

A Few Microchemical Experiments

Introduction

Not so long ago when modern analytical equipment enabling a full analysis of extremely small samples of only a few mg was not available, wet chemical methods were used. An example of that type of analysis one might remember from school days. The way to determine whether chloride ions are present in a solution is to add a small amount of silver nitrate to that solution. The formation of a white deposit demonstrates the presence of chloride ions ($\text{Ag}^+ + \text{Cl}^- \rightarrow \text{AgCl (s)}$).

At the end of the 19th and for a large part of the 20th century microchemistry was used for trace analysis whereby the microscope was an essential apparatus. The microscope was used for observing a chemical reaction taking place in a droplet placed on an object glass and observe the crystals formed. The experiments described in this article result in the formation of typifying crystals.

Unless otherwise stated the methodology employed for these experiments is straightforward. A droplet of the solution to be investigated is put on an object glass. After placing the object glass under the microscope a grain or a few microliters of the reactant is positioned alongside the droplet (See Figure 1). The microscope can be focused using this grain of reactant. Subsequently the materials are mixed. After having observed the reaction one can evaporate the water by placing the object glass on a hot plate warmer and make a dry mount slide.

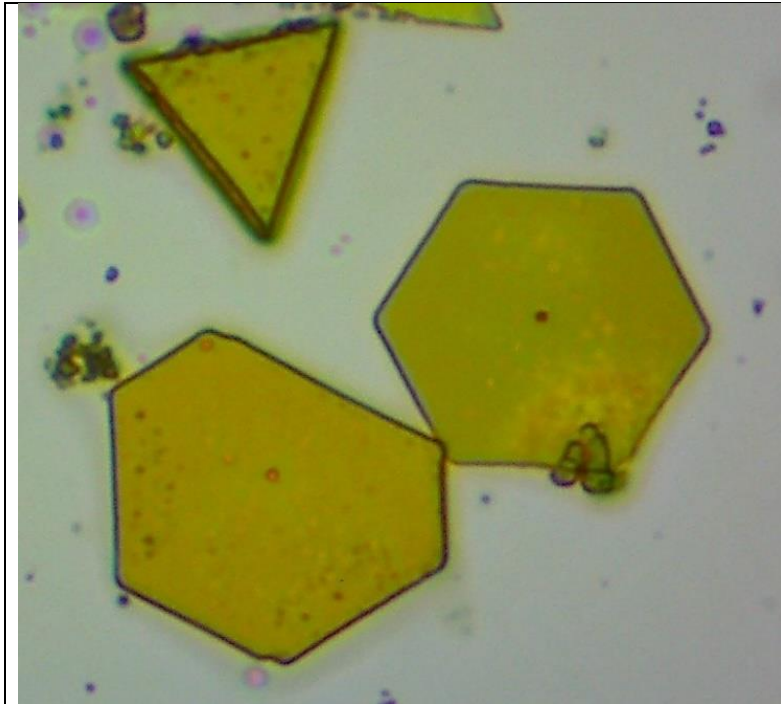


Figure 1: Methodology

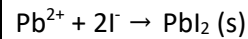
For these experiments an Euromex BioBlue BB.4253 Trinocular microscope extended with a polarization kit was used. The pictures were made with a 2MP Celestron Microscope Imager 822484 ocular (magnification approximately 15x).

Lead iodide crystals

Place a small droplet of lead acetate ($\text{Pb}(\text{Ac})_2$) solution on the object glass. Add a bit of water to dilute the solution. Move a grain of potassium iodide (KI) inside the drop let and mix.



Reaction:



Observation:

Immediate formation of a yellow precipitate.

Crystal structure:

hexagonal or rhombic

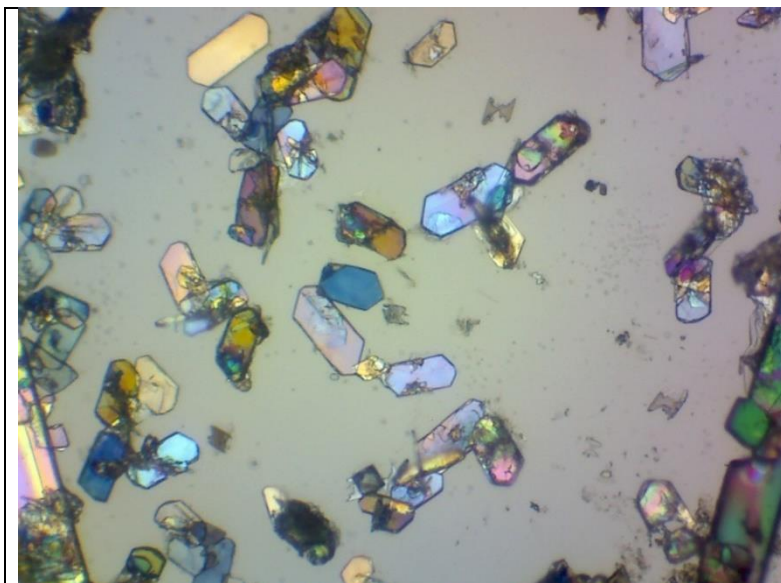
Magnification objective lens:

4x

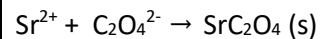
Figure 2: Lead iodide crystals

Strontium oxalate crystals

Add a grain of oxalic acid to a drop of strontium chloride solution.



Reaction:



Observation:

After some time one can observe the formation of the strontium oxalate crystals. In the dried sample one can also observe the presence of needle like crystals from oxalic acid.

Crystal structure:

Bipyramidal or prismatic

Magnification objective lens:

10x

Figure 3: Strontium oxalate crystals

Silver phosphate crystals

Add to a drop of silver nitrate solution (AgNO_3), 1M a grain of mono ammonium phosphate ($\text{NH}_4\text{H}_2\text{PO}_4$).

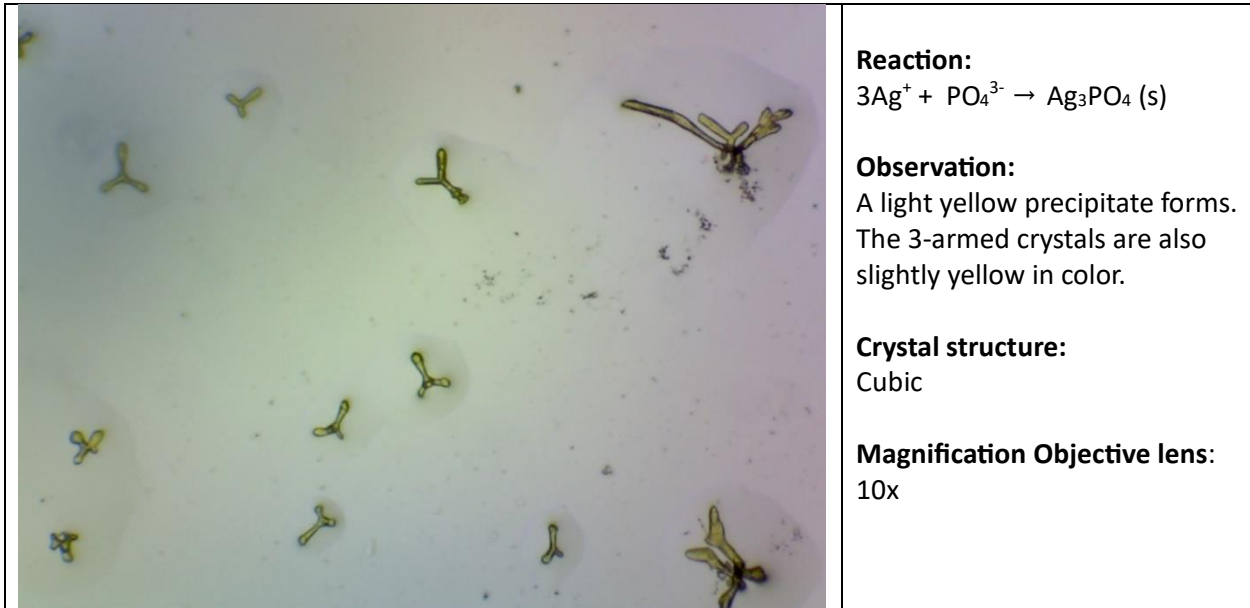


Figure 4: Silver phosphate crystals

Silver chromate crystals

Add a grain of potassium bichromate ($\text{K}_2\text{Cr}_2\text{O}_7$) to a solution of silver nitrate (AgNO_3), 1M.

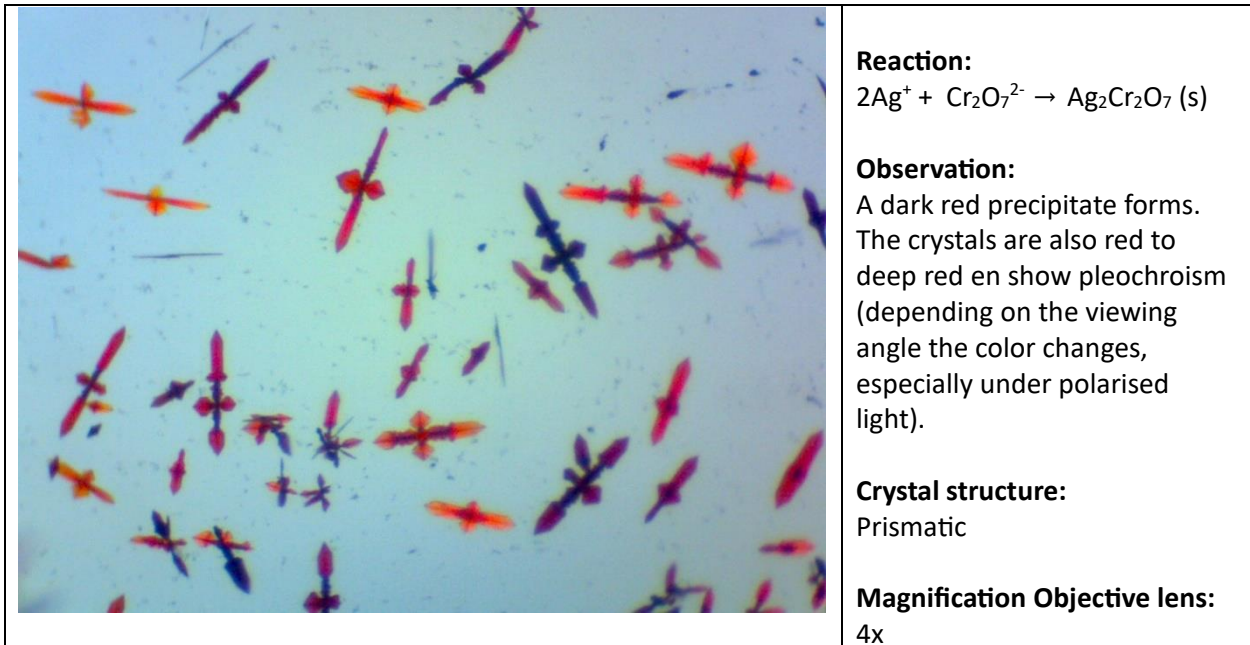


Figure 5: Silver chromate crystals

Barium chromate crystals

Add to a solution of barium acetate ($\text{Ba}(\text{Ac})_2$) a grain of potassium bichromate ($\text{K}_2\text{Cr}_2\text{O}_7$).

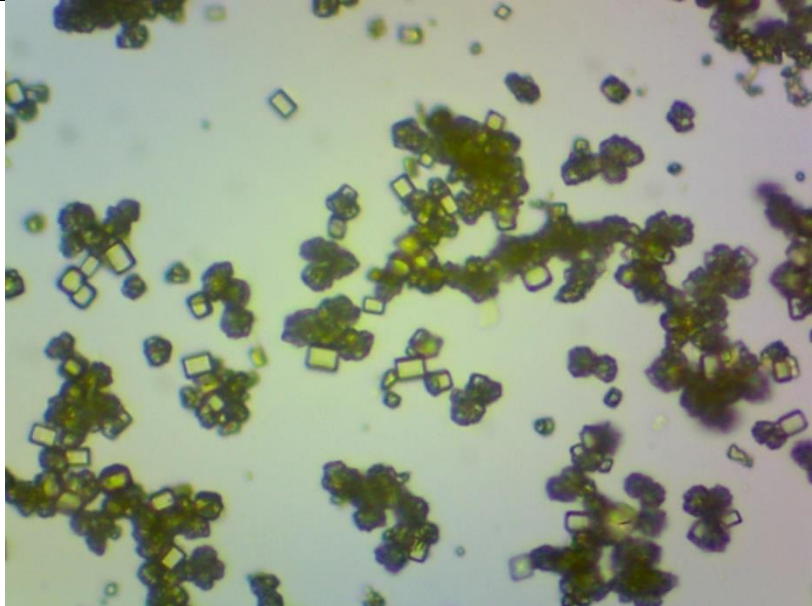
	<p>Reaction: $\text{Ba}^{2+} + \text{Cr}_2\text{O}_7^{2-} \rightarrow \text{BaCr}_2\text{O}_7 (\text{s})$</p> <p>Observation: The precipitate formed has a light yellow color. The crystals formed have a slightly yellow color and a shape of rectangles and diamonds</p> <p>Crystal structure: Rhombic</p> <p>Magnification objective lens: 40x</p>
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Figure 6: Barium chromate crystals

Ammonium tin chloride crystals

Acidify a solution of tin chloride (SnCl_2) with diluted hydrochloric acid (HCl). Put a droplet on an object glass and add a grain of ammonium chloride (NH_4Cl). Mix and put the object glass on a hot plate.

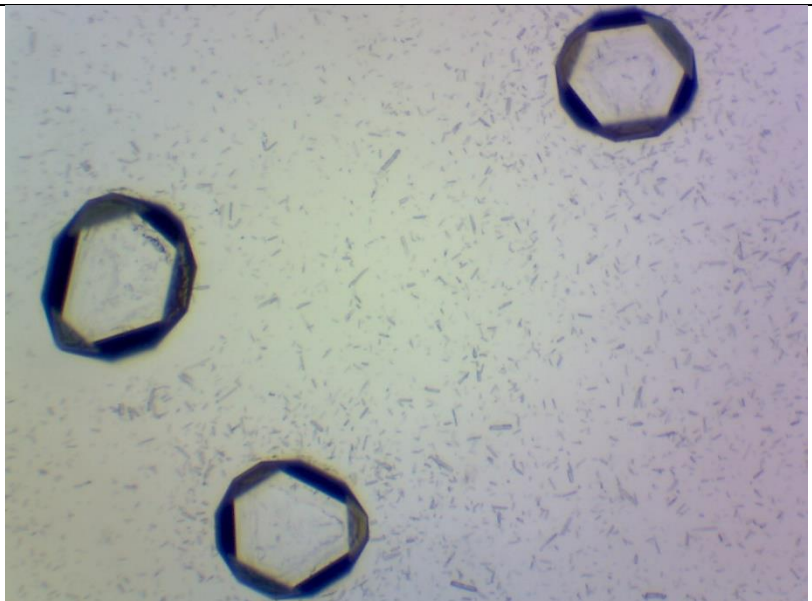
	<p>Reaction: $\text{Sn}^{2+} + 4\text{Cl}^- + 2\text{NH}_4^+ \rightarrow (\text{NH}_4)_2\text{SnCl}_4 (\text{s})$</p> <p>Observation: White crystals form after evaporation of the water</p> <p>Crystal structure: Octahedric</p> <p>Magnification Objective lens: 4x</p>
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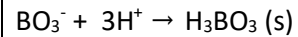
Figure 7: Ammonium tin chloride crystals

Boric acid crystals

A solution of sodium perborate ($\text{NaBO}_3 \cdot 4\text{H}_2\text{O}$) is acidified by adding hydrochloric acid (HCl) solution. A droplet is put on an object glass and placed on a hot plate.



Reaction:



Observation:

Colorless /whitish crystals form after evaporation of the water.

Crystal structure:

Triclinic

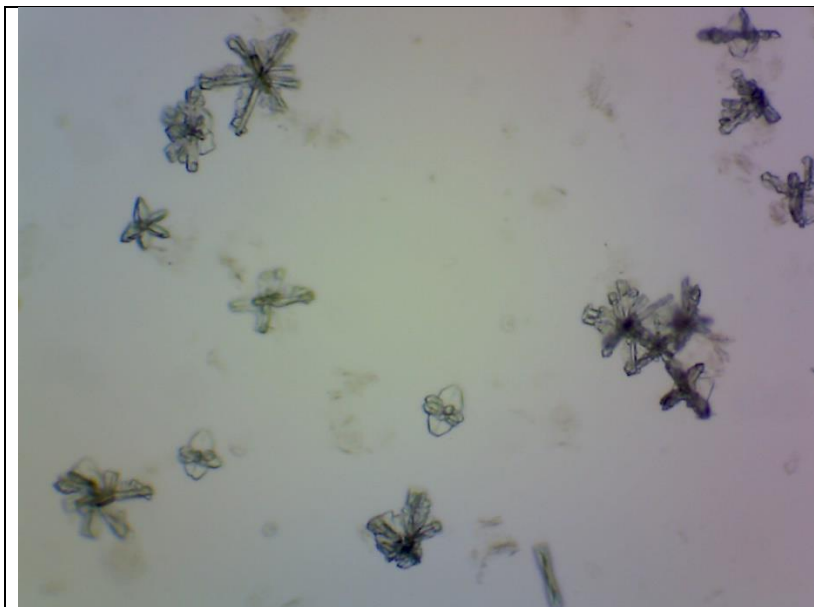
Magnification Objective lens:

10x

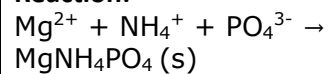
Figure 8: Boric acid crystals

Magnesium ammonium phosphate crystals

Place a drop of magnesium carbonate solution (MgCO_3) on an object glass. Add ammonium chloride (NH_4Cl) and a drop of concentrated ammonia solution (25%). Mix well. Place a grain of mono ammonium phosphate ($\text{NH}_4\text{H}_2\text{PO}_4$) in the solution



Reaction:



Observation:

Formation of colorless crystals. Radially growing, feather like structures.

Crystal structure:

Magnification Objective lens:

10x

Figure 9: Magnesium ammonium phosphate crystals

Potassium hydrogen tartrate crystals

Add a grain of tartaric acid ($C_4H_6O_6$) to a solution of potassium chloride (KCl).

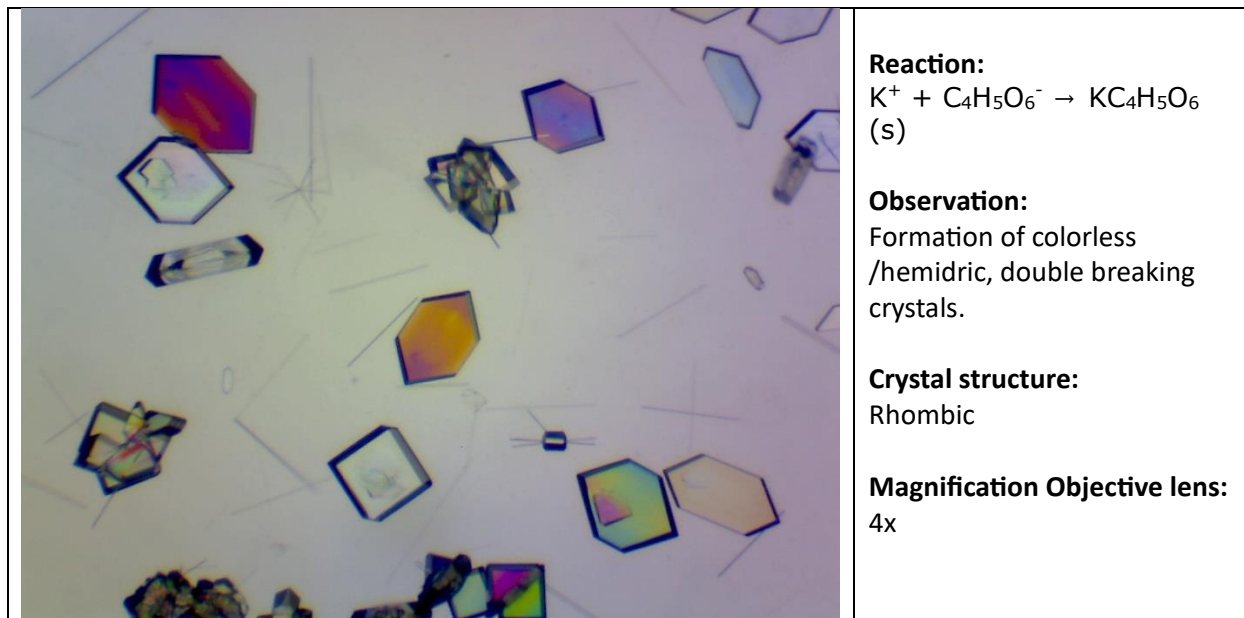


Figure 10: Potassium Hydrogen Tartrate crystals

Remarks

- An oxalic acid solution is commonly used to remove stains from wood and stone. In the Netherlands it can be found as an approximately 10% solution in DIY/Home Improvements stores where it is called “ontweringswater”. One can remove the water by evaporation. Oxalic acid itself forms large needle like crystals.
- The lead acetate solution used in this experiment was made by first putting a piece of lead in a solution of 3% hydrogen peroxide and after decanting, in an acetic acid solution. Leave the solution standing for some time to ensure that lead has dissolved in the acetic acid. Filter the solution before use.

Literature

- Wilhelm Geilmann; “Bilder zur Qualitativen Mikroanalyse Anorganische Stoffe”; 3^e druk; Verlag Chemie; 1960.
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