Experiment #5

Synthesis of Inorganic Pigments by Precipitation

Adapted from Patricia Hill

**Objective** The objective of the experiment is to synthesize two artist pigments: Prussian Blue and Chrome yellow.

**Background**

A pigment is any substance that when finely powdered gives its color to another material. A pigment is usually mixed with a “binding medium” such as oil or egg tempera. Pigments remain suspended in the binding medium when mixed to form paint in contrast to dyes, which are colored substances that dissolve in liquids and give their color effects by staining[[1]](#footnote--1).

Artist’s pigments are prepared in many ways. They are ground from naturally occurring minerals such as cinnabar (cinnamon to scarlet color), azurite (deep blue color), or malachite (green color). Or they may be prepared synthetically in the laboratory. Most pigments that we know are inorganic compounds. They are prepared by the precipitation of aqueous ions in solution, by the oxidation of metals, or by combustion or burning processes. A few colored pigments are formed through chemical synthesis and chemical change involving various organic chemicals as starting materials.

Many inorganic pigments are prepared by the mixing of two colorless or lightly colored solutions of inorganic ions to produce a highly colored, insoluble precipitate of the desired pigment. Other inorganic pigments are made by the direct combination of two or more chemicals in the solid or gaseous phase to produce the desired product.



Prussian blue is formed as a colored precipitate when two solutions of ionic, inorganic compounds are mixed together. There are several different inorganic compounds, which can be used to produce this pigment. In this experiment, the reaction of ferric chloride (FeCl3) and potassium ferrocyanide (K4(Fe(CN)6) will produce Prussian blue.



The original chrome yellow chemical formula is PbCrO4. Chromate pigments contain the ion chromate, with formula CrO42− which must be carefully disposed as it is toxic and considered hazardous waste. Unfortunately, chrome yellow also contains lead, a very toxic heavy metal. Zinc chrome yellow is an alternative yellow pigment that does not contain lead. It is formally basic zinc chromate, ZnCrO4•Zn(OH)2. We will make this pigment first by mixing potassium chromate K2CrO4 and zinc chloride ZnCl2 solutions to produce Zinc chromate through a precipitation reaction[[2]](#footnote-0) and then adding NaOH to make the solution basic. The result is solid yellow ZnCrO4•Zn(OH)2.

**Reactions**

**Prussian Blue**

4FeCl3🞄6H2O + 3K4(Fe(CN)6)🞄3H2O 🡪 Fe4(Fe(CN)6)3🞄14H2O +12 KCl

**Zinc Chrome Yellow**

Na2CrO4 + ZnSO4 🡪 ZnCrO4 + Na2SO4

ZnCrO4 + NaOH 🡪 Zn(OH)2

Apparatus Chemicals

Balance Ferric Chloride

Filter Paper Potassium ferrocyanide

Stirring Rod Sodium chromate

3 beakers (25 mL) Zinc Sulfate

10 mL graduated Cylinder 6M NaOH

Buchner Funnel

Filtering flask

**Procedure**

Part I: Preparation of Prussian Blue

1. Weigh accurately about 1.480 g of FeCl3 (ferric chloride)in a small beaker labeled Prussian blue and mix with 5 mL of distilled water. Mix the solution until it is all dissolved.
2. Weigh accurately 0.556 g of potassium ferrocyanide, K4[Fe(CN)6] into a small beaker and mix with 5 mL of distilled water . Stir until dissolved.
3. Slowly pour the solution of potassium ferrocyanide into the beaker labeled Prussian Blue and stir the mixture. Let the reaction continue and record the results you see.
4. Weigh accurately a small filter paper and place in the filtration apparatus. Filter the precipitate of Prussian blue that forms using the house vacuum.
5. Allow the pigment to dry uncovered on the filter paper until the next lab period in your lab drawer on top of a paper towel.
6. Weigh the dried pigment and filter and subtract the initial weight of the filter paper to obtain the weight of the pigment.

Part II: Preparation of Chrome Yellow

1. Weigh approximately 0.20 g of sodium chromate (Na2CrO4) in a beaker labeled “Chrome Yellow” and mix with 2 mL of distilled water. Stir the solution until completely dissolved.
2. Weigh approximately 0.90 g of zinc sulfate (ZnSO4) in a small beaker and mix with 2 mL of distilled water and stir.
3. Pour the solution of zinc sulfate into the solution labeled “Chrome Yellow” and mix thoroughly.
4. Add about 0.5 mL of 6M NaOH to make the solution basic. Stir well. The color will change from yellow-orange to bright yellow.
5. Weigh a small filter and place in the filtration apparatus. Filter the precipitate of zinc chromate (ZnCrO4), which forms if necessary.
6. Allow the pigment to dry uncovered on the filter paper until the next lab period in your lab drawer on top of a paper towel.
7. Weigh the dried pigment and filter and subtract the weight of the filter paper to obtain the weight of the pigment.

Post-Lab Assignment

**Write a lab report for this experiment.**

*In your introduction* describe a brief history of the pigments to be synthesized and make sure you include the following:

1. When were Prussian blue and chrome yellow first synthesized or discovered.
2. When were these pigments popular? Are they still used today?
3. What are the actual health hazards of lead, chromium and zinc.
4. Make sure to cite your sources.

*In your experimental section*

1. Explain in detail the experiment procedure followed and describe your observations.

*In your results and discussion*

1. Show your experimental data in table format (tabulated)
2. Calculate the percent yield based on the chemical reaction and the limiting reagent for the synthesis of Prussian blue.
3. Explain why it was not possible ot calculate the percent yield for zinc chrome yellow.
4. Show all your calculations.

*In your conclusion* describe what you learned in this lab.

1. Mayer, R. The Artist’s Handbook of Materials and Techniques. 1981 Viking Press. p.30 [↑](#footnote-ref--1)
2. Brady and Senese. Chemistry: Matter and its Changes, 2009, 5th ed. p.146 [↑](#footnote-ref-0)