Inks

Naomi Rosenkranz, The Making and Knowing Project

Presentation adapted from work originally conducted for 1) The Metropolitan Museum of Art, 2015 – 2016 (Intern, Departments of Paper Conservation and Scientific Research) and 2) The Columbia University Conservation Lab, 2015 (Science Resident in Conservation, Columbia Ancient Ink Lab)

Writing Ink

Writing inks are a colored viscous fluid composed of a water-based liquid, a binding agent, and a colorant.

Inks are often liquid, but can also come in the form of pastes, cakes, and sticks.

Ingredients:

- Liquid examples
  - Water
  - Wine
  - Vinegar
- Binding media examples
  - Gum or resin – hardened sap of plants
    - Gum arabic (from acacia trees), tragacanth gum (from *Astragalus* genus*)*
  - Animal glue – adhesive made of animal proteins extracted from bones, hides, etc.
- Colorant examples
  - Mineral pigments (azurite, lapis lazuli/ultramarine, verdigris)
  - Earths and ochres (burnt sienna, yellow ochre, red bole)
  - Organic pigments (charcoal, madder lake, sap green)

Plimpton MS36, Columbia University Libraries

Short History of Black Writing Inks

Carbon Inks

- Carbon-based inks are the earliest known writing inks, dating from at least 2500 BC.
- Carbon ink is prepared by mixing powdered soot or charcoal in a solution of water and binding media (gum/resin or animal glue).
  - Soot – collection of smoke particles (e.g. burning a candle and placing a plate above the flame to collect black matter)
  - Charcoal – burned vegetable matter (e.g. completely burned wood)
- Black pigmented particles are suspended in a viscous solution of water and gum, creating a fluid black ink.
- When applied to a writing surface (paper, parchment), the carbon particles sit on top, adhered to the surface by the binding media.

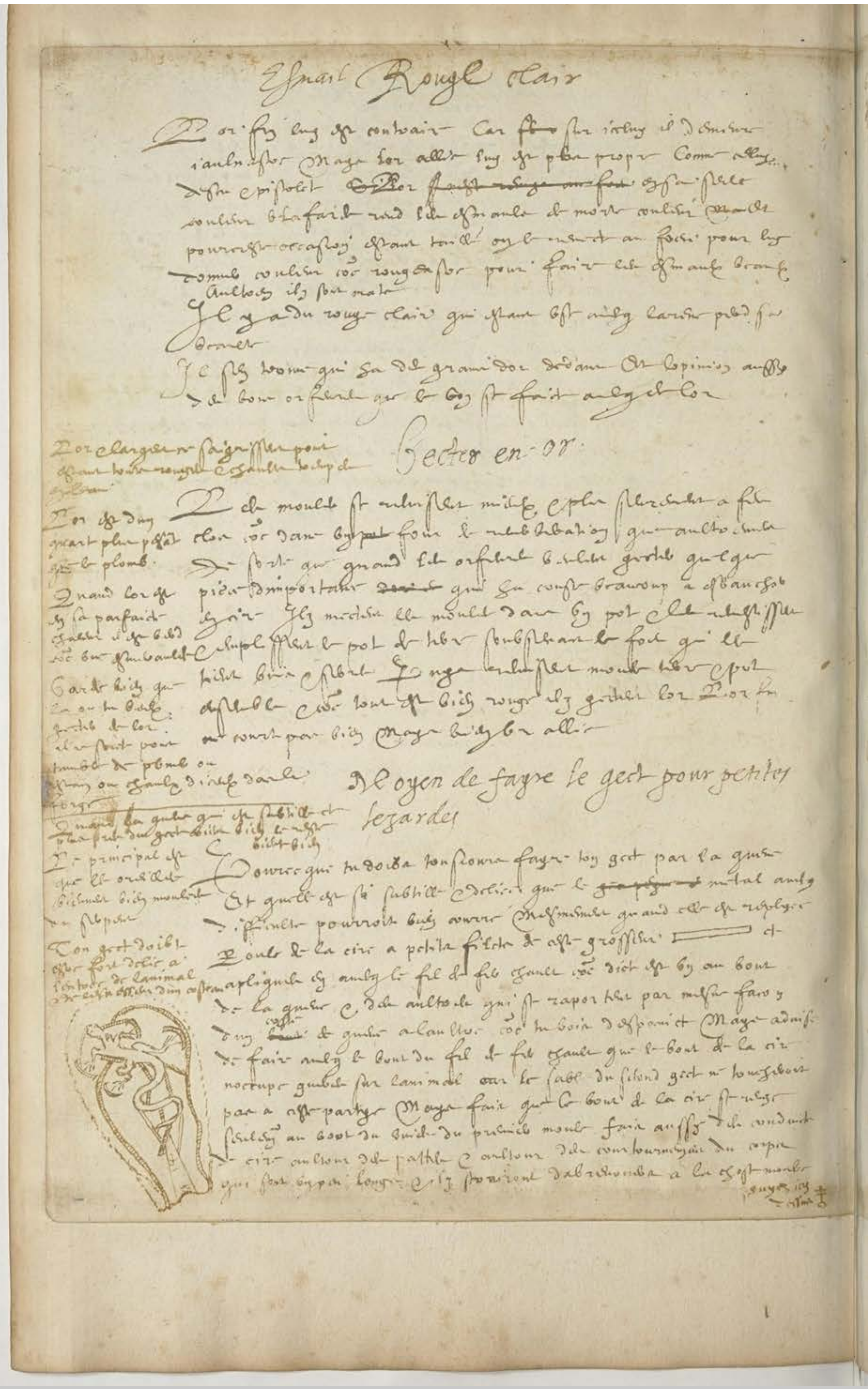
Book of the Dead (APIS P1905), 500 BCE, Columbia University Libraries

Short History of Black Writing Inks

Iron Gall Inks

- Dating from around the 9th century, iron gall inks become “common ink” in the western world by 11-13th centuries, almost completely replacing carbon inks as the standard writing ink.
- In the 19th century, iron gall inks were subsequently replaced by synthetic.
- Unlike carbon inks, the color of iron gall inks are the result of a chemical reaction (and are closer to dyes than to pigments). When combined in solution, tannic and/or gallic acid (an astringent commonly found in oak galls) reacts with iron to form a blue-black iron gallotannate complex.
- A binding media such as gum is added to make the solution more viscous.
- As the solution comes in contact with air, it oxidizes and darkens to a deeper black.
- When applied to a substrate (paper, parchment), the ink chemically bonds with the surface.
- Because they interact with the substrate, iron gall inks are much more indelible than carbon inks which can be easily smudged or wiped away with water.
- However, this same property makes iron gall inks much more reactive than the stable carbon pigments. Over time, they can degrade, turn brown, damage or completely corrode paper and parchment, and react with other pigments.
- The precise reason for how and why some historical iron gall inks are extremely degraded or harmful while others have stayed unchanged is still not fully understood. Some theories include the formation of corrosive metal salts, off-gassing of sulfuric acid from the iron, and chemical bonding problems with the substrate. The degradation of objects with iron gall is one of the most common problems dealt with by conservators.
- Factors affecting this include: ink composition and method of preparation, ratio of ink ingredients,age of ink at time of application, environmental conditions, flow of ink from pen (i.e. when and how the writer dipped quill in inkwell), and interaction with the substrate.
- While we have come to expect manuscripts from the medieval period or the renaissance to have sprawling brown and faded text, the original manuscripts actually had very dark black “permanent” ink.

Folio 124v, BnF Ms. Fr. 640, 16th century

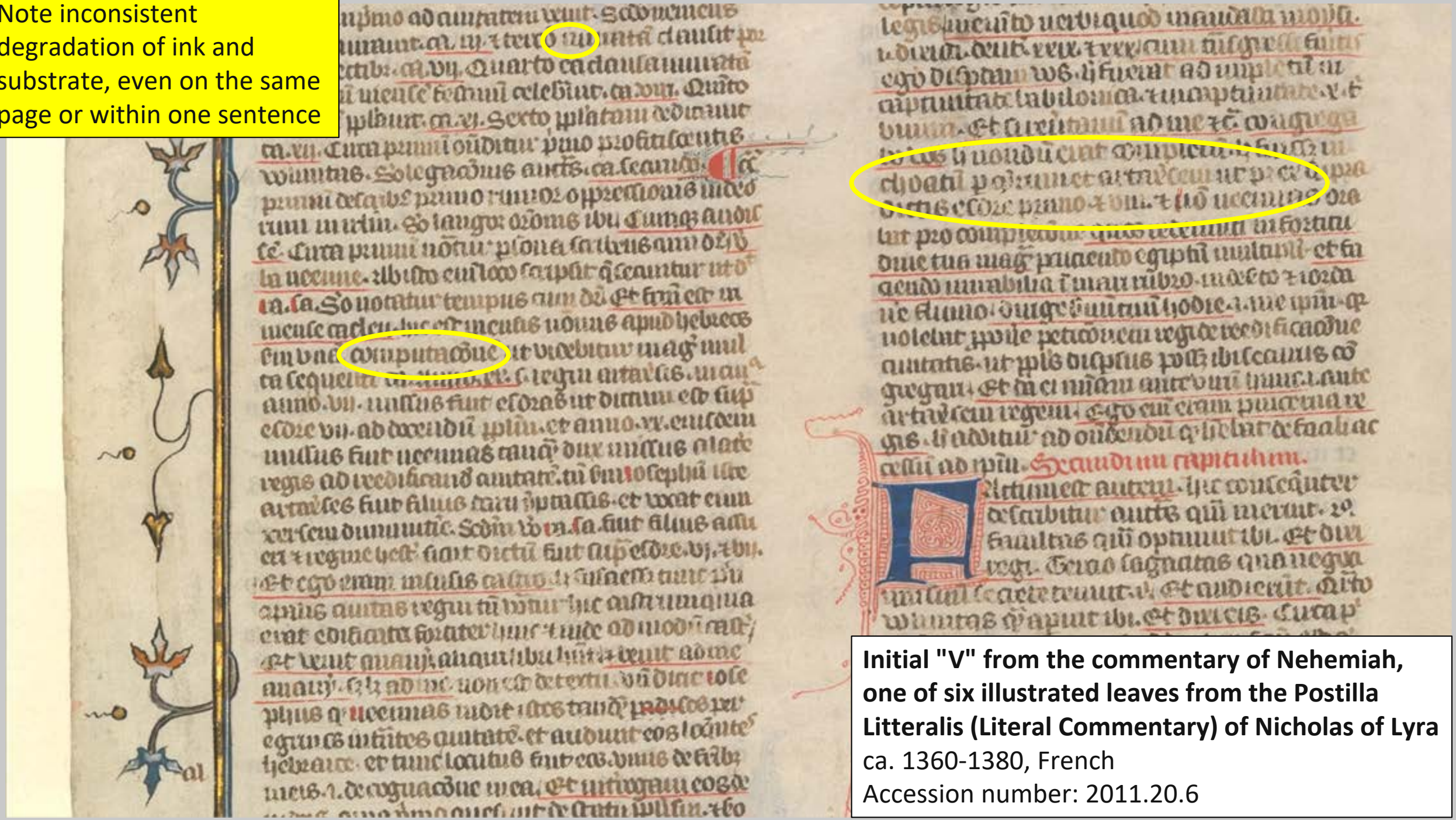


Example of iron gall ink degradation

Classic example of “shadow” letters – areas where the ink has eaten through the paper

Photo by Ceinturion - Manuscript from Igreja de Sao Francisco, Evora, Portugal

Note inconsistent degradation of ink and substrate, even on the same page or within one sentence



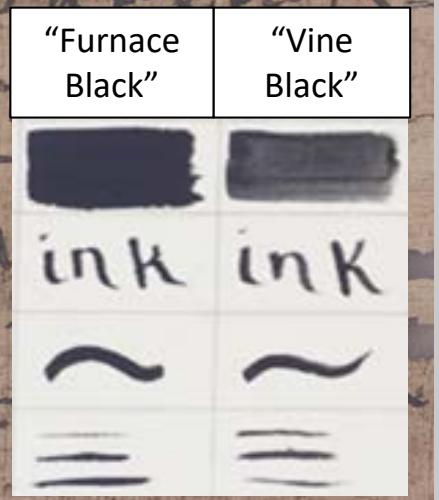
Initial "V" from the commentary of Nehemiah, one of six illustrated leaves from the Postilla Litteralis (Literal Commentary) of Nicholas of Lyra

ca. 1360-1380, French

Accession number: 2011.20.6

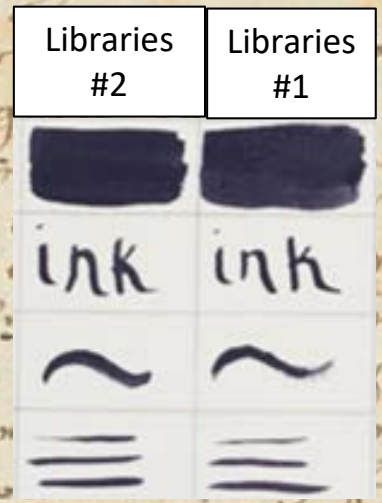
Carbon ink

- Soot/char
- Binder
- Liquid



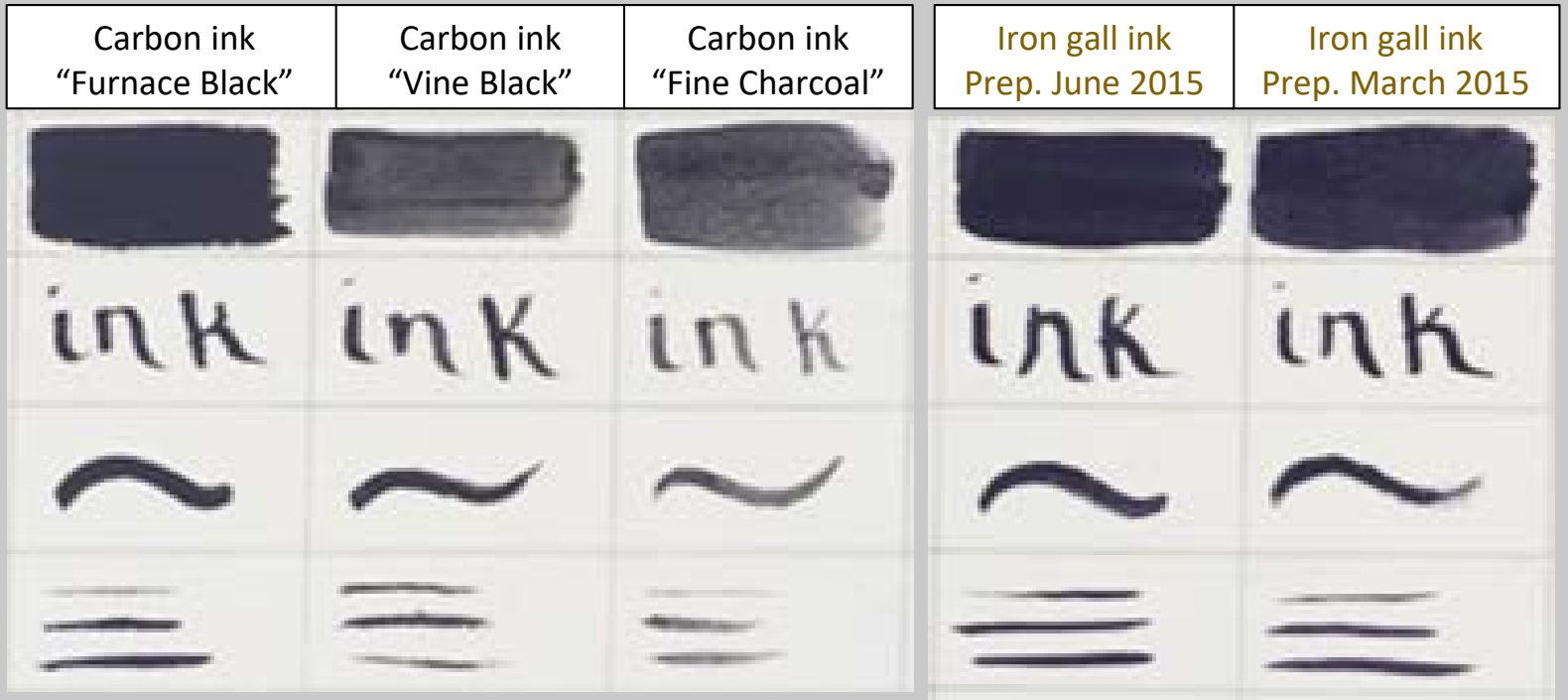
Iron gall ink

- Tannin
- Iron
- Binder
- Liquid



Ink ingredients

	Carbon	Iron gall ink
Solution	Liquid	Liquid
Binder	Gum/glue	Gum/glue
Colorant	Soot or char	Iron & tannin



Iron Gall Ink

Ingredients

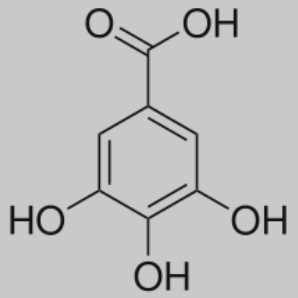
- The most basic requirements for iron gall ink (explained in subsequent slides) are:
  - Tannin
    - Plant-derived polyphenol that yields tannic or gallic acid
  - Iron
    - Commonly in the form of iron sulfate, but can also be in other forms, such as iron acetate
  - Binder
    - Gum, resin, or glue
  - Liquid
- Variations and additives include:
  - Copper sulfate in place of or in addition to iron sulfate
  - Carbon black pigment mixed into the solution

Tannin

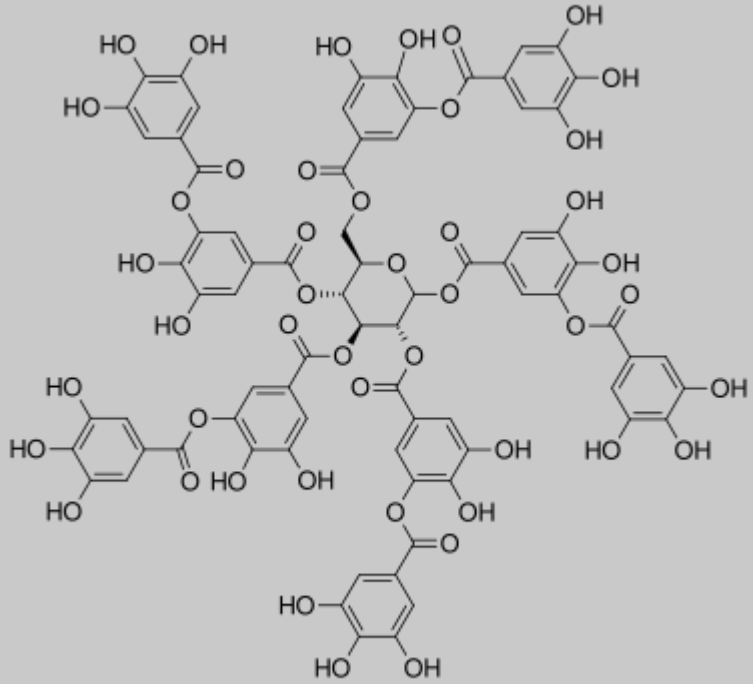
Plant polyphenol, astringent.

Extracted from botanical sources such as gall nuts, bark, and leaves to obtain hydrolysable tannins.

A hydrolysable tannin - a tannin that yields gallic acid through hydrolysis (breakdown of a molecule with water).



Gallic acid
$C_7H_6O_5$



Tannic acid
$C_{76}H_{52}O_{46}$

Tannin

For ink, often extracted from the galls of various species of oak, the sumac plant, pomegranate rinds, and walnut husks

Persian or English Walnut (*Juglans regia*)

Sicilian Sumac (*Rhus coriaria*)

Aleppo oak (*Quercus infectoria*)

Pomegranate (*Punica granatum*)

Gall nuts

also known as galls, oak galls, or oak apples

Gall nuts are the parasitic reaction of trees to egg-laying insects.

The tree secretes a solution high in tannic acid which forms around the eggs. It is this hardened formation that serves as an important source of tannin.

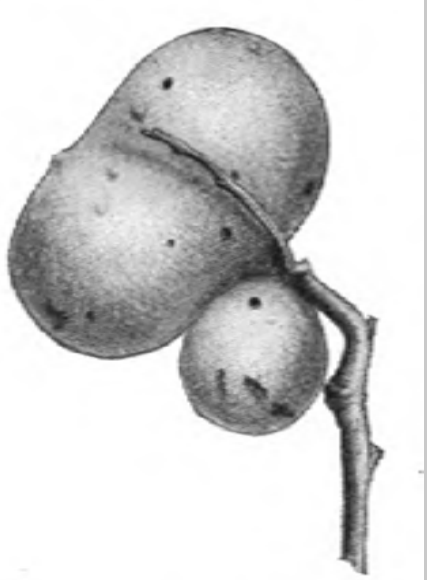


Fig. 11.—English double oak-apple gall.

Fig. 12.—English oak gall.

Fig. 13.—English gall.

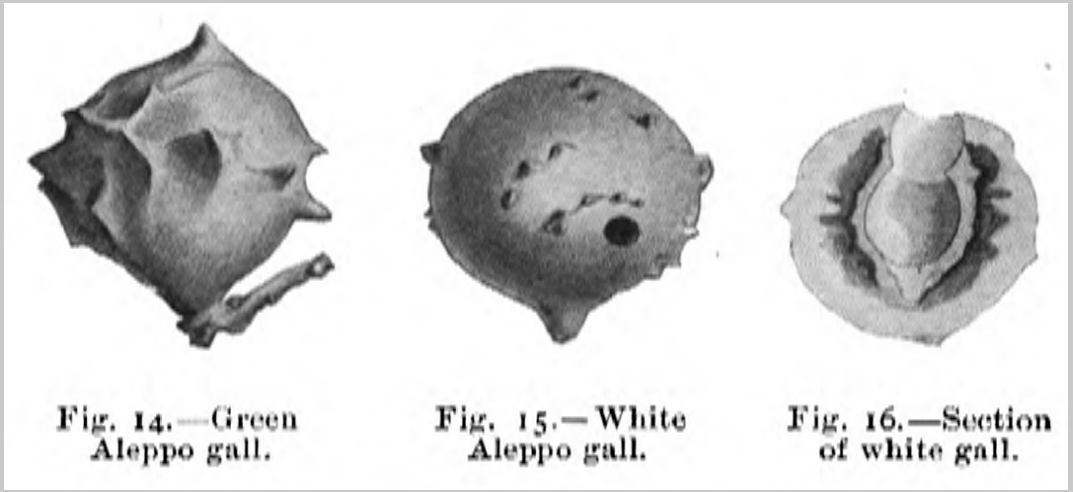


Fig. 14.—Green Aleppo gall.

Fig. 15.—White Aleppo gall.

Fig. 16.—Section of white gall.

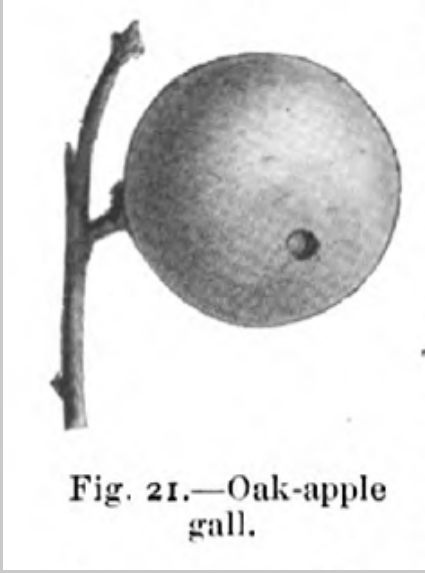


Fig. 21.—Oak-apple gall.

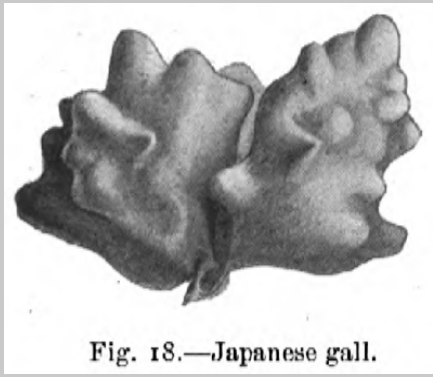


Fig. 18.—Japanese gall.

Fig. 17.—Chinese gall.

Mitchell, C. Ainsworth 1867-. Inks: Their Composition And Manufacture. London: C. Griffin & company, limited,

Aleppo Galls

Aleppo oak (*Quercus infectoria*)

The most commonly used galls are Aleppo galls, formed by *Quercus infectoria,* a member of the oak family native to Asia Minor.

Aleppo galls have been prized for their high concentration of gallotannic acid (60-80%), more than any other source of tannin.

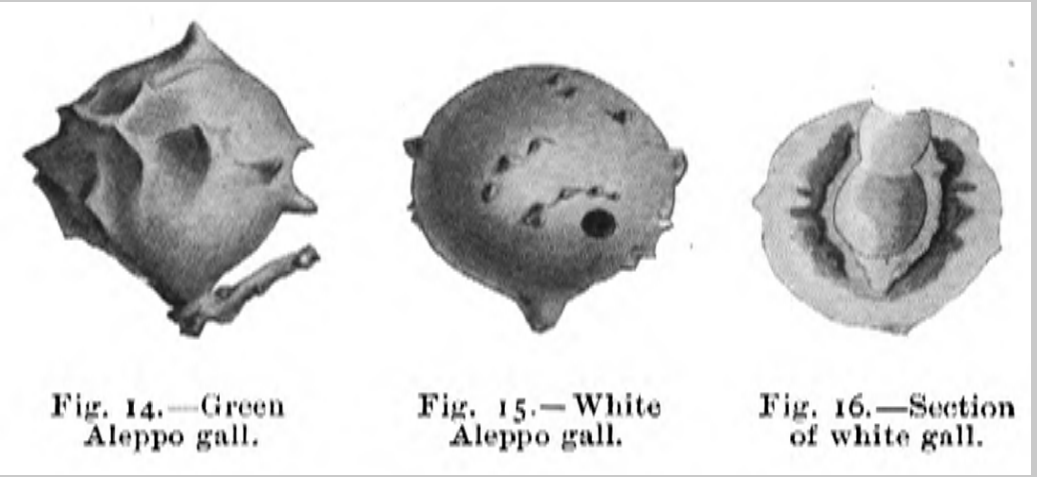


Fig. 14.—Green Aleppo gall.

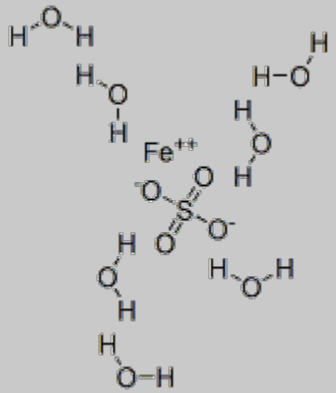
Fig. 15.—White Aleppo gall.

Fig. 16.—Section of white gall.

Crushed galls

Iron Sulfate

also known as vitriol, green vitriol, or copperas



$(FeSO_4 \cdot 7H_2O)$
Water soluble iron salt

Historical uses:

- Ingredient in ink
- As a mordant & colorant in dyeing
- Medicinal (e.g. for tooth decay)
- In metallurgy (e.g. to purify gold)
- Discovery of sulfuric and nitric acids

Georgius Agricola, *De re metallica*

Melanterite (mineral form)
Adam Szilveszter, <http://www.mindat.org/photo-322573.html>

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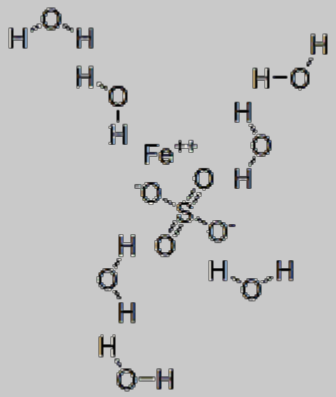

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A—Tunnel. B—Bucket. C—Pit.

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Iron mordant with madder
"The Enduring Appeal of Natural Colour. <http://www.textilestudio.ca/guest%20artists/Haines.html>.

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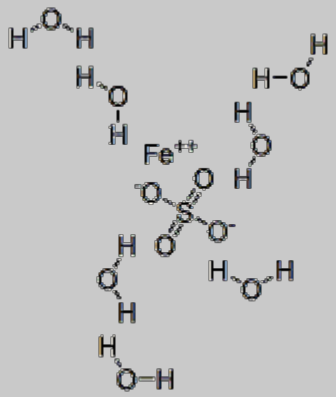

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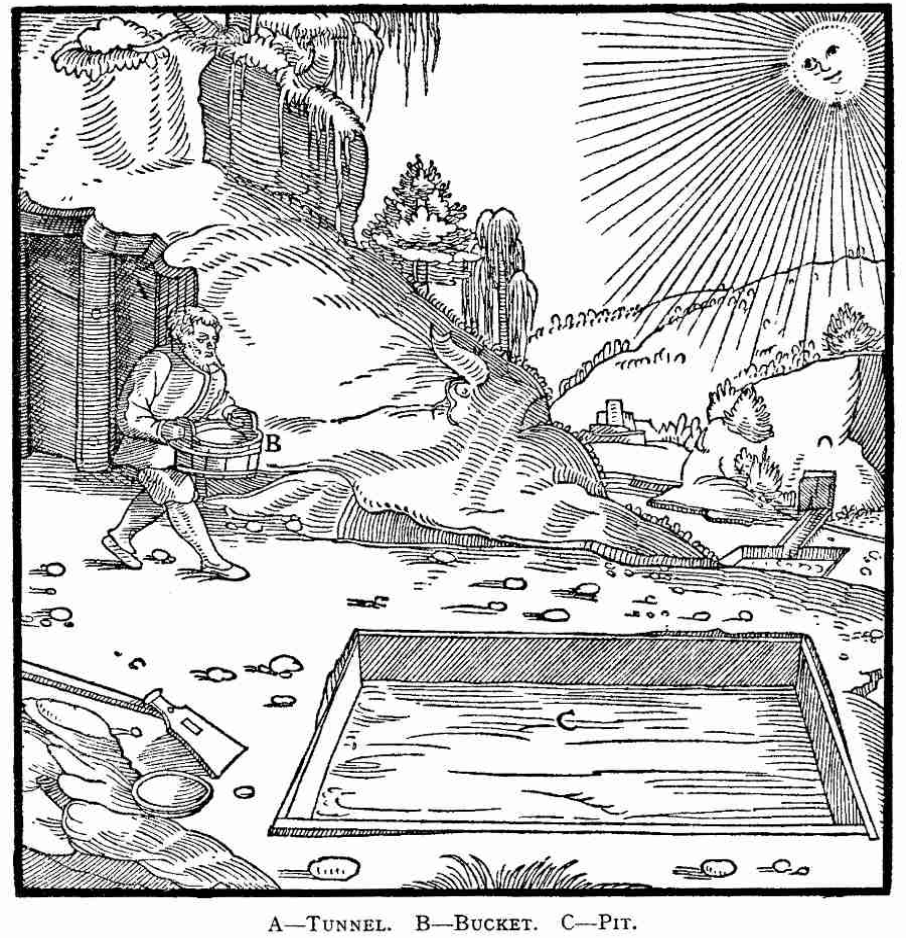

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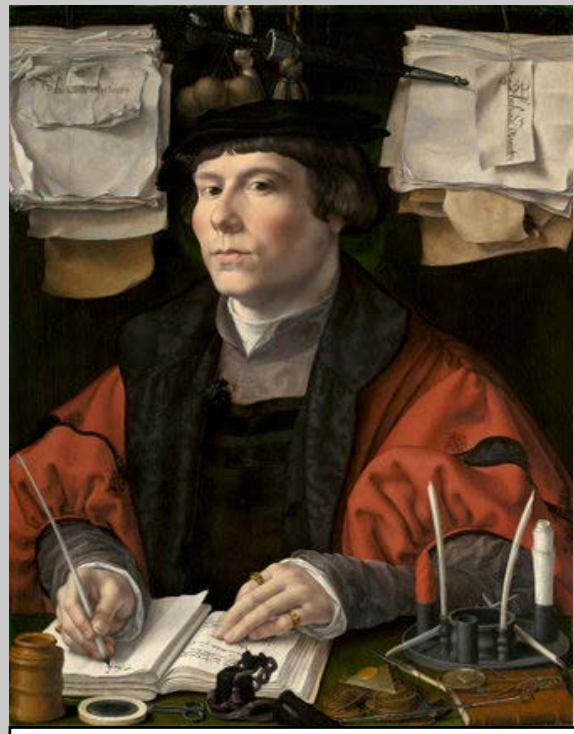

Jan Gossaert, *Portrait of a Merchant*
National Gallery of Art, London

Tannin: Gall nuts

Crushed or powdered
for faster extraction

Water
+ tannin

Gallic acid extraction

Tannin: Gall nuts

Crushed or powdered
for faster extraction

Water
+ tannin

Gallic acid extraction

Binder: Gum Arabic

Ground hardened sap
of acacia tree

Water
+ tannin
+ gum

Adhesion, dispersion, viscosity

Tannin: Gall nuts

Crushed or powdered
for faster extraction

Water
+ tannin

Gallic acid extraction

Binder: Gum Arabic

Ground hardened sap
of acacia tree

Water
+ tannin
+ gum

Adhesion, dispersion, viscosity

Iron: Iron sulfate

Iron(II) sulfate
heptahydrate $FeSO_4 \cdot 7H_2O$

Water
+ tannin
+ gum
+ iron sulfate

Ferrous tannate complex

Preparation

- There are four common preparation methods:
  - Heated
    - Gallotannic acid is extracted in a liquid with heat before gum and iron are added
  - Cold and quick
    - All ingredients are brought together in solution, usually relying on a fine powder of a tannin to facilitate extraction
  - Soaked
    - Tannin is added to a liquid and allowed to soak for a longer period of time (days, weeks, or even months) to extract gallotannic acid
  - Dry
    - All ingredients are mixed together except the liquid, which is added only until the mixture comes together to form a cake. To use the ink, the cake is dissolved in liquid.

Examples of iron gall ink recipes

1) A Booke of Secrets (Dutch, 1596)

Another of the same sort, but easie to make:

Take the beaten gauls, and put them in the water, doe the like with the victriall in a pot by it selfe, let those two waters stand, and when you have cause to use inke, poure out of each pot a like quantitie, and it will be blacke, then put into it a little beaten gum, and it will bee good inke.

2) The Bologna Manuscript Secrets for making colours, (15th century)

To make good writing ink.—Take a bocale of good and strong white wine, 4 oz. of galls well crushed, one handful of dried rinds of pomegranates, one handful of the fresh bark of mountain ash scraped with a knife, and one handful of fresh bark of roots of walnut trees, and 2½ oz. of gum arabic; mix the whole together with the wine, and let the mixture remain for 6 or 8 days in the sun, stirring it well 4 or 6 times every day. Then add 2½ oz. of Roman vitriol, and mix it frequently, and let it remain so for several days; then put it over the fire to boil for the space of one miserere, let it cool, and then strain it and leave it for 2 days in the sun. If you then put in It a little roche alum It will make It much brighter, and it will be a good and perfect writing ink.

3) Recipe for making iron gall ink (c. 1483)

To make hynke. Take gall & coporos & or vitrial quartryn & gumme of eueryche a quartryn oþer helf quartryn & a halfe quartryn of gall more & breke þe gall a ij oþer a iij & put ham togedere euery-che one in a pot & stere hyt ofte & wy3t wythinne ij wykys after 3e mow wryte þer wyþ. & yf 3e have a quartryn of eueryche take a quarte of watyr yf halfe a quartryn of eueryche þan take half a quartre of watyr.

4) The Book of the Staff… (ibn Badis, 11th century)

Description of instant ink.
Gallnut of the terebinth – yellow green, is taken, Greek vitriol, and gum arabic – of each a mithqal. It is all pulverized and put into a vessel which is wide-mouthed. Two ounces of salt water are poured on it. It is well beaten. It can be used for writing immediately on paper and parchment. This is the description of the Iraqi way.

5) The Book of the Staff of the Scribes and Implements of the Discerning with a Description of the Line, the Pens, Soot Inks, Liq, Gall Inks, Dyeing, and Details of Bookbinding (ibn Badis, 11th century)

Preparation of another ink. Gallnut and pomegranate rind are taken, pressed together, and soaked in some water for three days. Then blue vitriol is poured on it little by little while stirring until it is seen that it has become a strong black. If the qalqant (green vitriol) is not available, then use its substitute, Persian vitriol. Gum arabic is added. It is then removed from the fire.

6) Rules made by E.B. For his children to learne to write bye

Book Containing Divers Sorts of Hands, by John de Beau Chesne and M. John Baildon, and published in 1571

To make common yncke of Wyne take a quart, Two ounces of gomme, let that be a parte, Five ounces of galles, of copres take three, Long standing dooth make it better to be; If wyne ye do want, rayne water is best, And as much stuffe as above at the least: If yncke be to thick, put vinegar in, For water dooth make the colour more dimme. In hast for a shift when ye have a great nead, Take woll, or wollen to stand you in steede; which burnt in the fire the powder bette small With vinegre, or water make yncke with all. If yncke ye desire to keep long in store Put bay salte therein, and it will not hoare. Of that common yncke be not to your minde Some lampblack thereto with gomme water grinde.

1) Page 24, translation, Thompson, Jack C., and Claes G. Lindblad. “Manuscript Inks: Being a Personal Exploration of the Materials and Modes of Production.” Portland, Oregon: Caber Press, 1996.
2) Page 590. Merrifield, Mary P. *Original Treatises on the Arts of Painting*. New York: Dover Publications, 1967. Print.
3) Recipe for making iron gall ink. The National Archives of the UK, C 47/34/1/3, (c. 1483). <http://www.nationalarchives.gov.uk/henryviii/parchment/inks.htm> Transcribed by Sarah Peverley, University of Liverpool https://sarahpeverley.com/2014/01/29/iron-gall-ink-a-medieval-recipe/
4) Page 18-19 from Levey, Martin. “Mediaeval Arabic Bookmaking and Its Relation to Early Chemistry and Pharmacology.” Transactions of the American Philosophical Society 52, no. 4 (1962): 1. doi:10.2307/1005932.
5) Ibid, page 21
6) irongallink.org

Griffini Recipes

"La Preparazione degli inchiostri *Hibr* e *Midad* di Differenti Colori: Esposta da un Anonimo Siciliano" by Eugenio Griffini
Journal: Scritti per il centenario della nascita di Michele Amari, V 1? V 2?, pgs. 444-448 (1990)

Working translation from Arabic:

(with the help of Ayman Elgohary, Metropolitan Museum of Art visiting research fellow, Museum of Islamic Art, Cairo)

Introduction and salute.

Please forgive the former religious leader.

This is now the chapter for how to make ink.
There are four types: cooked, squeezed, soaked in water, and dry.

You must use the parts from the tannin tree, and they should be almost dark. Crush them to dust and put them in a bottle, which must be coated well or you can use a copper pot which must be very smooth.

Here are the four recipes:

1. "Cooked"

On a "soft fire", add 4 tannin and 10 water. Cook until mixture is reduced to 2/3. Remove from fire and add 4 gum arabic and cool to room temperature. Separately, add 1 part vitriol (زاج"zag/zak") *to vinegar (* خل"khal"). *Leave vitriol on vinegar to settle. Put it on tannin water (first part) - you will get great color.*

2. "Squeezed"

Crush/pound tannin. Sift/garble and put in coated bottle. Add 5 parts water and mix. Leave for 1 hour, then filter. Leave again to settle. Add 1 crushed gum (prepared well). Separately, put 10 parts vitriol on vinegar like tea on hot water. Leave vitriol on vinegar to settle. Put it on tannin water.

3. "Soaked"

Crush/pound 1 part tannin so "you can't see dust". Put 10 parts water on it and leave for days (the more you leave the better color you get). When it has soaked, prepare 1/2 part crushed gum and add to tannin water. Separately, add vinegar to ½ part vitriol and leave to settle. Put it on tannin water.

4. "Dry"

Grind 1 tannin, ½ vinegar, ½ vitriol, and 1 gum. Sift. Make a paste by mixing with as much water as needed. Keep as a solid ball in open air, but away from sunlight. Take some as needed and mix with water to make liquid ink.

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Crush/pound 1 part tannin so "you can't see dust". Put 10 parts water on it and leave for days (the more you leave the better color you get). When it has soaked, prepare 1/2 part crushed gum and add to tannin water. Separately, add vinegar to ½ part vitriol and leave to settle. Put it on tannin water.

4. "Dry"

Grind 1 tannin, ½ vinegar, ½ vitriol, and 1 gum. Sift. Make a paste by mixing with as much water as needed. Keep as a solid ball in open air, but away from sunlight. Take some as needed and mix with water to make liquid ink.

Griffini Recipes

"La Preparazione degli inchiostri *Hibr* e *Midad* di Differenti Colori: Esposta da un Anonimo Siciliano" by Eugenio Griffini
Journal: Scritti per il centenario della nascita di Michele Amari, V 1? V 2?, pgs. 444-448 (1990)

Working translation from Arabic:

(with the help of Ayman Elgohary, Metropolitan Museum of Art visiting research fellow, Museum of Islamic Art, Cairo)

Introduction and salute.

Please forgive the former religious leader.

This is now the chapter for how to make ink.
There are four types: cooked, squeezed, soake

You must use the parts from the tannin tree, d
dark. Crush them to dust and put them in a b
well or you can use a copper pot which must b

Quercus infectoria

Here are the four recipes:

1. "Cooked"

On a "soft fire", add 4 tannin and 10 water. Cook until mixture is reduced to 2/3. Remove from fire and add 4 gum arabic and cool to room temperature. Separately, add 1 part vitriol (زاج "zag/zak" *) to vinegar (* خل "khal"). *Leave vitriol on vinegar to settle. Put it on tannin water (first part) - you will get great color.*

2. "Squeezed"

Crush/pound tannin. Sift/garble and put in coated bottle. Add 5 parts water and mix. Leave for 1 hour, then filter. Leave again to settle. Add 1 crushed gum (prepared well). Separately, put 10 parts vitriol on vinegar like tea on hot water. Leave vitriol on vinegar to settle. Put it on tannin water.

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Iron sulfate

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Reconstructions of Griffini recipes (2015-16)

1. Griffini - “Cooked”
2. Griffini - “Squeezed”
3. Griffini - “Soaked”
4. Griffini – “Dry”

Deposited on filter paper

Deposited on glass slide


GRIFFINI R1
"COOKED"
GRIFFINI R2
"SQUEEZED"
GRIFFINI R3
"SOAKED"
GRIFFINI R4
"DRY"

WGaS.10
2015.12.11
WGaS.80
2015.12.11
WGaS1.0
2015.12.11
2016.03.25
F-VGA
F-VTA
F-VGaA
5g IRON FILING
30ml VIN
A B C D
WTS.30 2015.12.11
WTS.50 2015.12.11
WTS.70 2015.12.11
WTS.80 2015.12.11
WTS1.0 2015.12.11
WTS2.0
SLIDE 2
F-VGA
F-VTA
F-VGa A
(2016.03.16) iron filings in vinegar
2016.02.19
O1 - WTS
O1 - WGaS
O2 - WGS
O2 - WGS, aged
B-ext - WTS
Deposited 2016.03.16
Griffini R1
Griffini R2
Griffini R3