

Procedure for making iron gall ink



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I. Introduction

The last year has brought very particular challenges to teaching institutions. At the Department of History of Art at University College London, we were keen to continue to provide hands-on experience to students on the BA programme in History of Art, Materials and Technology. Building on existing sources and studies, we designed two experiments that could be performed at home to develop both manual and critical skills. This document aims to serve as a resource for teaching and academic staff involved in teaching subjects at the intersection of art and science, such as technical art history, introductory chemistry, materials and techniques of artists, and conservation. It highlights the source materials used for designing those experiments, describes the experimental procedure and the quantities used for the home kits that were sent to students all over the world, and indicates possible pedagogical uses for this procedure.

One of the challenges encountered when developing these at-home experimental procedures was related to the diverse geographic locations of the students. For example, for this experiment – making iron gall ink – we were not able to send vitriol to our students based in China or Russia, as chemicals are on the list of prohibited items. This led us to develop a slightly different variant of the procedure. We looked for alternatives that would allow students to perform the experiment without the use of vitriol. As a fundamental step in the design of our experiments and assembly of the home kits, we researched methods that had been used and published by others. The resources used in developing this procedure can be found in the *Resources* section below.

II. Source materials

The recipe comes from a manuscript kept at the British Library, London, Harley 3915, fol. 148v. The Latin and French versions of this recipe were taken from Monique Zerdoun Bat-Yehouda, *Les encres noires au Moyen Âge (jusqu'à 1600)*, Éditions du CNRS, Paris, 1983, p. 247. The English version was translated from the French by the Special Collections Conservation Unit of the Preservation Department of Yale University Library.

Source: Special Collections Conservation Unit of the Preservation Department of Yale University Library, *Medieval to Early Modern Manuscripts: Some Ink & Pigment Recipes*. Booklet available at: https://travelingscriptorium.files.wordpress.com/2014/06/scopa-recipes-booklet_web-june-2014.pdf, accessed 14.11.2020.

English

Latin Ink. Take an earthen vase (or jar or pan) that can contain 8 pounds of water; then (add) half a pound of small gall nuts and crush them well; then boil until (the water, the mixture) is reduced by half; then take three ounces of gum arabic and grind it well; and pour (add) the gum to the mixture in the jar and boil it until reduced by half. Remove the jar from the fire and take 4 ounces of vitriol and one pound of warm wine and mix them together in another jar and add little by little to the ink, stirring well. Leave it to rest for two days, and afterwards, every day, stir four times with a stick.

Latin

Inc [austum] latinum; accipe vas de terra quod capiat VIII l. |libras| aque; postea mediam libram gallette et tere bene; postea bulli usque ad medietatem, tunc accipe tres untias gummi arabici et tere bene et colato illo quod est in oll |a| apponatur gumma, tunc bulliat ad medietatem. Postea aufer ab igne et tunc accipe 4 uncias vitreoli et l. |libram| vini calidi aliquantulum et debes miscere vinum et vitreolum in alio vase bene, tunc paulatim apponatur ad inc [austum] miscendo senper bene; ita stet per duos dies et quolibet die moveatur quarter cum baculo postea.

French

Encre latine. Prends un vase de terre (ou un pot ou un poêlon) qui contienne 8 livres d'eau ; ensuite (ajoute) une demi-livre de petites noix de galle et écrase bien ; ensuite fais bouillir jusqu'à ce que (l'eau, le mélange) soit réduit de moitié ; puis prends trois onces de gomme arabique et écrase bien ; et que la gomme soit versée (ajoutée) au mélange qui est dans le récipient, puis que cela bouille (faire bouillir) jusqu'à réduction de moitié. Ensuite, retire du feu et puis prends 4 onces de vitriol et une livre de vin un peu chaud, et tu dois mélanger le vin et le vitriol dans un autre vase (récipient), puis l'ajouter peu à peu à l'encre, toujours en mélangeant bien. Que cela reste ainsi pendant deux jours, et après cela, chaque jour, remue quatre fois avec un bâton.

III. List of materials and equipment

Equipment

- 1 plastic funnel
- 1 small glass bottle (30 mL)
- 3 round filter papers
- 1 sheet (A4) of parchment-like paper
- 4 small squares of aluminium foil
- 3 pH paper sticks (pH 0-14)
- 1 feather quill
- 2 wooden stirring sticks
- 2-3 pairs of examination gloves

Materials

- 5 g gallnut powder
- 3 g gum arabic
- 7 g Vitriol (Iron (II) Sulfate heptahydrate)

(Caution! May cause skin and eye irritation. Harmful if swallowed. Please wear gloves when handling and pay special attention!)

OPTIONAL – if NOT using vitriol

- app. 50 mL malt vinegar (6 small sachets à 8 mL)
- 1 piece of steel wool (app. 5 x 10 cm)

IV. Experimental Procedure

OPTIONAL STEP – if NOT using vitriol

1. Place the provided chunk of steel wool in the container.
2. Pour 4 or 5 sachets of malt vinegar in your container.
3. Cover the container with a lid (if no lid is available, you can use cling film).
4. Leave it to soak for at least three weeks.

Result: You will obtain a solution of vinegar with iron, referred to as *Solution Fe* in the next section of the experimental procedure.

Procedure for both versions

1. Add app. 50-60 mL water to a pot or pan. (Best to use an old vessel.)
2. Add the gallnut powder, stir, and bring to the boil. Let the mixture cook for 20 minutes. Make sure to apply enough heat to allow cooking but avoid the water to evaporate completely. *
3. Use the plastic funnel and one filter paper (both provided with your kits) to filter the (cooled down) solution into a glass bottle.

Please note that this solution will stain everything it touches, so be sure to protect yourself (wear gloves and old clothes) and the surface you are working on. Clean the vessel used for boiling, as well as the plastic funnel immediately after use!

4. VERSION 1 - USING VITRIOL

- a. Pour 25 mL of the filtered gallnut solution to one of the small glass bottles.
- b. Add the vitriol (see warning above in List of materials and equipment!). You need to add at least 1/3 of the weight of vitriol to your solution. The provided amount of vitriol (7 g) can be added to 25 mL of gallnut solution. However, you can also try to add small amounts of vitriol at a time to see the various hues you can obtain.
- c. Now add the gum arabic in small amounts at a time, until the ink reaches a consistency that pleases you.

VERSION 2 - USING SOLUTION FE (NO VITRIOL)

- a. Pour 15 mL of the filtered gallnut solution into one of the small glass bottles.
- b. Add around 5mL of the *Solution Fe* (try to avoid pouring in any solid particles). You need to add at least 1/3 of *Solution Fe*'s volume to the gallnut solution.
- c. Now add the gum arabic in small amounts at a time, until the ink reaches a consistency that pleases you. If you have any leftover gum arabic, *Solution Fe*, and gallnut solution, you can add more of each of these (in equal proportions).

5. Let the ink rest for one day.
6. Use the quill and parchment-type paper to try out your ink!

*** Alternative procedure (if no stove is available)**

1. Add around 25 mL water to a glass bottle.
2. Add the gallnut powder to the glass container, stir the solution, and cover the container with aluminium foil or a lid.
3. Put the glass container in a warm place, e.g. next to a heat source, like your windowsill. Leave it there for five days.
4. Follow the above protocol from point 3.

V. Resources

- Monique Zerdoun Bat-Yehouda, *Les encres noires au Moyen Âge (jusqu'à 1600)*, Éditions du CNRS, Paris, 1983, p. 247
- *Medieval to Early Modern Manuscripts: Some Ink & Pigment Recipes*. Booklet compiled by the Special Collections Conservation Unit of the Preservation Department of Yale University Library. Available at:
https://travelingscriptorium.files.wordpress.com/2014/06/scopa-recipes-booklet_web-june-2014.pdf
- <https://threadreaderapp.com/thread/1035508214757027841.html>
- <https://www.quirkbooks.com/post/how-tuesday-how-make-medieval-ink>
- <https://www.bl.uk/medieval-english-french-manuscripts/videos/4-making-manuscripts-oak-gall-ink>
- <https://gilbertredman.com/medievalmanuscripts/codicology/SCRIPTORIUM-INKS/>

VI. Teaching subjects

Under the remit of history of art, technical art history, and conservation, this procedure can be used to discuss themes such as: interpretation of historical sources, understanding of the materials of art, issues of degradation and conservation.

In the area of chemistry, this procedure can be used to discuss issues pertaining to organic chemistry, acids and bases, and chemical reactions. Depending on the depth of analysis, this experiment can fit the curriculum of GCSE and A-level chemistry.

VII. Activities

Various activities can be developed from this experiment. Here, we highlight four of the many pedagogical pathways that can be explored in class:

- ***Interpreting historical sources***
Students are asked to interpret the recipe – i.e. understand how to successfully replicate it – over the course of several weeks, paying particular attention to the terms used in reference to materials (e.g., vitriol) and quantities. This will allow students to develop their own theoretical models on how one could replicate this recipe, confirming or refuting their expectations through this experiment. This activity can also take place in groups.
- ***From the lab to the home***
NOTE: This activity requires access to analytical data.
The activity starts with students looking at FT-IR spectra of a sample iron gall ink. Ideally, these spectra would have been produced in the laboratory, using a sample from a specific object. Students try to recognise and label the different IR peaks of the spectra to develop theoretical models on what one should expect the molecule to be. Then they proceed with the experiment and see if they can confirm their expectations. This activity can be further expanded to explore the degradation of cellulose in contact with iron gall inks.

- **Comparing historical sources**

NOTE: This activity takes more than 10 weeks to be developed in the context of remote teaching.

Students are asked to analyse two different historical recipes for producing iron gall ink. They develop the experimental procedure based on their expectations and interpretations of the historical sources. Students are then given access to the materials they need (and home kits are sent to them if the experiment happens remotely). They try both recipes and discuss the results both in terms of the chemical reactions that took place and the useability of the materials. This experiment can be complemented with FT-IR analysis to confirm the results obtained.

- **Raw materials**

Students interpret the recipe by tracing back the origin of the raw materials mentioned. This activity will allow students to explore various issues: from trade routes to knowledge exchange, to the material conditions needed to create these materials.

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List of Contributors

Tobias Preuten and Hélia Marçal: development and testing of the experiments, authors of the first version of this document.

Tobias Preuten: risk assessment, procurement, assembly, packing, and posting of the home kits during the academic year 2020-2021.

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