

# Necessity is the mother of invention—DIY iron (II) test paper

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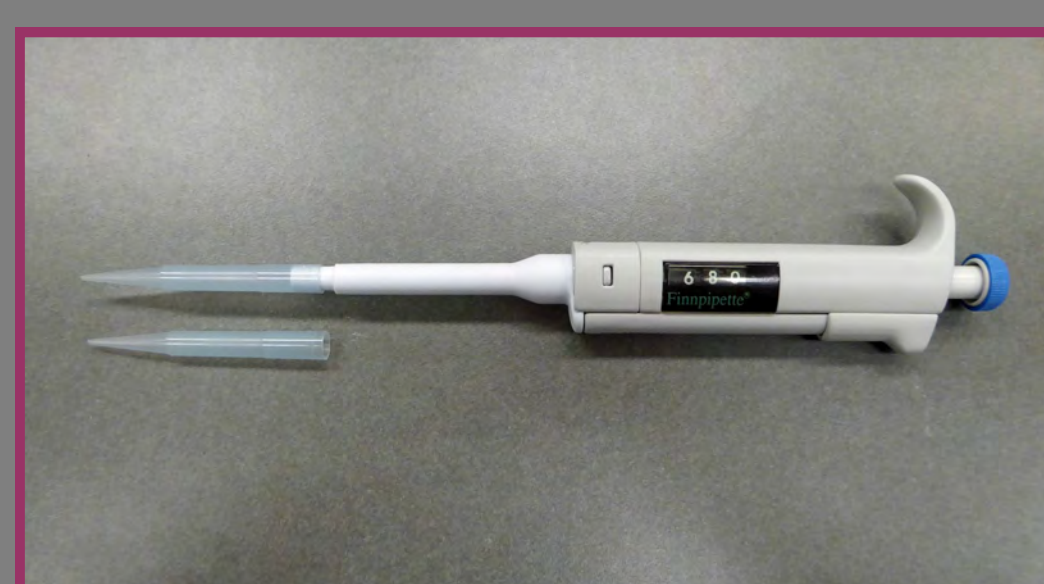
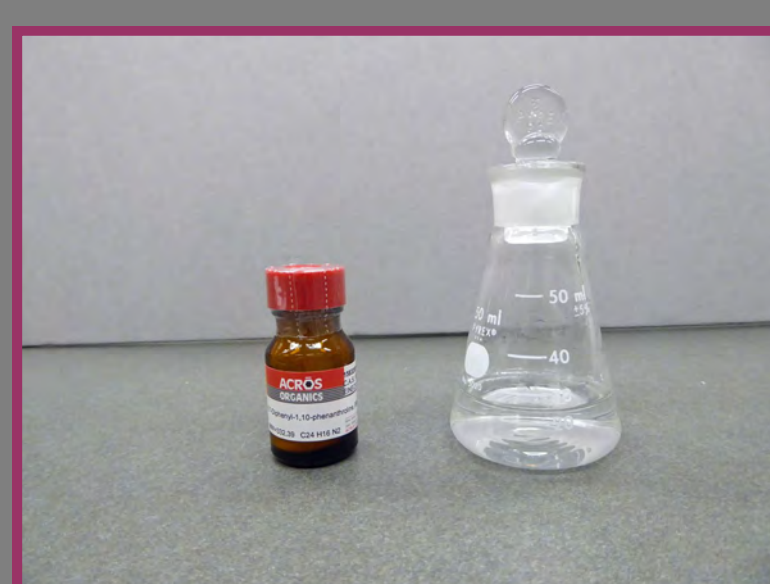
## BACKGROUND

One of the tools used by conservators at the Library of Congress for detecting corrosive iron (II) ions is a non-bleeding indicator paper developed by researchers working at the Netherlands Cultural Heritage Agency (RCE)<sup>2,3,4,5</sup>. This simple qualitative test consists of Whatman filter paper impregnated with an iron-sensitive bathophenanthroline solution. A small strip of test paper is dampened and placed against media to establish the presence or absence of iron species. When iron(II) ions are present, the test paper turns magenta to varying degrees, corresponding to the amount of iron (II) ions in the sample that bind with the bathophenanthroline indicator. The iron (II) indicator paper is available commercially in small strips<sup>6</sup>. Our current research group<sup>7</sup> needed larger pieces of the indicator paper, so we developed a method for making the test paper in-house.

### MAKING THE INDICATOR PAPER

Equipment & materials to make approximately 36 iron (II) test paper discs

0.04 g bathophenanthroline crystals (4,7-diphenyl-1,10-phenanthroline, 99%)  
25.0 ml ethanol  
Erlenmeyer flask with stopper or other container  
Micro-pipette (such as Finnpiquette® Micro-pipette & tips)  
90mm filter paper circles (Whatman 1001-090 Qualitative Filter Papers)  
6 watch glasses (90mm dia)  
6 glass or polystyrene petri dishes with covers (140mm dia)  
Fume hood; scale  
Stainless steel or plastic tweezers  
Corrugated paper board or polyethylene strips



### Procedure

1. Make a 1.6% solution by mixing 0.04g bathophenanthroline crystals into 25ml ethanol and stirring until dissolved.
2. Place each 90mm watch glass into a glass or polystyrene petri dish, then center a 90mm Whatman filter paper circle on each watch glass.
3. Place two stacks of corrugated board on opposite sides of the polystyrene petri dish.
4. Pipette 0.5ml solution slowly onto each filter paper circle. Start in the center and work towards the outer edge in concentric motions. Try to disperse the solution so it wicks evenly across the filter paper. Immediately place the cover on the petri dish to limit uneven evaporation. The corrugated board will support the cover over the petri dish, so it does not touch the filter paper or watch glass. Allow the solution to spread across and saturate the filter paper for at least 10 minutes.



5. When the test papers are dry, indicate the side of solution application by marking the edge lightly with pencil.
6. Examine the dried filter papers under long-wave UV (365 nm) and eliminate any papers that appear uneven.



Uneven distribution

7. Store the indicator papers in a covered petri dish.

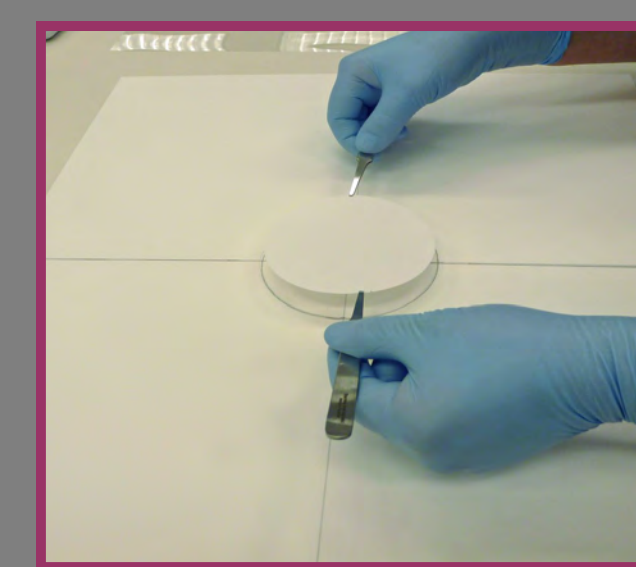
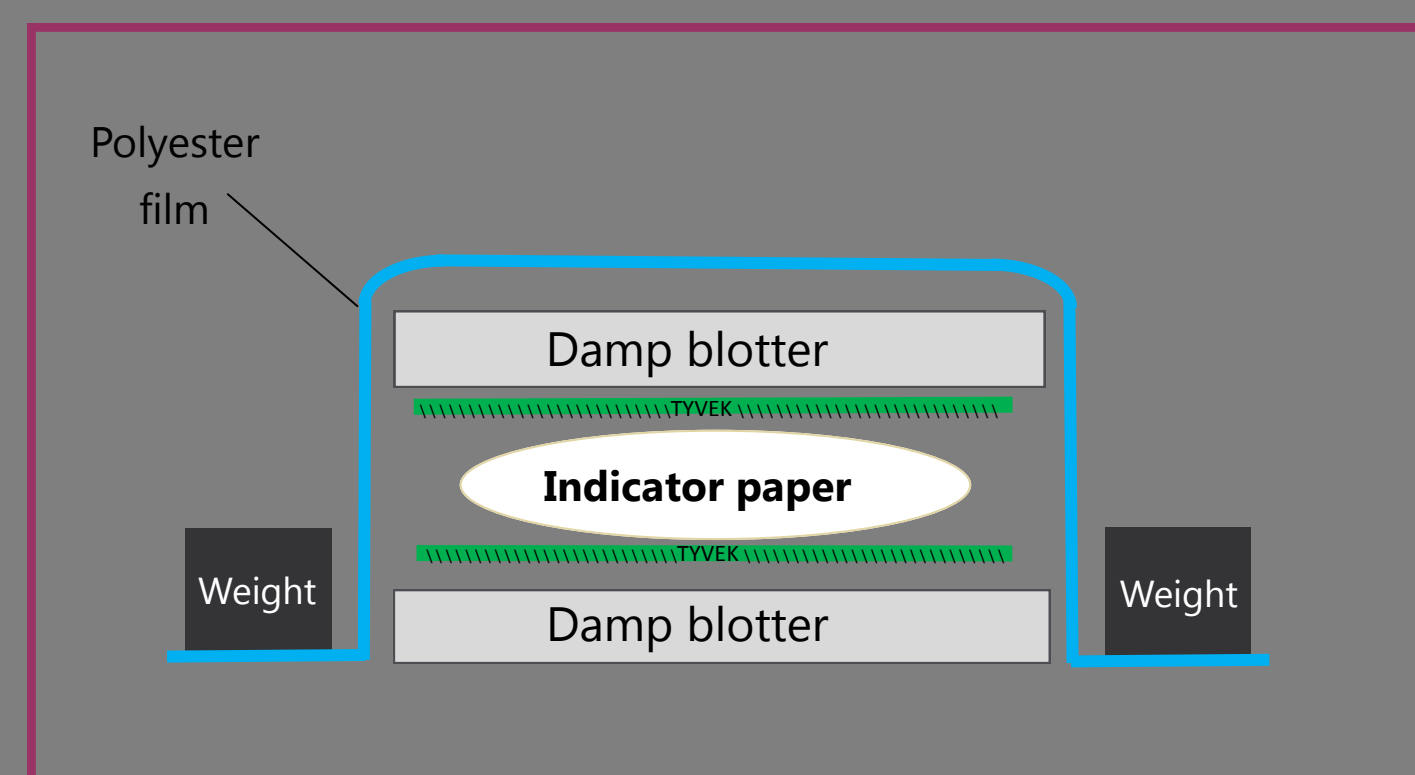
### USING THE INDICATOR PAPER

Equipment and materials

Bathophenanthroline-impregnated filter paper circles\* (stored in clear polyester sleeve).  
\*Note which side has writing; the side opposite of the pencil notation holds the most even application of bathophenanthroline indicator solution as seen under UV radiation.  
Two sets of stainless steel or plastic tweezers  
Finnpiquette® Micro-pipette & tips  
Small beaker of deionized water  
Nitrile gloves  
Timer

### Procedure

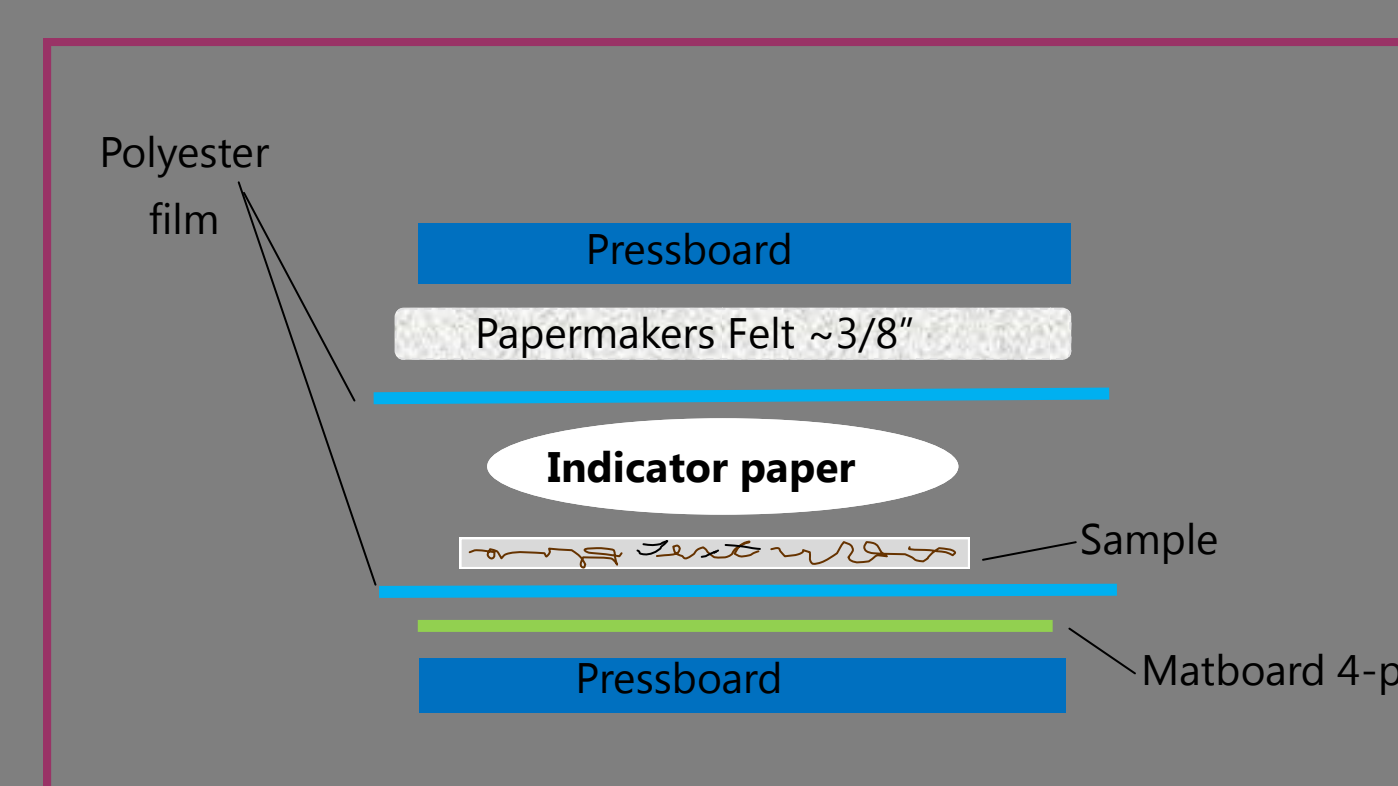
1. Prepare a working surface by cleaning a sheet of clear polyester film (about 8" x 8") with ethanol.
2. Place a discarded filter paper circle beneath the polyester work surface to use as a target.
3. Prepare a humidity pack of damp towels layered between smooth non-woven high-density polyethylene (Tyvek®). Cover the damp pack with clear polyester film held down with weights along the edges.
4. Place a humidity indicator card between Tyvek® sheets. Allow humidity to reach 80-90% RH (about 15 minutes) before starting to humidify the test paper.
5. Placing the marked side of the test paper DOWN in the damp pack, humidify the test paper circles between the Tyvek® sheets for 15 minutes at 80-90%RH.
6. Load the pipette with ~680 µl (or other pre-determined amount) of deionized water and dispense a small circular puddle on to the polyester sheet, centered over the target.
7. Use both sets of tweezers to place a humidified test paper circle onto the center of the deionized water (writing side face DOWN). Start a timer for 1:30 minutes. During the first 45 seconds, lightly push down any edges of the paper that curl with tweezers, to aid even saturation. After 45 seconds, flip the paper circle over for an additional 45 seconds (marked side will be face UP) to ensure even wicking of the water into the paper.
8. After 1:30 minutes, lift the paper circle with both sets of tweezers and place it onto the clear polyester sheet designated for iron testing.
9. The test paper circle is now ready to be placed in contact with the sample.



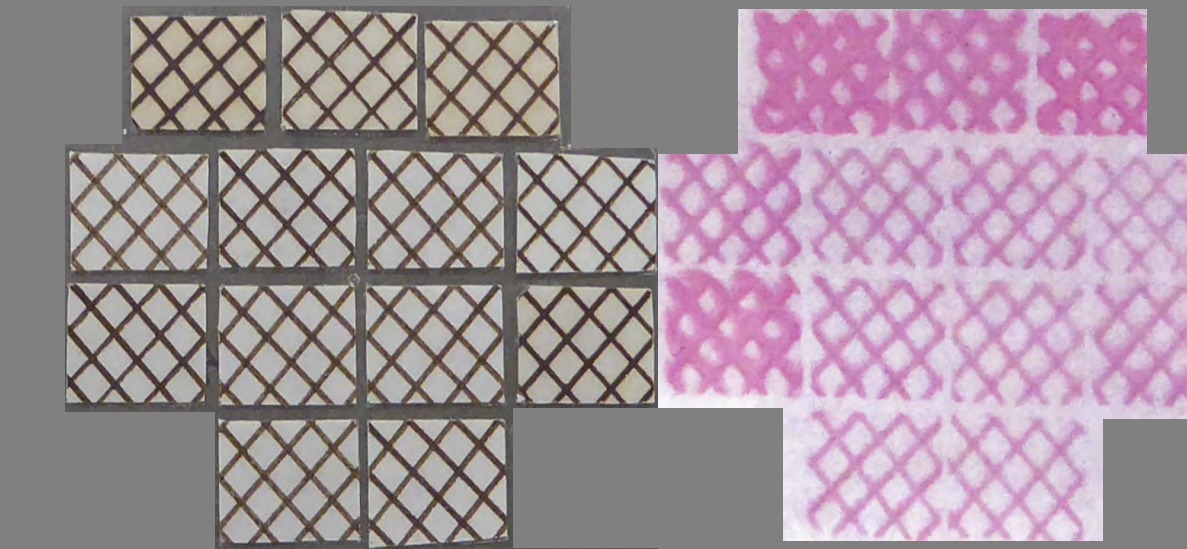
### APPLYING PRESSURE

Our experiments with various types and positions of weights demonstrated that hand weights did not result in sufficient, consistent pressure across the surface area of the iron (II) test paper.

To achieve the necessary contact between the test paper and samples taken, we used a screw press (e.g. Vaggelli book press with platen approx. 15.5 x 19.5 in.). The humidified test paper was placed in contact with the sample, and both were then placed between polyester film, in a stack as shown, and inserted quickly into the press.



The application of consistent pressure helps differentiate between various iron gall ink samples as seen by the intensity of the pink color of the iron (II) test paper.



### TIPS

- Consistency is important; the same person should operate the micropipette.
- Note which side is marked; the side opposite the pencil notation holds the most even application of bathophenanthroline indicator solution as seen under UV radiation. Note which side is marked and place the marked side DOWN to begin.
- To achieve greater consistency in the pressure used for the group of samples, the press was closed by the same person to a point marked on the press. Prior experimentation had determined how much to screw down the press. Too much pressure causes lateral spread of iron (II) and its bound dye, resulting in a muddled impression.
- The samples were pressed for 2 minutes, then removed, uncovered, and allowed to dry.

### References

<sup>1</sup> Currently, Cyntia Karnes is a paper conservator in private practice, Toronto, CA. She worked at the Library of Congress 2003-2014.

<sup>2</sup> Johan Neevel and Birgit Reissland, Bathophenanthroline indicator paper: development of a new test for iron ions 2005, *Papier Restaurierung* 6, no.1 (2005), 31.

<sup>3</sup> Elmer Eusman, "Aqueous treatment effects of iron gall ink: Monitoring iron migration with the Fe (II) indicator test," in *The Broad Spectrum: the art and science of conserving colored media on paper*, ed. Harriet K. Stratis et al. (London: Archetype Publications Ltd, 2002), 122-127.

<sup>4</sup> Sylvia Albro, Julie Biggs, Claire Dekle, Mary E. Haude, Cyntia Karnes, and Yasmeen Khan, "Developing Guidelines for Iron-Gall Ink Treatment at the Library of Congress," *The Book & Paper Group annual* 27 (2008), 129-165.

<sup>5</sup> Claire Dekle and Mary E. Haude, "Iron-gall ink treatment at the Library of Congress: old manuscripts—new tools," *The Book & Paper Group annual* 27 (2008), 15-26.

<sup>6</sup> University Products, US; Preservation Equipment Limited, UK.

<sup>7</sup> Current group is evaluating ethanol-modified solutions for treatment of iron gall ink.

### Acknowledgements

Elmer Eusman, Chief of Conservation Division, Library of Congress Preservation Directorate

Allison Brewer, Library of Congress Conservation pre-program intern 2013; currently, she is a first year student in the Buffalo State College Art Conservation Program