

BENZOIC ACID CRYSTALS

INTRODUCTION

Preparing microcrystals by sublimation is a method described in scientific literature but is not that easy to conduct in a home laboratory as it can be difficult to find suitable materials. With sublimation we change the state of the material from solid to gas applying heat and subsequently deposit the material on cold surface. One of the materials suitable for making micro crystals in this manner is benzoic acid as it has a relatively low melting point (122.4 °C)¹.

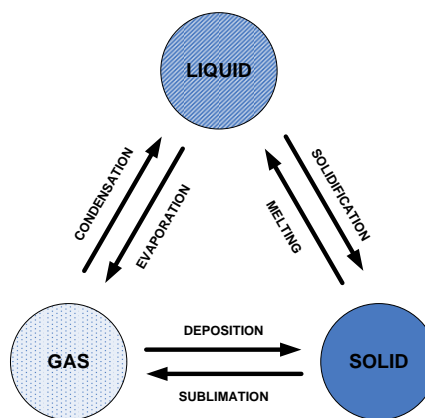


Figure 1: Changes of State Triangle

The primary aim of this experiment is to create benzoic acid crystals by sublimation. A secondary goal is to produce a permanent slide of these micro crystals.

MATERIALS

- Benzoic Acid
- USB Heating plate
- USB Peltier Heater/Cooler
- Test tube with stopper
- Polarisation microscope (Euromex ML2000)
- Microscope camera (Lucky Zoom USB5M)
- Glass slides
- Cover glasses
- Bronze weights
- Dissecting Needle
- Spatula
- Cutting knife
- Adhesive tape
- “Spacers”



Figure 2: Euromex ML2000 microscope



Figure 3: Lucky Zoom 5Mp camera

For preparing crystals (see also Figure 5)

- Put weights on the USB cooler and switch it on in cooling mode.
- Place a glass slide on the USB heater and switch the heater on.
- Put a small amount of benzoic acid in the middle of the glass slide.
- Position the spacers alongside the glass slide.
- Place a second glass slide perpendicular to the first one on the spacers.
- Put a cold weight on top of this glass slide.
- Exchange the cold and hot weights on a regular basis.
- After a short period of time can observe the formation of micro crystals on the second glass slide.
- Take the glass slide away for further handling.

For making permanent slides

- Place a cover slide on the glass slide containing the sublimated benzoic acid crystals.
- Fasten the cover glass using adhesive tape in the manner as depicted in step 3 and 4 in scheme presented in Figure 4.
- Use a cutting knife and a second glass slide to cut away the superfluous adhesive tape.

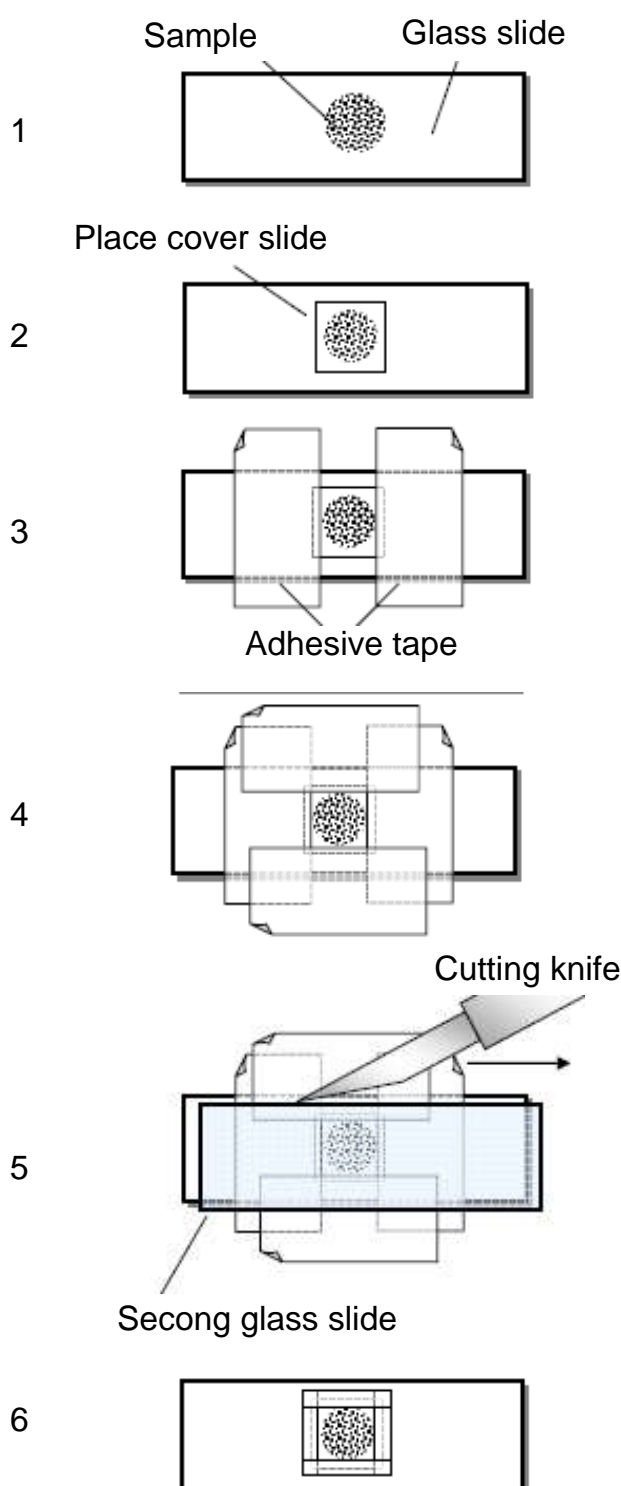


Figure 4: Methodology for making a permanent slide of micro crystals



Figure 5: Setup for sublimation

RESULTS

The pictures below were taken with the Euromex ML2000 polarization microscope and the Lucky Zoom YW500 HD 5MP USB CMOS Camera Electronic Digital Eyepiece using a Euromex Phase DM 20X / 0.40 DIN objective lens.



Figure 6: The permanent slide produced.



Figure 7: Benzoic acid crystals prepared by sublimation



Figure 8: Benzoic acid crystals prepared by sublimation – darkfield

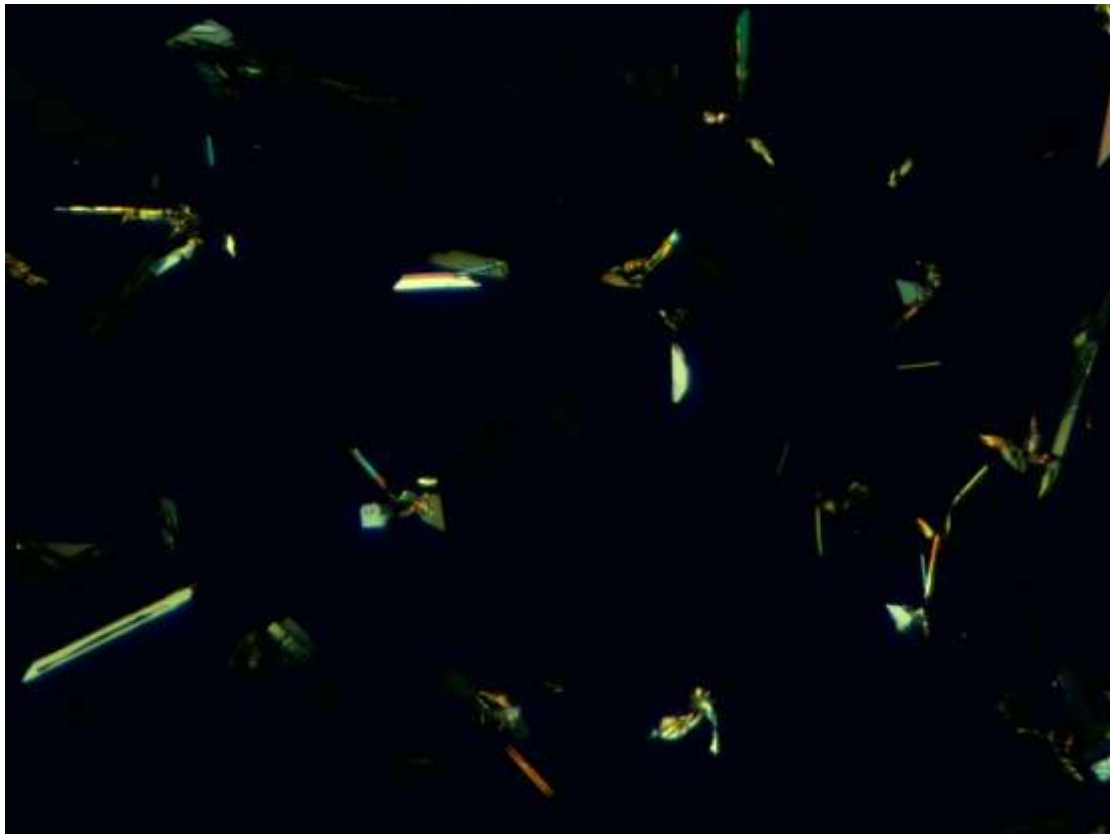


Figure 9: Benzoic acid crystals prepared by sublimation - polarization

DISCUSSION

Benzoic acid crystals are monoclinic in shape and should therefore have a needle like shape. This can indeed be observed in the pictures taken. However, I was not able to find a reference picture of individual benzoic acid crystals prepared by sublimation (on the web). The only reference I could find was in Molisch², who isolated the crystals by sublimation from berries.

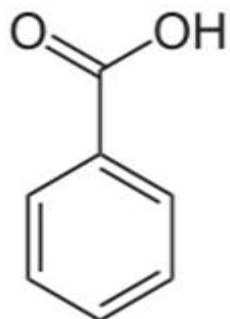


Figure 10: Structural formula Benzoic Acid
(m.w. 122.12 g/mol, C₇H₆O₂)

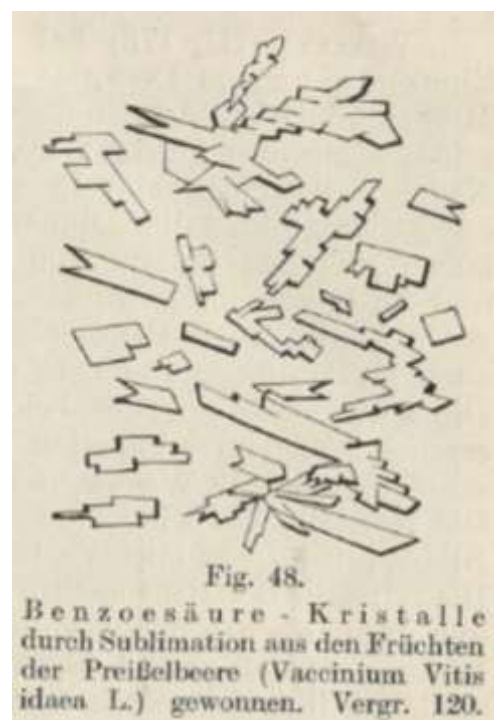


Figure 11: Reference picture

REMARKS

- I used weights but any solid block of heat conducting metal should work as well.
- Off-course one could also use a small container filled with ice, instead of the cooled down weights.
- The spacers I used were taken from a Meccano kit.
- The methodology for making permanent slides in this manner was found on a German website "[Einführungskurs in die Mikroskopie](#)".
- The [USB Peltier Cooler/Heater](#) was purchased at AliExpress

LITERATURE

1. The Merck Index; 11th Ed.; 1989; ISBN 91191028X; p. 1099.
2. Hans Molisch; "Mikrochemie der Pflanze"; Jena; 1913; p.140, 141.

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