

# MICROSCOPICAL EXPLORATION TWELVE

## An investigation into the crystal structures of commercially available aspirin brands following addition of caffeine and recrystallization from solution at high concentration in ethyl ethanoate

### Introduction

The solubility of acetylsalicylic acid (aspirin) decreases in the order of ethanol, ethyl ethanoate, carbon tetrachloride, xylene, and water. Due to the easy availability of, the relatively high solubility of aspirin in and the low boiling point/high volatility of ethyl ethanoate, this is the preferred solvent for this investigation. For the investigation six different brands of 300mg aspirin tablets were chosen. The procedure detailed below was carried out separately for each brand. A control solution of caffeine alone was also prepared and treated similarly.

### Aim

To identify by conventional and polarised optical microscopy any variations in the crystal structures of commercially available aspirin brands following the addition of caffeine when recrystallized from solution in ethyl ethanoate.

### Applicability

The procedure is applicable to any commercially available aspirin tablets.

### Equipment

Small glass bottles with lids.

Filter funnel and filter paper

Mortar & pestle or two teaspoons (for crushing tablets)

Vickers M10A optical microscope equipped with incandescent filament bulb white light illumination and sub-stage condenser.

Polarising filter and analyser

Digital eyepiece camera (Brunel Microscopes Ltd. Eyecam) to replace the x10 microscope eyepiece.

Microscope slides

Dropping pipette

### Materials/Reagents

Branded aspirin tablets: Anadin Original, Aspar brand, Boots brand, Morrisons brand, Tesco brand, Wilko brand. It should be noted that Anadin Original tablets also contain approx. 4.4%w/w caffeine.

Caffeine tablets (50mg)

Ethyl ethanoate (acetone free nail polish remover).

### Procedure

Tablets containing a total of 900mg of aspirin were finely crushed, and the resulting powder was well mixed with 20cm<sup>3</sup> of ethyl ethanoate, at room temperature, and allowed to equilibrate for 30 mins. Assuming complete dissolution of the aspirin this yields a solution approaching saturation at approximately 45 mg/cm<sup>3</sup>. To this solution was added, with mixing, one finely crushed 50mg caffeine tablet. The solution was allowed to equilibrate once again and then clarified by filtration through filter paper circles cut from coffee filters. Two drops of the clear solution were applied to the centre of a glass microscope slide and allowed to evaporate slowly overnight at room temperature. The crystals of aspirin/caffeine thus formed on the slide were observed microscopically and photographed for each brand as follows:

**Image 1** X4 objective with transmitted white light illumination and sub-stage condenser only.

**Image 2** X4 objective with transmitted white light illumination, polarizing filter immediately above the sub-stage condenser and analyser between the microscope objective turret and the digital eyepiece camera.

Observations

Anadin Original ( containing c.4.4% caffeine)

Image 1

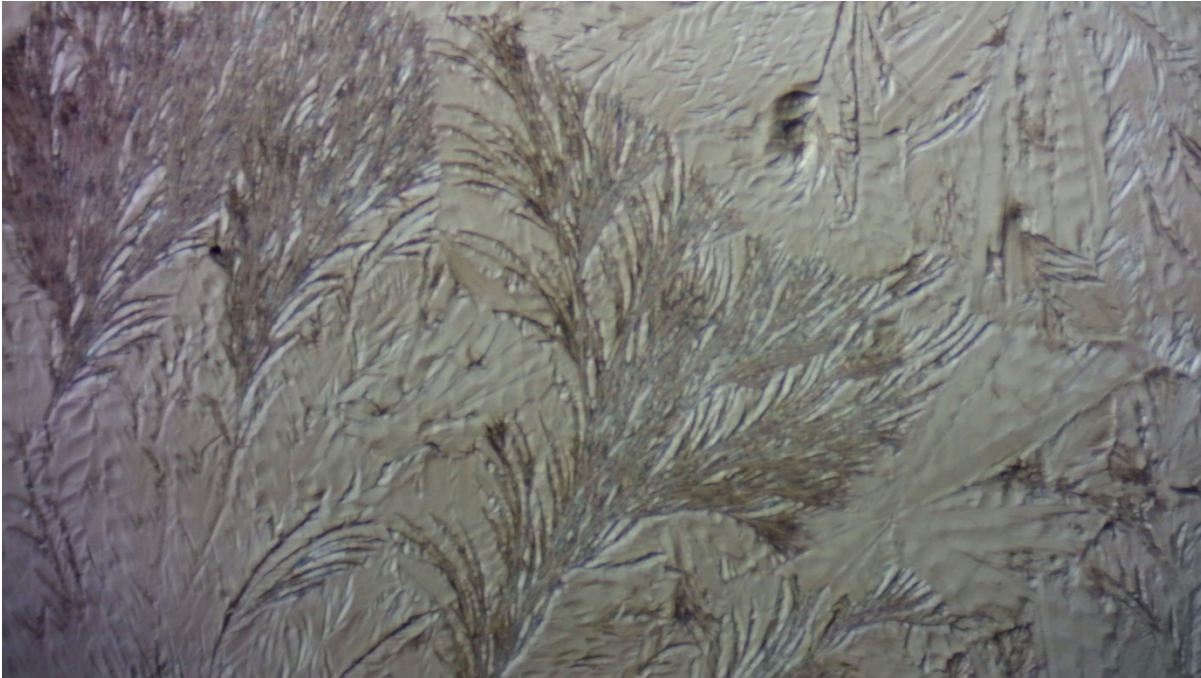
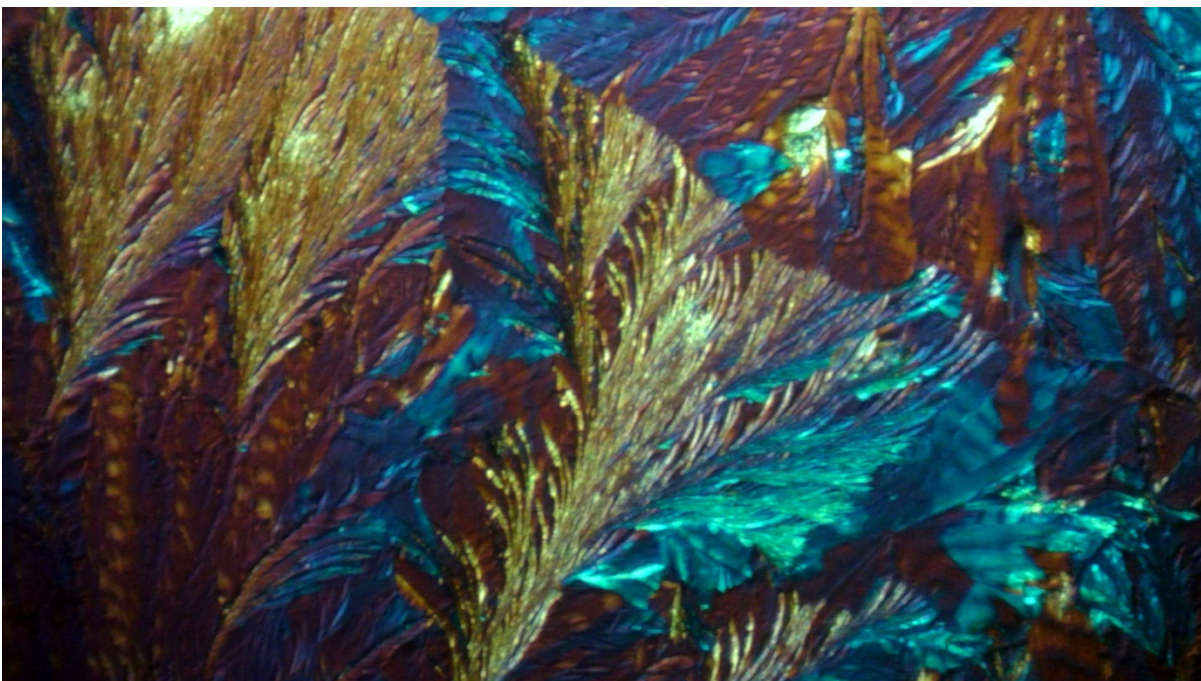


Image 2



Aspar Brand + caffeine

Image 1

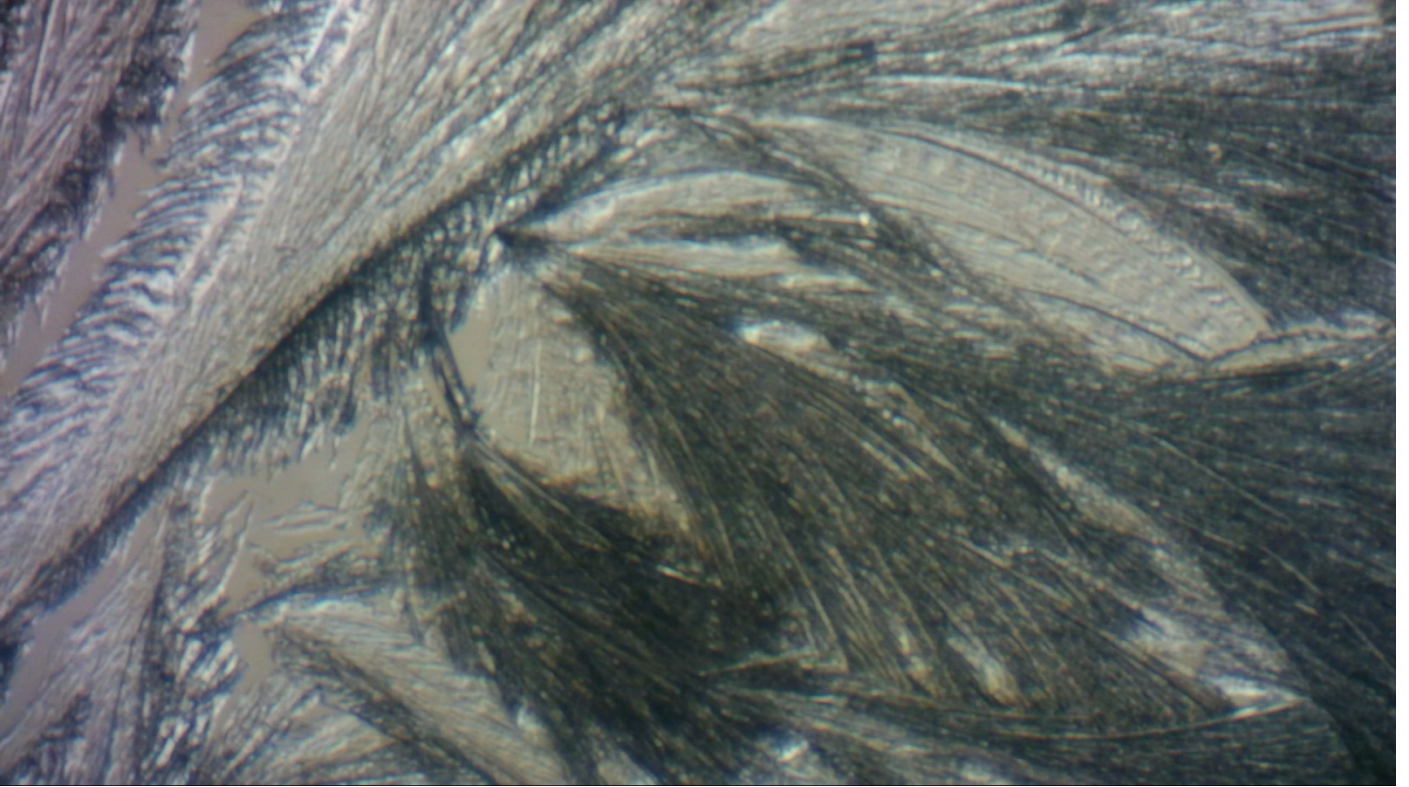
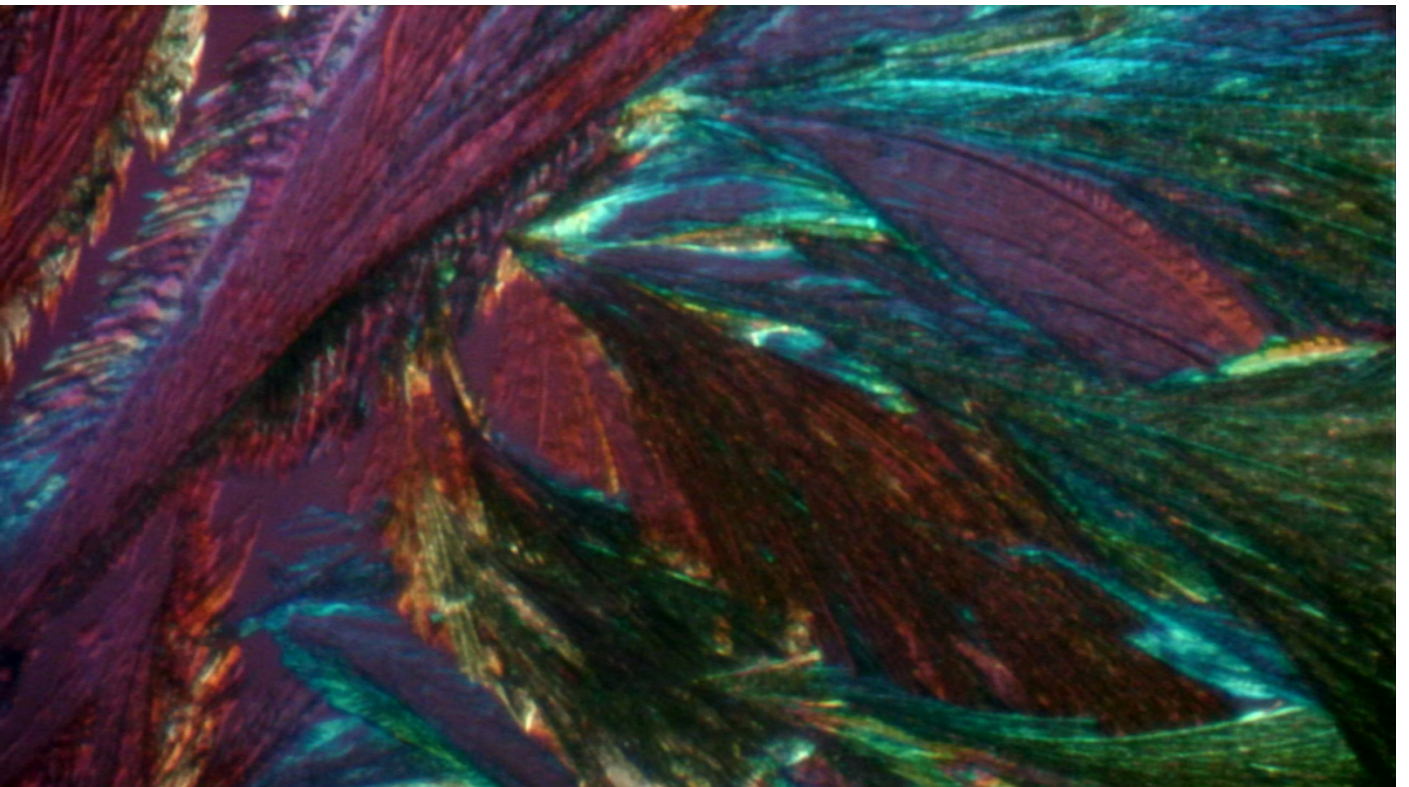
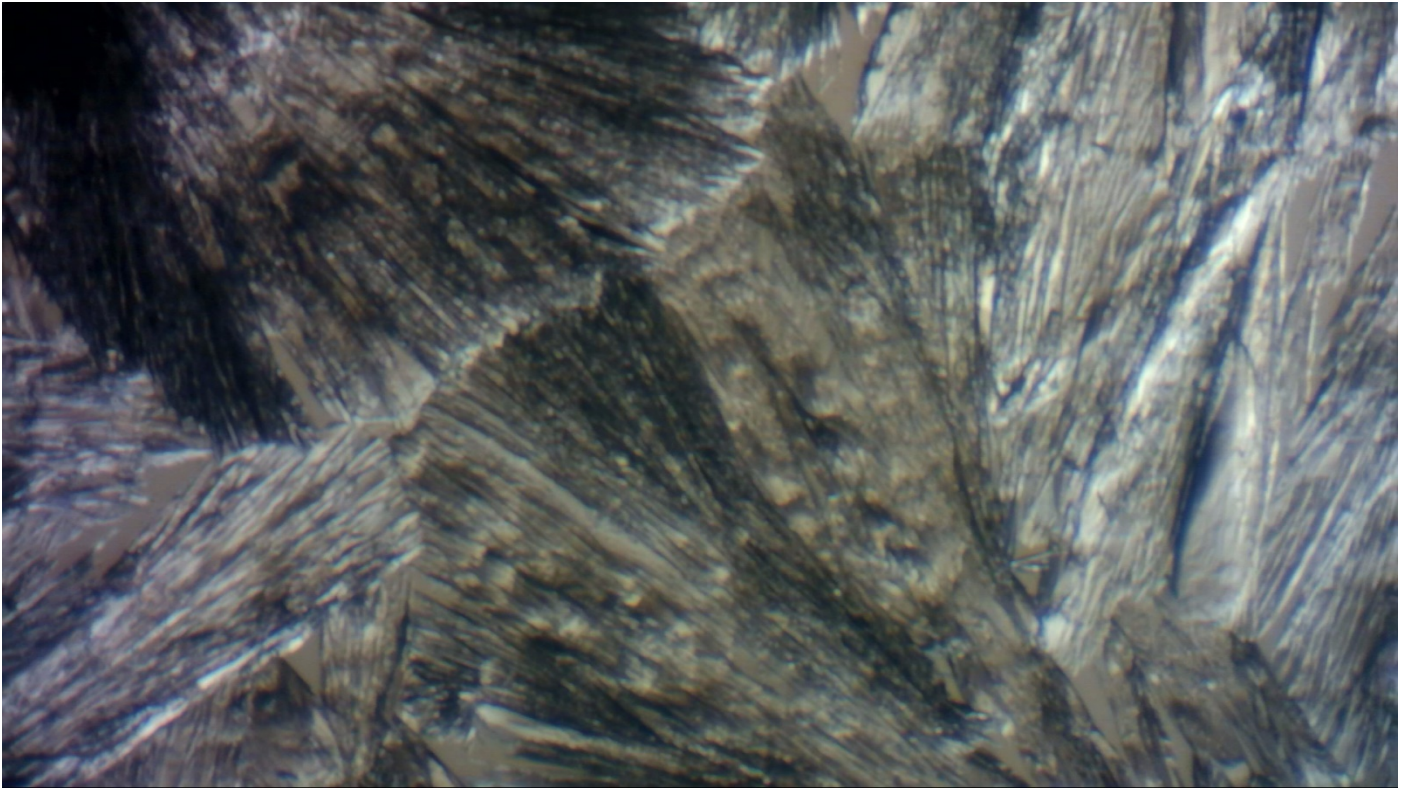


Image 2

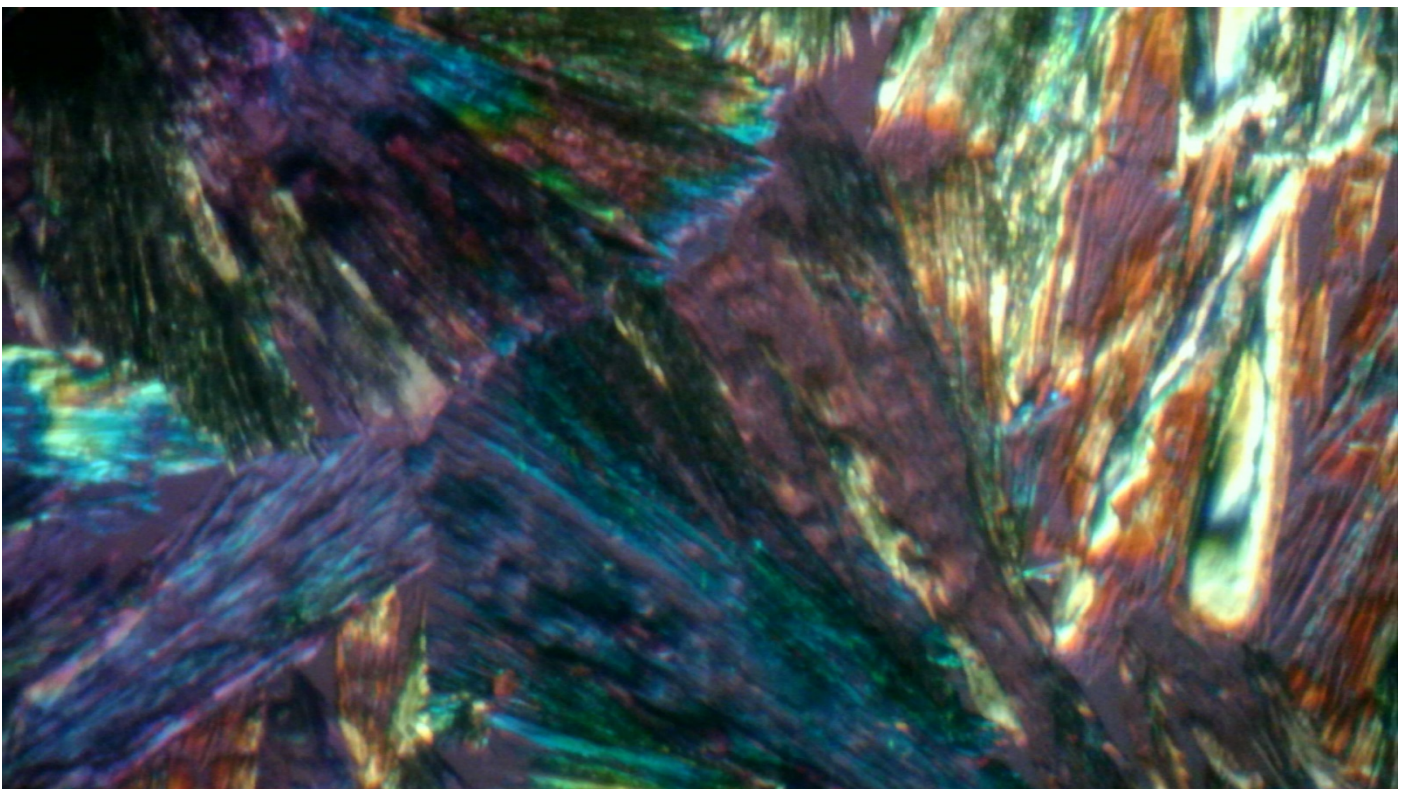


**Boots Brand + caffeine**

**Image 1**



**Image 2**



Morrison's Brand + caffeine

Image 1

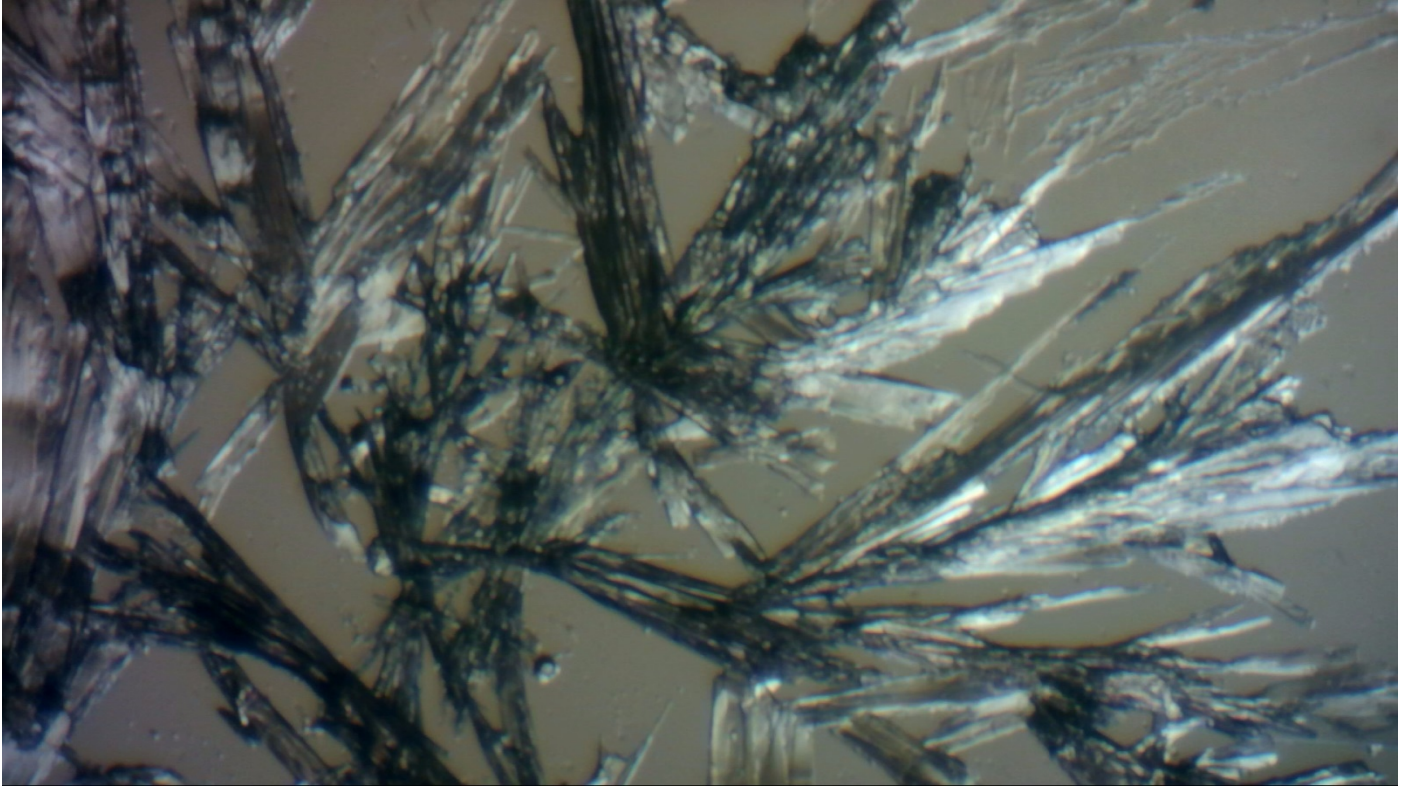
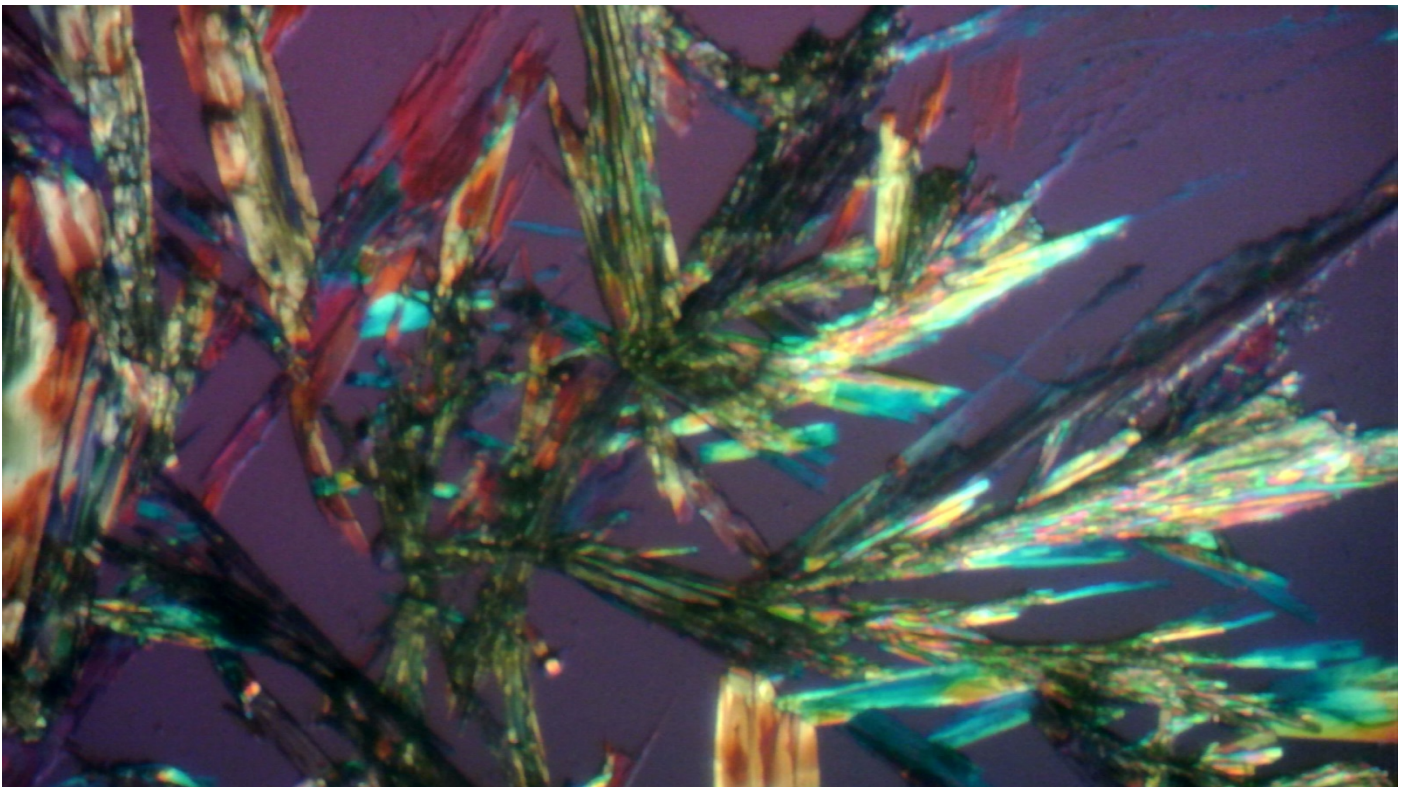


Image 2



Tesco Brand + caffeine

Image 1

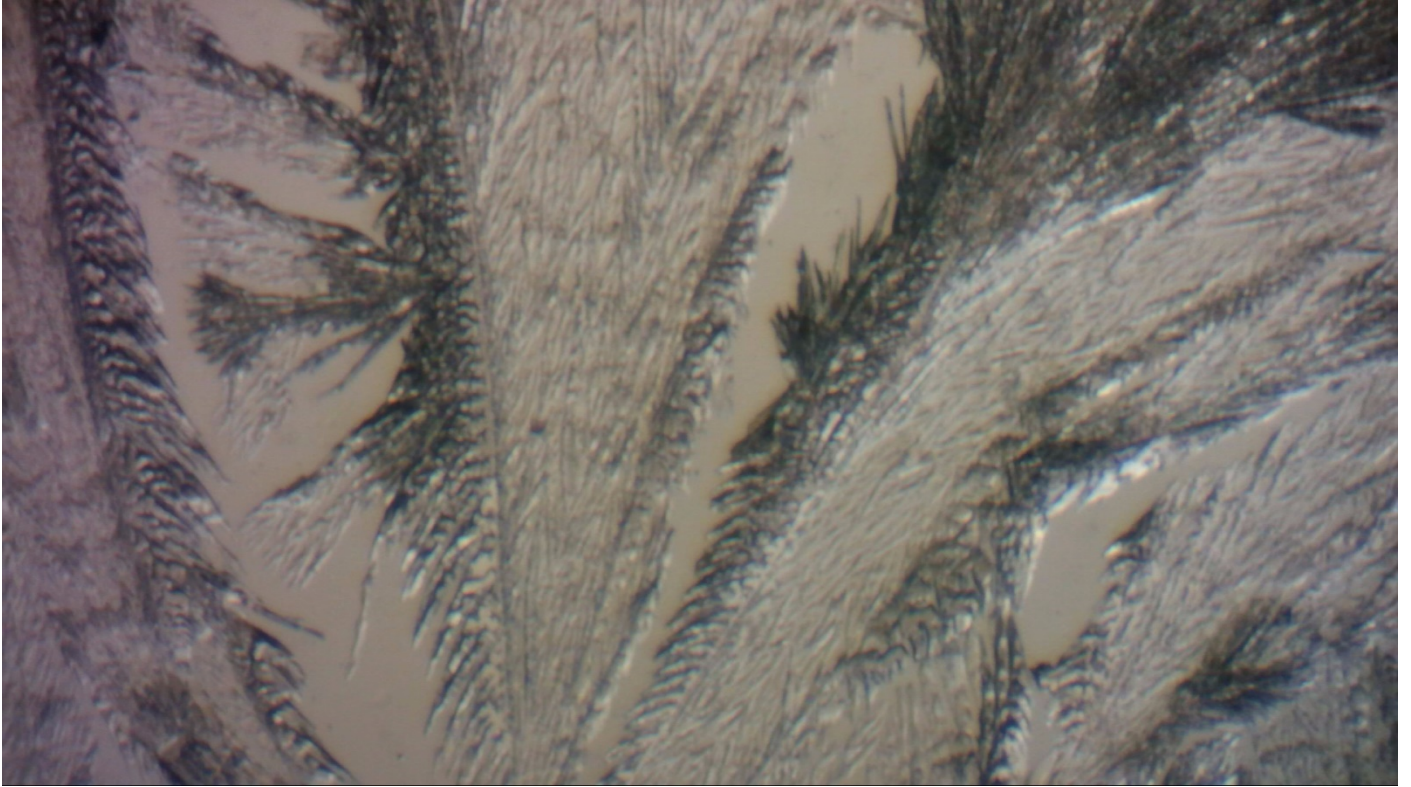
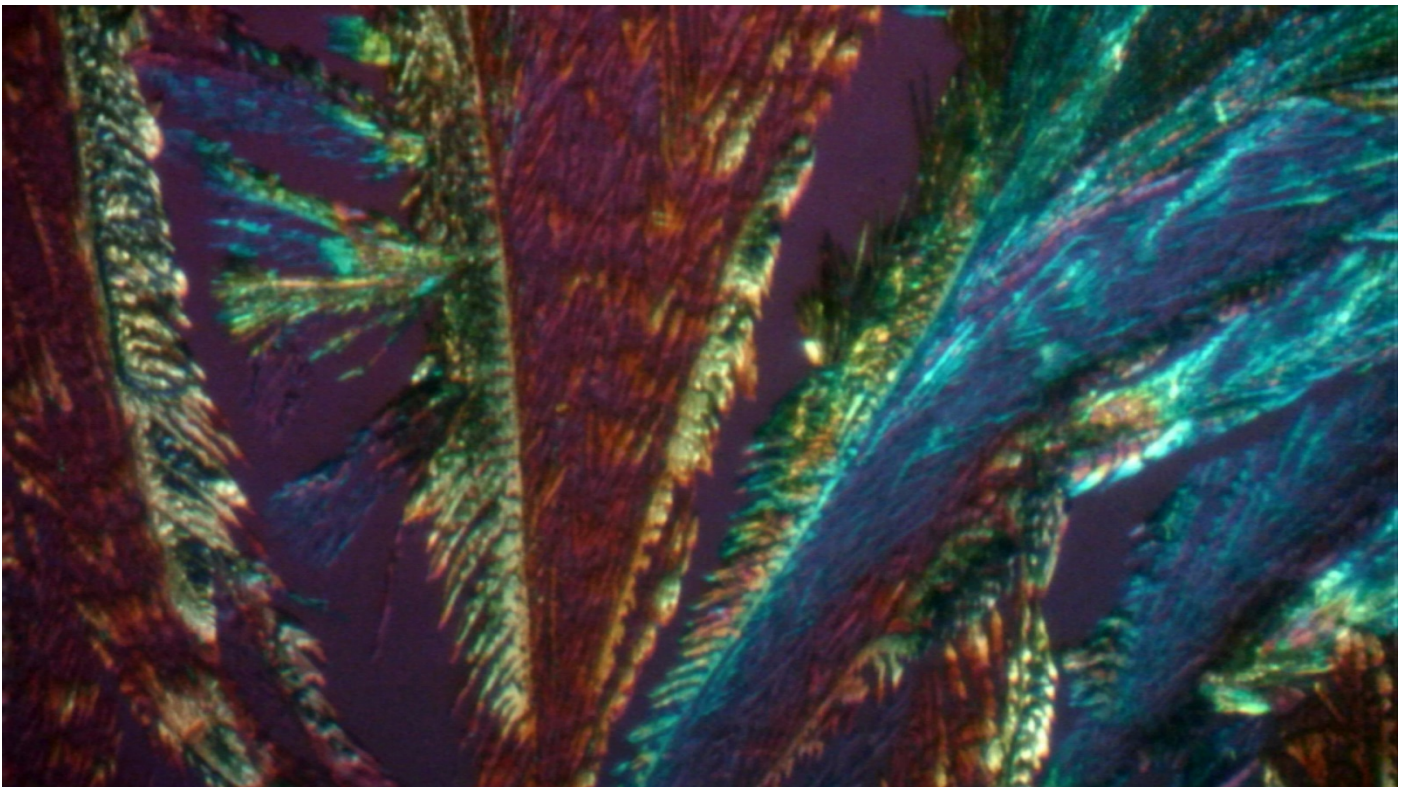


Image 2



Wilko Brand + caffeine

Image 1

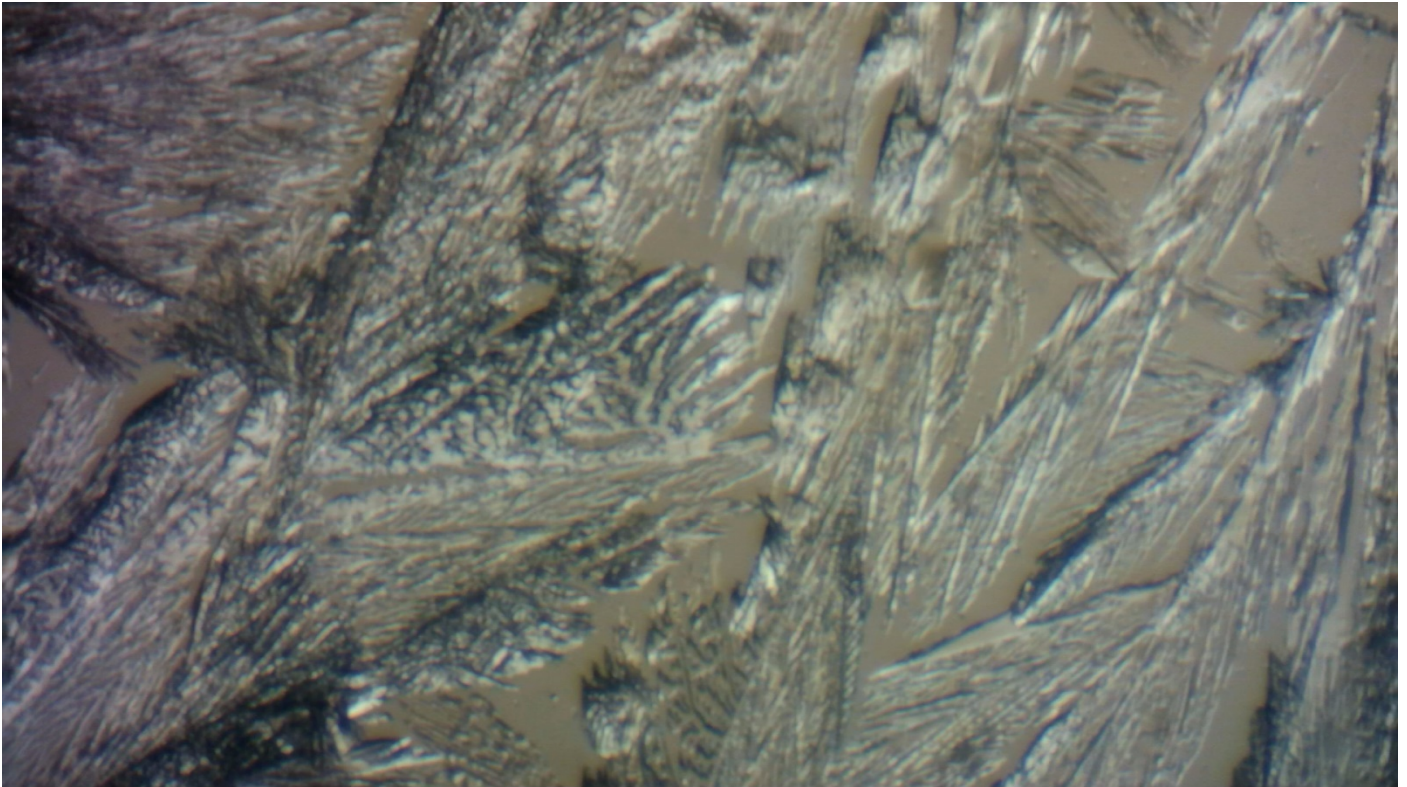
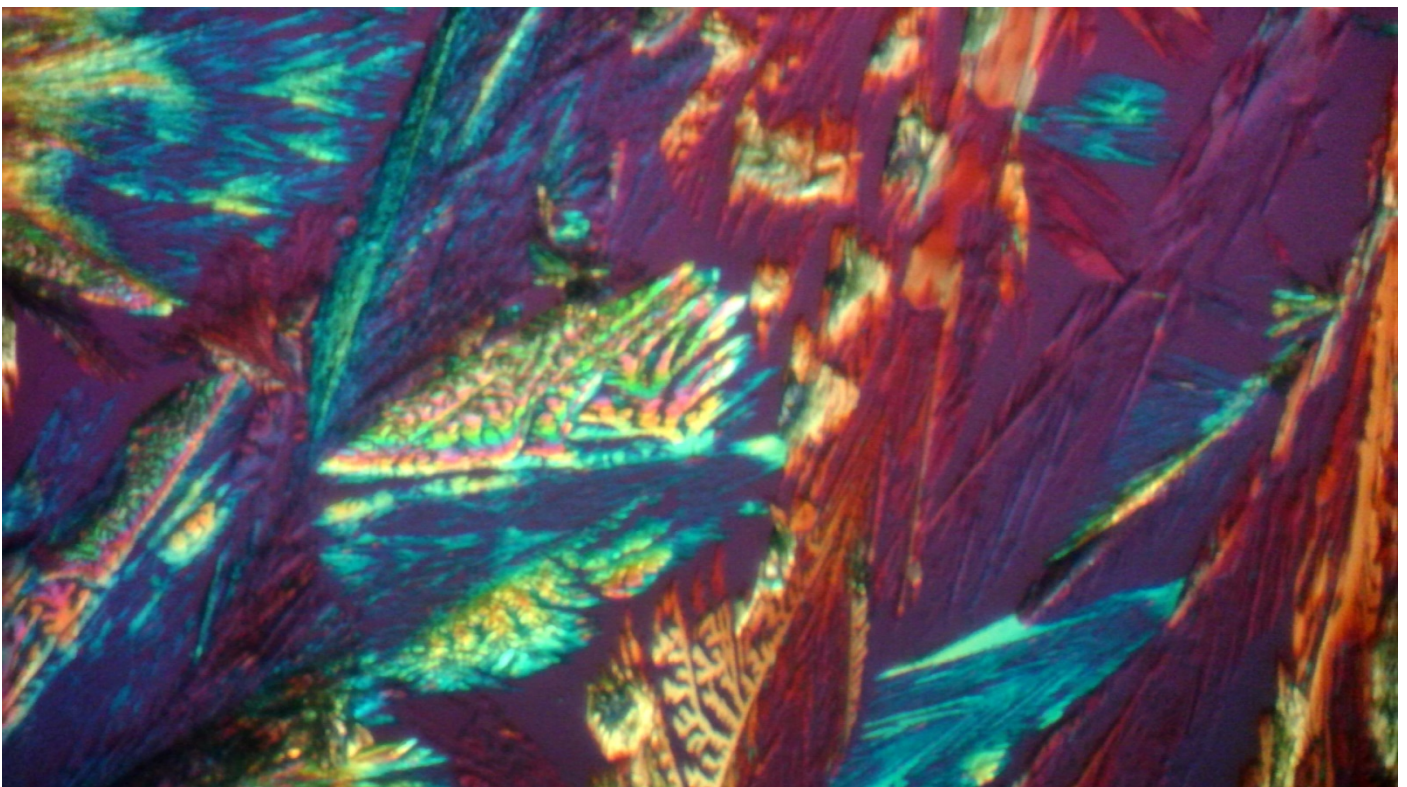


Image 2



Caffeine Control

Image 1

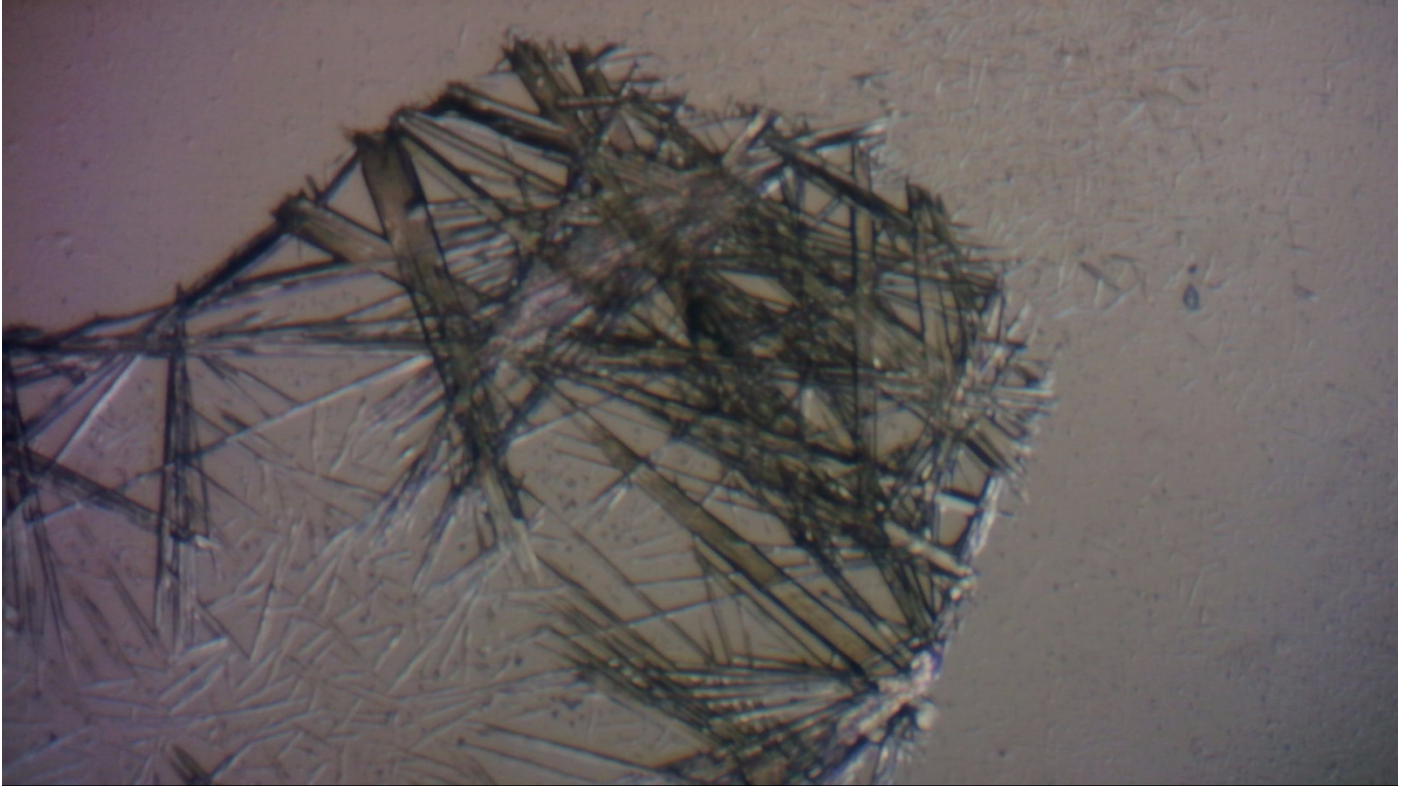
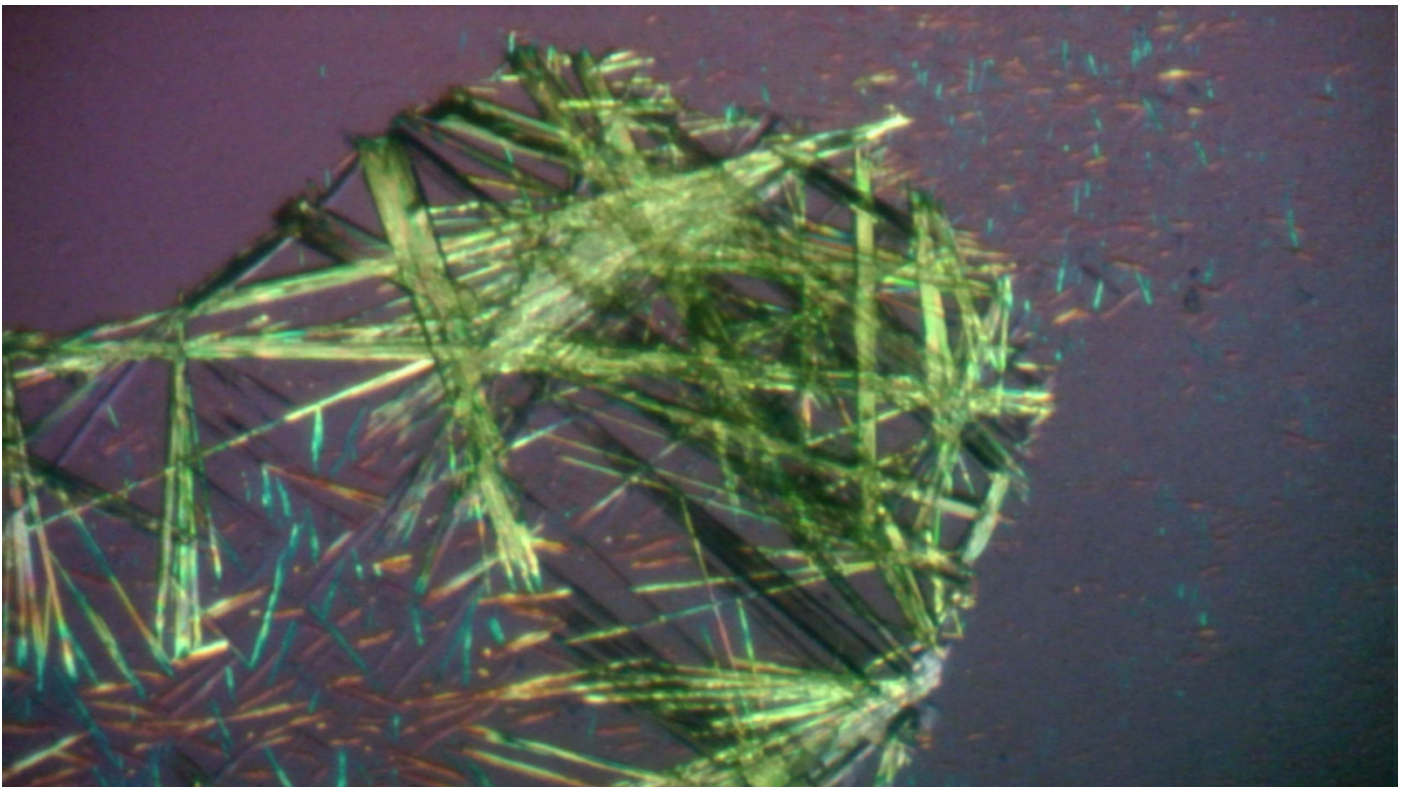


Image 2





### **Conclusions**

If the images captured during this experiment are compared with those captured in [Microscopical Exploration 11](#), it is evident that the addition of caffeine to the solutions of the five aspirin brands has a marked effect on the crystal structures obtained upon recrystallization from ethyl ethanoate solution. The crystal structure becomes more complex and feather-like due to the co-crystallization of the aspirin and caffeine, each of which appears to have more discrete crystals when re-crystallized separately. The caffeine control solution yielded crystals of a decidedly needle-like structure.

### **References**

CRC Handbook of Chemistry and Physics CD ROM 2005.

CRC Handbook of Chemistry and Physics 58<sup>th</sup> Edition 1977-1978.

Electronic Supplementary Material (ESI) for CrystEngComm. © The Royal Society of Chemistry 2015.

Email – [stewartr178 AT yahoo DOT co DOT uk](mailto:stewartr178@yahoo.co.uk) Published in the October 2021 issue of *Micscape* magazine.

[www.micscape.org](http://www.micscape.org)