

## Student Sheet

In this practical I will be:

- Learning about how lake pigment paints are made, using key terms such as **pigment, binder** or **liquid medium, lake pigment, organic, inorganic** and **fugitive**.
- Preparing lake pigment paints, analysing any property changes and indications that a chemical reaction has occurred.
- Evaluating the quality of the paints on different surfaces, determining which binder produced the best paint.

### Introduction:

You are an ancient Greek science-artist who has been commissioned to paint a fresco of your patron's daughter. Like most women of the time, she likes to wear brightly coloured clothes of blue, red and orange.

You want to be as accurate as possible with your painting and you wonder if it's possible to create paints using the same dyes used on the clothes. Like all good science-artists, you decide to investigate further...

**Wear eye protection.**

**Wear disposable plastic gloves.**

### Equipment:

- 30 g potassium aluminium sulfate, also known as alum
- 25 g potassium carbonate IRRITANT
- 100 cm<sup>3</sup> ripe buckthorn berries or juice or alternative(s) provided
- 2 beaker (250 cm<sup>3</sup>)
- 200 cm<sup>3</sup> water
- 2 Bunsen burners
- 2 heat resistant mats
- 2 tripods
- 2 gauzes
- 2 thermometers (0-110 °C)
- 2 stirring rods
- 1 filter paper
- 1 filter funnel
- 1 conical flask and bung
- 1 jar with lid for the product
- 1 dropping pipette



- 1 mortar and pestle
- Kettle, hot plate, water bath
- Thermometer
- Stirring rod
- Beaker or disposable plastic cup
- Egg yolk
- Linseed oil
- Spatula (or spoon)
- Paintbrushes

**Method:**

1. Take 100 cm<sup>3</sup> of the buckthorn juice (the dye) (or the alternative provided for you) and filter it into a flask to remove any solids that are in the solution.
2. Pour 200 cm<sup>3</sup> of water into a 250 cm<sup>3</sup> beaker. Place the beaker on a tripod and gauze, over a Bunsen burner on a heat resistant mat. Light the Bunsen burner and warm the water to 60 °C.
3. Add 10 g of alum to the beaker of warm water and stir to dissolve as much as possible.
4. Keep the solution at 60 °C and add the 100 cm<sup>3</sup> of dye.
5. Now pour 25 cm<sup>3</sup> of water into another 100 cm<sup>3</sup> beaker. Place the beaker on a tripod and gauze, over a Bunsen burner on a heat resistant mat. Light the Bunsen burner and warm the water to 60 °C.
6. Add 10 g of potassium carbonate to the warm water and stir to dissolve it. Be careful the potassium carbonate is an irritant so wash it off your skin if spilt.
7. Very carefully and slowly pour the potassium carbonate solution into the dye solution. Make sure to do this gently since there can be some effervescence. It may be advisable to place the beaker onto a tray before doing this.  
There should be some precipitation of the lake pigment.

- What gas do you think is being given off? How could you find out?
- Why do you think this chemical reaction occurred?

Leave the mixture to cool and settle. This is best if the contents are left overnight. (If time is short you can allow the solution to cool just enough to place into a cold water / ice bath.)

8. When you look at the mixture after it has settled you will see the precipitate at the bottom.



- Where do you think the precipitate has come from
  - Why do you think the precipitate has gone to the bottom of the beaker?
9. Remove the excess liquid using a dropping pipette and then pour the remains through a filter funnel with filter paper and into a conical flask. This is a very slow process since the particles are small and can clog the filter paper.
  10. Once the mixture has been filtered, lay the filter paper out on a flat surface so that the precipitate can dry. The filter paper can be placed onto a white tile and this can be placed onto a hot plate. **Needs supervision.** It can then be scraped off and collected in a jar. This is the lake pigment.
  11. Make paint with the lake pigment by using a mortar and pestle to grind the dried powder, then mixing the fine powder with either egg yolk or linseed oil.
  12. Try painting on a piece of paper, a canvas and a piece of wood.
    - Which is the best surface and if you used both egg yolk and linseed oil which made the best paint and why?

### Going further:

Evaluate the lake pigment for colour fastness with regard to light.

This will require you to design an investigation based upon what you have already done.

Try using onion skins, flower petals or red cabbage to make a lake pigment.

### Theory:

Whatever material is used the extracted colour colours the alum and this can be used as a **lake pigment** mixed with a **binder** such as egg yolk or linseed oil. The potassium aluminium sulfate (or alum,  $\text{AlK}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ ) is the reagent that forms the solid (**precipitates**) as hydrated alumina bound to the dye. This is known as the lake pigment.

The metallic salt, in this case alum, is used as the binder or **mordant** for the lake pigment must be inert, colourless, **chemically neutral** and **insoluble** in the liquid medium. In the Greek and Roman periods alum, chalk, white clay, and crushed bones were often used as sources of the binder for lake pigments. In today's industry the binders used for lake pigments are barium sulfate, calcium sulfate, aluminium hydroxide and aluminium oxide (alumina).

Many lake pigments are azo dyes. Lake pigment compounds generally have areas on the **molecule** that have a **negative charge**, often on the **chromophore** (the part of the molecule that gives it colour). The chromophore **absorbs** some **wavelengths** of light and **reflects** others that give the substance colour.



If using buckthorn you will get sap-green colour, if using woad it gives you blue, madder you get red and cochineal you will get crimson red. Changing the **pH** of the solution can change the **hue** and even in some cases the colour.

The ancient Greeks and Romans used madder to get a red and in 1815 Humphrey Davy describes a range of Roman pigments and hints at one being madder. This sample, now at the British Museum, has since been analysed and shown to be madder on a calcium sulfate (gypsum) substrate. Madder was also used by the Egyptians to dye cloth as far back as 1567 BCE. The lake pigments were important pigments for the Greek, Roman and Renaissance artists.