

Student Sheet

In this practical I will be:

- Learning and using key terms, such as, **suspension, reaction, pigment, opacity** and **tack**.
- Creating and using my own ink.
- Evaluating the effects of various factors on: the ease of use; opacity; drying time and pH of my ink.

Introduction:

You have finished a piece of art you were commissioned to create. Unfortunately, as you live in ancient times, mobile phones have not been invented yet. Therefore you will need to write a letter to your patron, informing them that their art is ready for collection.

Your bad luck continues as you have just used up the last of your ink! Luckily, as a science-artist, you can make some more...

Equipment:

- 90 g of dry oak galls
- 400 cm³ of water
- 60 g of iron(II) sulfate,
- 30 g of gum arabic
- 2 mouth masks
- 1 mortar and pestle
- 1 beaker (600 cm³)
- 1 measuring cylinder (500 cm³)
- 1 sheet of card to cover the 600 cm³ beaker top
- 1 sheet of coarse filter paper
- 1 stirring rod
- 1 pen and pen nib
- 1 sheet of white paper

Alternatively, if using tealeaves

Equipment:

- Dry black tealeaves (could be labelled as 'Oak Galls substitute')
- Pestle and mortar
- 1 beaker (250 cm³)
- 1 beaker (100 cm³)
- Stirring rod
- 1 measuring cylinder (100 cm³)
- Distilled water



- Iron(II) sulfate (HARMFUL and IRRITANT)
- Gum arabic (or craft glue)
- Plastic pipette
- Spatula
- Weighing boat
- Watch glass (or cover for the beaker)
- Filter paper
- Filter funnel
- 1 conical flask (100 cm³)
- Universal indicator paper and colour chart

Access to:

Straws

Scissors

A4 paper

Digital balance

Paper towelling

Disposable gloves

Blotting paper

Hot water

Wear safety glasses. Wear disposable gloves.

Method:

Wear safety glasses. Wear disposable gloves.

1. In advance: Make sure the galls are very dry so leave them in a warm dry place for 24 hours.
2. Wearing a mouth mask; break the galls into pieces, then grind them with a pestle in a mortar or use a coffee mill to form a fine powder of the galls.
3. Transfer the gall powder to a 600 cm³ beaker.
4. Measure the 400 cm³ of water in a measuring cylinder; add the water to the ground galls. Cover with a card top and leave the mixture to ferment in a sunny corner at room temperature for 3 days.
5. Using a coarse filter paper filter the mixture and add the iron(II) sulfate to the solution. Stir well and leave for another 3 days.
6. Add the gum arabic crystals; stir the mixture to form your ink.



7. Now test the ink using a pen nib.

Alternative method if using tealeaves:

1. Place the tealeaves into the pestle and mortar and grind them to a powder.
2. Put this powder into a 250 cm³ beaker.
3. Use the measuring cylinder to measure 50 cm³ of hot water and add the water to the powder in the beaker. Stir, then cover and leave to cool for 10 minutes.
4. After ten minutes, stir again before using the filter paper and funnel to filter the solution into a conical flask.
5. Rinse the beaker so it's ready to use again. The powder and filter paper can now be disposed of in a bin.
6. Pour the solution from the conical flask into the 250 cm³ beaker.
7. Carefully weigh 6 g of iron(II) sulfate and add this to the solution in the beaker.
8. Stir to dissolve the iron(II) sulfate and mix it into the solution.
9. Add a plastic pipette full (or two teaspoons) of gum arabic liquid to the solution in the beaker and stir the mixture again to form the ink. Pour the solution back into the conical flask.
10. Use the scissors to make a diagonal cut on one end of a straw to form a nib so that you can use the straw to write with.
11. Now test the ink using your pen (straw).
 - Why do you think the galls need to be dry?
 - Why do you think the galls are ground to a fine powder?
 - What is fermentation?
 - Test the pH of the ink. Is it acid or alkali?
 - Why would the pH be important for the long time use of the ink?

Going further:

Keep your writing samples and examine them after several days. Did their appearances change? If so, what do you think caused the changes?

Find illustrations in art books showing drawings made by Leonardo da Vinci and describe the appearance of the lines he drew with iron–gall ink. How does iron(III) tannate pigment change over long periods? How would you explain this colour?



Theory:**Explanation appropriate for KS4**

Tannic acid is a complex organic compound naturally found in bark, fruits, nuts, leaves, and insect galls. There are several forms of the acid, and it is known as tannin, gallotannin, gallotannic acid, and coriligan. Hence the chemistry of making ink is a complex one but a simplified explanation follows.

When the powdered galls are added to water and left to ferment two reactions occur:

- the fermentation of gallotannic acid to gallic acid by the enzyme tannase;
- followed by the formation of the ink pigment by the addition of the iron(II) sulfate.

The ink is very acidic and that acidic nature, over the centuries, makes the ink fade, discolour and damage the paper.

Explanation appropriate for KS5

The iron-gall ink pigment is made from gallic acid. However galls contain large amounts of gallotannic acid and relatively little gallic acid so the galls are fermented. During the fermentation the fungi *Aspergillus niger* and *Penicillium glaucum*, found in the galls, release the enzyme tannase which, over three days, catalyses the **hydrolysis** of gallotannic acid to form gallic acid and glucose.

When the iron(II) sulfate is added an acid-base reaction takes place followed by a redox reaction. Gallic acid and iron (II) sulfate form iron(II) gallate (a colourless water-soluble compound), H_3O^+ and SO_4^{2-} . Then, almost immediately, the iron(II) gallate reacts with atmospheric and dissolved oxygen to produce water and iron(III) pyrogallate.

Iron(III) pyrogallate is a black insoluble octahedral complex in which the **ligands** of each Fe^{3+} cation are two molecules of gallic acid. The presence of the H_3O^+ ions makes the solution acidic.

The acidic nature has, over the centuries, made the ink fade, discolour and damage the paper. There is an excess of Fe^{2+} ions. The 2:3 ratio of iron(II) sulfate to galls means there is more iron(II) sulfate than is necessary to react with the gallic acid and some of these surplus cations oxidise to form Fe^{3+} oxide which, as it is paler than iron(III) pyrogallate, fades the original black colour of the ink.

This method is based upon the article in Issue 6 Science in School *Galls* which can be found at <http://www.scienceinschool.org/2007/issue6/galls>.

