SILVER ACETATE CRYSTALS

INTRODUCTION

Having an interest in older Science books I regular browse the web for books that are being made electronically available, to see if I can find something that interests me. On the website of the Dutch Royal Library if found an old Dutch book "Organische chemie in een proefbuis"[1], which described some interesting experiments. On page 51 of that book an identification reaction for acetic acid was described that caught my attention. A qualitative identification reaction for acetic acid that creates characteristic microcrystals when using silver nitrate as a reagent. This surprised me as to my knowledge, nowadays, ferric chloride is used to test for the presence of carbonic acids. All in all, interesting enough to investigate further.

MATERIALS

- Acetic Acid
- Sodium hydroxide solution (NaOH, ca. 4% aq)
- Silver nitrate solution ($AgNO_3$, 0.05 M aq)
- Gedemineraliseerd water
- Ferric chloride solution ($FeCl_3$, ca. 0.5 M aq, neutralized)
- pH papers
- Erlenmeyer 30 ml
- Stirring rod
- Polarization microscope (Optika B-165)
- Microscope camera (Lucky Zoom USB5M)
- Glass slides
- Cover glasses
- Dissecting Needle
- USB heating plate
- Spot test plate

Figure 1: Microscope

Figure 2: USB heating plate

Figure 3: Lucky Zoom 5Mp camera

METHOD

- Pour a little bit of acetic acid in the erlenmeyer
- Add, while stirring with the stirring rod, small amounts of sodium hydroxide
- Regularly use a pH paper to measure the pH
- Stop adding sodium hydroxide when the solution is neutral
- Take a drop of the solution and put it on the glass plate
- Add a drop of silver nitrate solution
- Put the glass slide on the heating plate en let some solution evaporate
- Carefully put a cover glass on the crystals
- Observe under the microscope

Figure 4: Preparing the test solution

- Take a few drops of solution and put these in a cavity on the spot test plate
- Add a drop of ferric chloride solution
- Observe
- Take a drop of the solution and put it on the glass plate
- Carefully put a cover glass on the crystals
- Observe under the microscope

Figure 5: Spot test plate

RESULTS

Figure 6: Silver acetate crystals (obj. 10x)

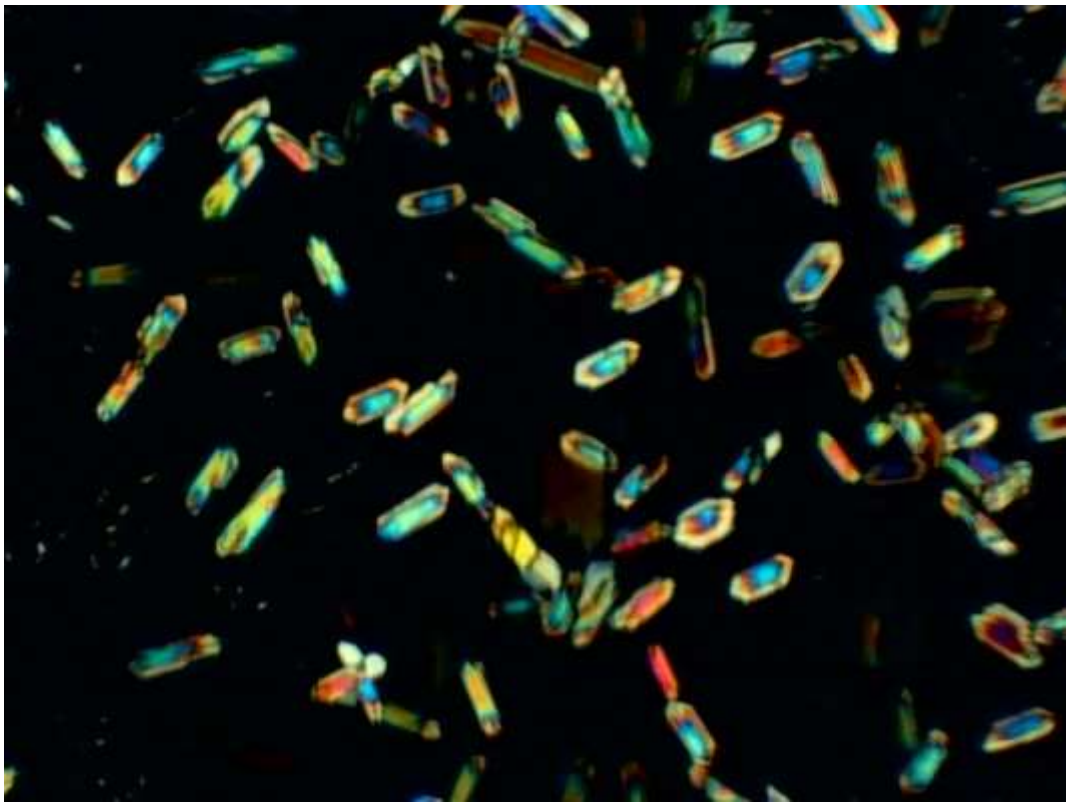


Figure 7: Silver acetate crystals in polarized light (obj. 10 x)

On the spot test plate, the solution turns red-brown and a precipitate forms. When observing the material under de microscope the precipitate is amorphous.

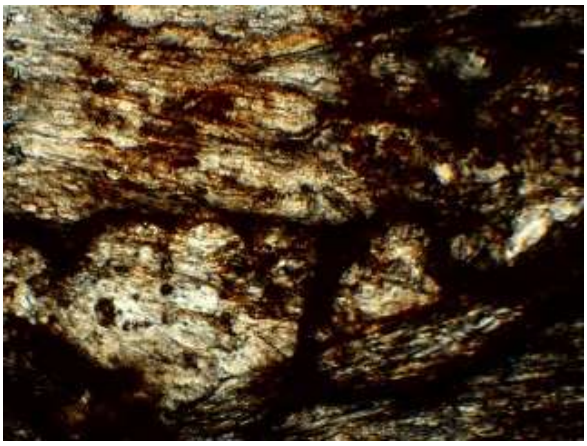


Figure 8: Amorphous ferric acetate (obj. 4x)

DISCUSSION

Ferric chloride reaction

In the more recent literature the qualitative identification reaction mentioned for acetates/carbonic acids where a precipitate is formed is indeed the reaction with ferric chloride ($FeCl_3$), whereby the formation of a red-brown color is indicative for the presence of these components [2-5]. Most probably according to the following reaction whereby an iron acetate complex is formed:

Fe^{3+} (aq) + $3CH_3COO^-$ (aq) ⇔ $(CH_3COO)_3Fe$ (s)

Silver acetate reaction

To find more information about the use of silver nitrate as identification reaction for carbonic acids a more extensive literature search was needed.

Behrens[9] mentions the reaction specifically as an identification reaction for carbonic acids. The illustration found in the book resembles the observations made, somewhat. The textual information presented more closely resembles the observations made i.e. the formation of six edged and diamond shaded (rhombic) crystals that can cluster to form star shaped groups.

The crystals in the pictures presented above are typically around 200 microns in length (Range: 150 - 350 micron)

The information found in the literature on silver acetate crystals agrees with the crystals, as they are observed under the microscope, in this experiment.

In general, there appears to be not much information available on silver acetate complexes [6-8], although the structural formula is known [7].

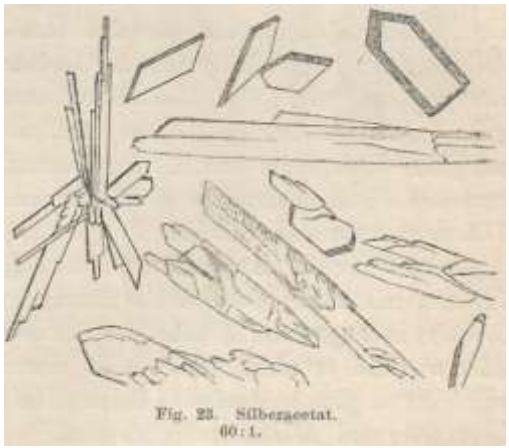


Figure 9: Reference pictures [9,1]

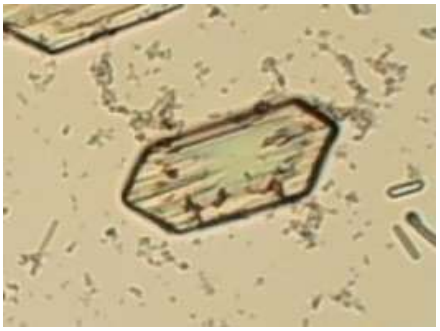


Figure 10: Picture of a single silver acetate crystal

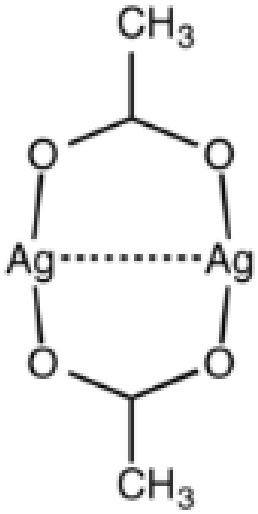


Figure 11: Structural formula silver acetate

The original question, that initiated this experiment was why an identification reaction for organic acids that is mentioned in older books, was replaced by the ferric chloride test. The most probable reason for that is that the test is less reliable than originally believed. A first indication that this could be the case can be found in the article of Giroud and Leblond [6]. They describe the identification of ascorbic acid (Vitamine C) with silver nitrate in animal cells. At the end of the article it is mentioned that a false negative result is found at certain conditions. In the article of Halsberghe et al [9] both reactions, the ferric chloride and the silver nitrate test are discussed as an identification reaction for acetates. In the article it is also mentioned that carbonates can form a white amorphous precipitate which cannot be distinguished from silver chloride precipitates (identification reaction for chlorides). That problem is circumvented by acidifying the solution which means that the solution cannot be used anymore to test for the presence of acetates. Especially for micro chemical analysis, whereby only a small

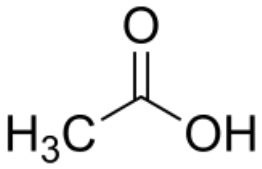
amount of sample is available this can be an issue. Final confirmation on why the silver nitrate reaction is not used anymore to test for carbonic acids was found in the book from Jander and Blasius[10]. The book states:

"$AgNO_3$
Nur in konz. Lsgg. Weisser Nd. Von $Ag(CH_3COO)$. Wenig charakteristisch."

Which can be translated as meaning that the reaction only works well in concentrated solutions and that the reaction is not unambiguous.

REMARK

- White vinegar, the acetic acid used for cleaning is more suitable for these experiments than the one used in cooking because the concentration of acetic acid is higher (8-15%).
- Structural formula acetic acid:



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