wiki.
b. To ensure standardization in the 2020 Round Robin, all participants should mail coupons to Alayna Bone at the MMA for photography. Coupons should be individually placed in corrosion intercept bags prior to mailing.

Appendices

Appendix I. Materials and Equipment

Materials Used in IMA/Winterthur Oddy Testing (figures last updated February 2020)			
ITEM	**VENDOR**	**QUANTITY**	**COST**
Forced Air Oven, 104 L (3.7 cubic feet)	VWR 89511-412	1	$2,871.13
Acetone, high purity (≥99.8%), 1L	Sigma Aldrich 34850	1	$84.30
PCC-54 Enzymatic Detergent Concentrate, 3L bottle	Thermo Scientific 72288	1	$86.00
Micro-Mesh (3200 grit)		1	
Alfa Aesar silver foil, 0.127 mm (0.005 in) thick, annealed, 99.9% (metals basis), 100 x 100 mm	VWR AA11440-GH	Varies according to number of tests needed	$82.86 each
Alfa Aesar copper foil, 0.127 mm (0.005 in) thick, annealed, 99.9% (metals basis), 20 cm x 20 cm	VWR AA13380-CU	Varies according to number of tests needed	$42.74 each
Alfa Aesar lead foil, 0.1 mm (0.004 in) thick, >99.998% (metals basis), Puratronic®, 50 x 50 mm	VWR AA12051-FI	Varies according to number of tests needed	$96.22 each
Avery White Laser Mailing Labels (30 labels, 1 in. x 2 5/8 in. per sheet)	Avery 5260	1	$17.99
Stainless steel specimen forceps, 305 mm, 12” long	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 VWR 10545-930	1 pack of 24 1 pack of 48	$85.70 $81.91
Saint Gobain - Versilic silicone stoppers, size 27D	LabPure D1069813		
Culture tubes, disposable, borosilicate glass, 0.75 mL, 6 x 50 mm	VWR 47729-566	1 case of 2,000	$266.20

Appendix 2. Material Sample Preparation Guide

Material	Proposed Cutting Method
Board - composite (eg. Corian, drywall)	Cut with universal blade band saw into 0.5” cubes. Use utility knife to produce smaller pieces to make 2.0g.
Board - natural (eg. wood, cotton)	Cut with universal blade band saw into 0.5” cubes. Use utility knife to produce smaller pieces to make 2.0g.
Board - plastic/polymeric/synthetic	Cut with universal blade band saw into 0.5” cubes. Use utility knife to produce smaller pieces to make 2.0g.
Carpet - natural/non-synthetic fiber	Cut with scissors or utility knife into 1 x 1” squares. To reach 2.0 g, cut pieces that represent the sample’s composition—example: the correct ratio of carpet material, blended fibers in carpet, and base of carpet.
Carpet - plastic/polymeric/synthetic fiber	Cut with clean scissors or utility knife into 1 x 1” squares. To reach 2.0 g, cut pieces that represent the sample’s composition—example: the correct ratio of carpet material, blended fibers in carpet, and base of carpet.
Coating - floor (e.g. stain, anti-wear, anti-slip)	Follow manufacturer’s instructions regarding dilution; if to be used dry, paint out on Mylar® and cure per manufacturer’s guidelines; if to be used wet, use 0.5mL of liquid in lieu of water.
Coating - grease, oil, wax	Into a 5mL beaker, weigh 2.0 g of sample. Carefully place into the sample jar. Other borosilicate vessels that fit are acceptable as long as they are no more than 4cm tall.
Coating - paint & primer	Paint material onto a Mylar® sheet and spread to a thickness that reflects how it will be used in the museum. Cure the material according to manufacturer’s instructions. After curing, cut the painted Mylar® into 1.5” wide strips and weigh to 2.0 g, taking the weight of the Mylar® into account by subtracting it from the total sample weight. Put a small piece of Mylar® at the bottom of the jar if there’s concern about the material sticking to the glass. Roll the sample strips into a coil with the sample material facing inward, and place in the bottom of the jar.

Coating - protective (e.g. anti-UV, -abrasion, -tarnish)	Paint the material onto a Mylar® sheet and spread to a thickness that reflects how it will be used in the museum. Cure the material according to manufacturer's instructions. After curing, cut the painted Mylar® into 1.5" wide strips and weigh to 2.0 g, taking the weight of the Mylar® into account by subtracting it from the total sample weight. Put a small piece of Mylar® at the bottom of the jar if there's concern about the material sticking to the glass. Roll the sample strips into a coil with the sample material facing inward, and place in the bottom of the jar.
Fabric - batting & padding	Cut material with clean fabric scissors into 1 x 1" squares. To reach 2.0 g, cut small segments from a 1" square.
Fabric - book cloth	Cut material with clean fabric scissors into 1 x 1" squares. To reach 2.0 g, cut small segments from a 1" square.
Fabric - exhibition/woven	Cut material with fabric scissors into 1 x 1" squares. To reach 2.0 g, cut small segments from a 1" square.
Fiber or Thread	Cut the length of sample that weighs 2.0 g. Wind it loosely and neatly around two gloved fingers, remove, and place in the bottom of the sample jar.
Inorganic - (e.g. fillers, salts, rocks)	Keep the sample as is or prepare it as it would be used in the museum. For example, if you are testing salts, do not grind them further unless that's how they are used in the museum setting. Weight 2.0 g of sample.
Metal - mechanical fastener	Place whole fastener in jar.
Paper-based (e.g. folder, cardboard, sheet)	Cut material with clean scissors into 1 x 1" squares. To reach 2.0 g, cut pieces from a 1" square.
Paper-based, Filled (e.g. fillers such as silica gel, zeolites, alumina)	Cut material with clean scissors into 1 x 1" squares. To reach 2.0 g, cut pieces from a 1" square.
Paste - filler/binder mixture (e.g. plaster, acrylic spackle, non-paint)	Extrude onto a Mylar® sheet. Spread to a thickness that reflects how it will be used in the museum setting. Cure material per manufacturer's suggestion. After cured, peel the material from the Mylar® if possible and weigh to 2.0 g. If the material cannot be freed from the Mylar®, remove excess Mylar® and weigh the material to

	2.00 g, taking the weight of the Mylar® into account by subtracting it from the total sample weight.
Polymer - adhesive - caulk or sealant	Extrude onto a Mylar® sheet. Spread a thickness that reflects how it will be used in the museum setting. Cure per manufacturer's instructions. After curing, peel the material from the Mylar® if possible and weigh to 2.0 g. If the material cannot be freed from the Mylar®, remove excess Mylar® and weigh the material to 2.0 g, taking the weight of the Mylar® into account by subtracting it from the total sample weight.
Polymer - adhesive - glues - liquid (e.g. acrylics, wood glues, starches)	Extrude adhesives onto Mylar® sheeting in a thickness that reflects the actual material application thickness. Cure per manufacturer's instructions. Cut the sample into 1 x 1" squares. Weigh the dried material on Mylar®, taking into account the weight of the Mylar® attached to the sample. To reach 2.0 g, cut pieces from a 1 x 1" square.
Polymer - adhesive - heat activated (e.g. hot melt, heat set)	Extrude 2" strips of melted material onto aluminum foil. Allow to cool. Peel from aluminum foil and weigh out 2.0 g of sample. If material does not remove from foil, repeat on Mylar® and account for the weight of the Mylar® in weighing the sample.
Polymer - adhesive - pressure-sensitive	Cut 2" lengths of tape, taping the adhesive sides, to the backed sides to form a small 2.0 g block of tape. Place the sample onto Mylar® to protect the jar from the adhesive
Polymer - adhesive tape - double sided	Fold the tape onto itself "accordion style" every 2" while removing the backing. Put 2.00 g sample on a piece of Mylar® to protect the jar from the adhesive, and carefully place in the bottom of the jar.
Polymer - adhesive tape - single sided	Cut 2" lengths of tape, taping the adhesive sides, to the backed sides to produce a small 2.0 g block of tape. Place the sample onto Mylar® to protect the jar from the adhesive
Polymer - block/bulk/pellet	If material comes in a block, cut into 0.5" cubes using a band-saw. If the material comes in small pellets that fit in the sample jar, use whole uncut pellets. To reach 2.0 g, shave material from one cube or pellet using a utility knife.

Polymer - foam - building insulation	For dense foam, cut with universal band saw into 0.5” cubes. To reach 2.0 g, remove material from one cube using a utility knife. For soft foams, cut material with clean scissors into a 1.5” wide strip that weighs 2.0 g. Compress the strip into a roll, and insert into the bottom of the jar. Make sure the foam does NOT touch the metal coupons when it expands.
Polymer - foam - non building insulation	For dense foam, cut with universal band saw into 0.5” cubes. To reach 2.0 g, shave any excess material on only one cube using a utility knife. For soft foams, cut material with scissors into a 1.5” wide strip that weighs 2.00 g. Compress the strip into a roll, and insert into the bottom of the jar. Make sure the foam does NOT touch the metal coupons when it expands.
Polymer - foam sealant	Cut material with clean scissors into a 1.5” wide strip that weighs 2.0 g. Compress the strip into a roll, and insert into the bottom of the jar. Make sure the foam does NOT touch the metal coupons when it expands.
Polymer - gasket	Cut material with clean scissors into 2” length strips.
Polymer - glove	Cut material with clean scissors into 1 x 1” squares. To reach 2.0 g, cut pieces from a 1” square.
Polymer - membrane (<1mm thick)	Cut material with clean scissors into 1 x 1” squares. To reach 2.0 g, cut pieces from a 1” square.
Polymer - sheet (>1mm thick)	Cut material with clean scissors into 1 x 1” squares. To reach 2.0 g, cut a minimal amount of strips from a 1” square.

Appendix 3. Test Record Spreadsheet

This appendix is supplied separately as an Excel Document. Digital copies are also are available from Julia Sybalsky, jsybalsky@amnh.org.

Appendix 4. Glossary of Corrosion Phenomena

A summary of corrosion phenomena is provided below, along with their associated ratings. For Round Robin 2020, the illustrated glossary will be supplied separately to testers, and will be available from Eric Brietung, Eric.Breitung@metmuseum.org. In future versions of the protocol, it will be included here.

Copper

Very slight red tarnish (P/T)

Slight red tarnish (T)

Red tarnish (T)

Extreme red tarnish (T)

Rainbow tarnish (T)

Light haze (T): specular reflection at most angles. Diffuse at glancing angles.

Heavy haze (U): diffuse reflection at any angle

Black corrosion spots (T/U)

Silver

White film/haze (NA/T): NA if it looks like a splotchy manufacturer defect. T if it looks like corrosion from the sample.

Orange/yellow film/haze (NA/T): NA if it looks like manufacturer defect. T if it looks like corrosion from the sample.

Yellow tarnish (T)

Rainbow tarnish (T/U)

Light purple tarnish (T/U)

Heavy purple tarnish (U)

Black tarnish (U)

Lead

Very slight darkening (P/T)

Slight darkening (T)

Darkening (T)

Extreme darkening (T)

Blue tarnish (T)

Blue compacted corrosion (T)

Rainbow tarnish (T)

Violet tarnish (T)

Thin orange compacted corrosion (T)

Thick orange compacted corrosion (U)

Thick/light white compacted corrosion (T/U)

- previously called 'light white haze)'

Thin Yellow/green compacted corrosion (T)

Thick Yellow/green compacted corrosion (U)

Fluffy white crystals (U)