

MICROSCOPICAL EXPLORATION ELEVEN

An investigation into the crystal structures of commercially available aspirin brands following 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 (ref i & ii), 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.

Aim

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

Applicability

The procedure is applicable to any commercially available aspirin tablets.

Equipment

Small glass jars with lids.

Filter funnel and filter paper

Mortar & pestle or two teaspoons (for crushing tablets)

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

Homemade 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.

Ethyl ethanoate (acetone free nail polish remover).

Procedure

Tablets containing a total of 900mg of aspirin were finely crushed between two teaspoons (I am at home in my man cave and the granite mortar & pestle in the kitchen are far too big!) and the resulting powder was well mixed with 20cm³ of ethyl ethanoate in a small glass jar 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/cm³. The solution was 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 illumination and sub-stage condenser only.

Image 2 X4 objective with transmitted illumination, polarizing filter immediately above the sub-stage condenser and analyser in 90° crossed configuration between the microscope objective turret and the digital eyepiece camera.

Observations

Anadin Original

Image 1

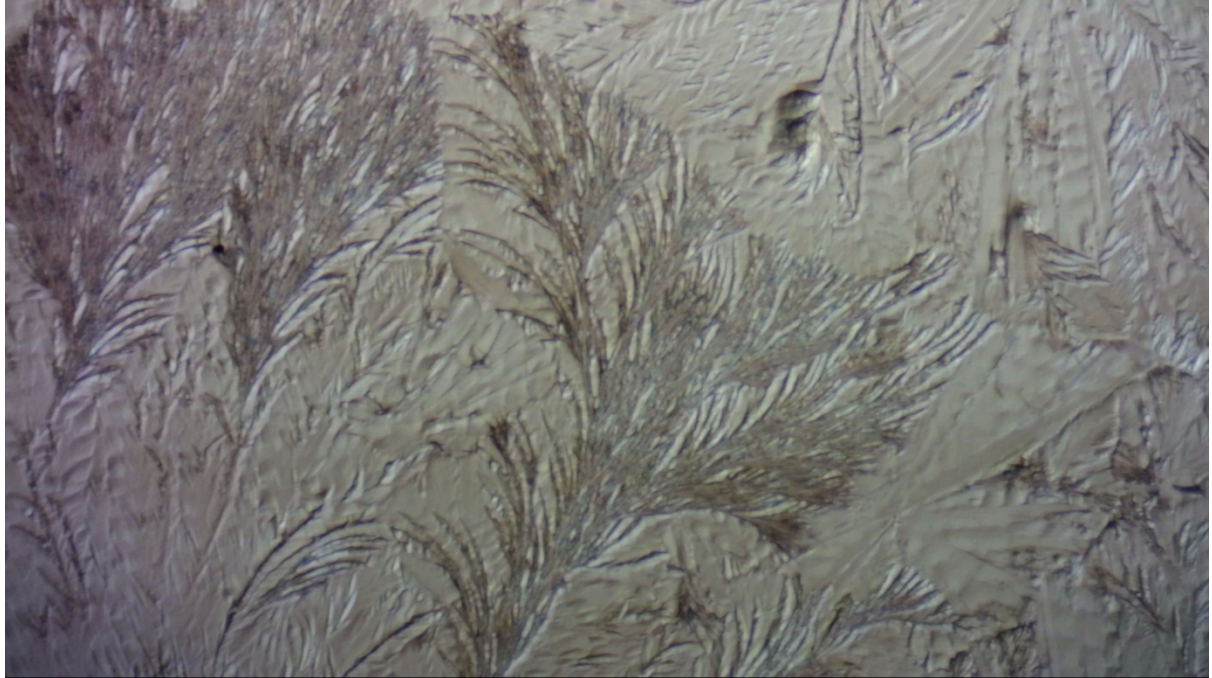
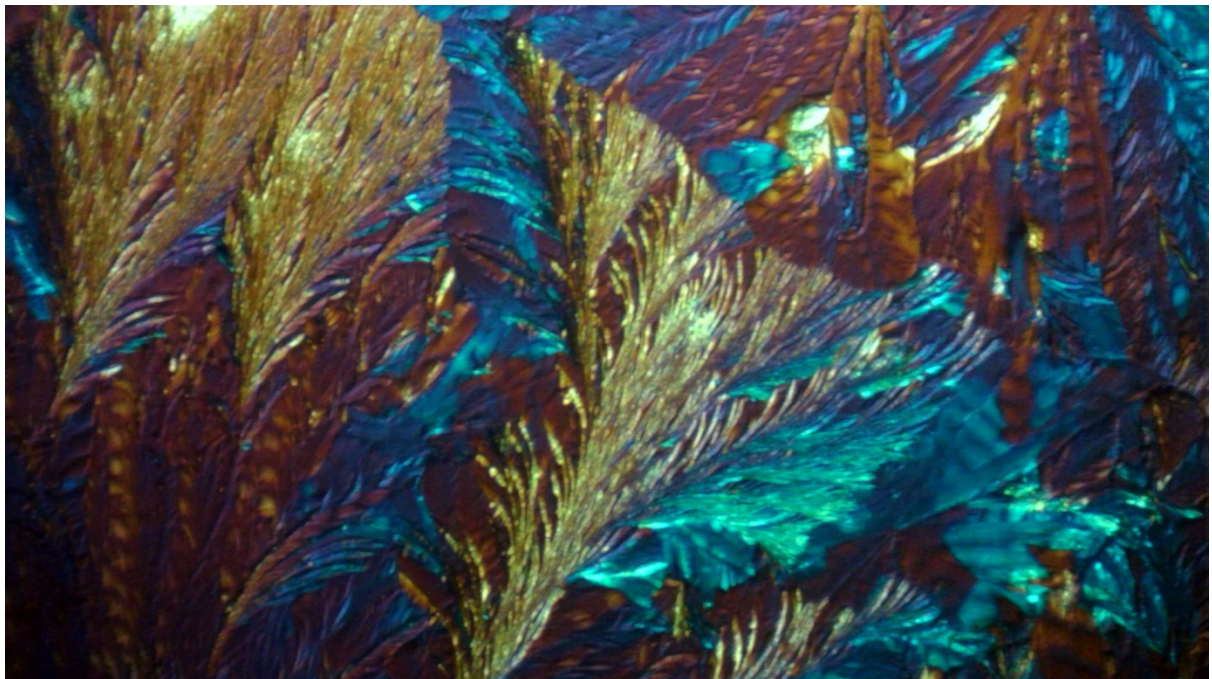


Image 2



Aspar Brand

Image 1

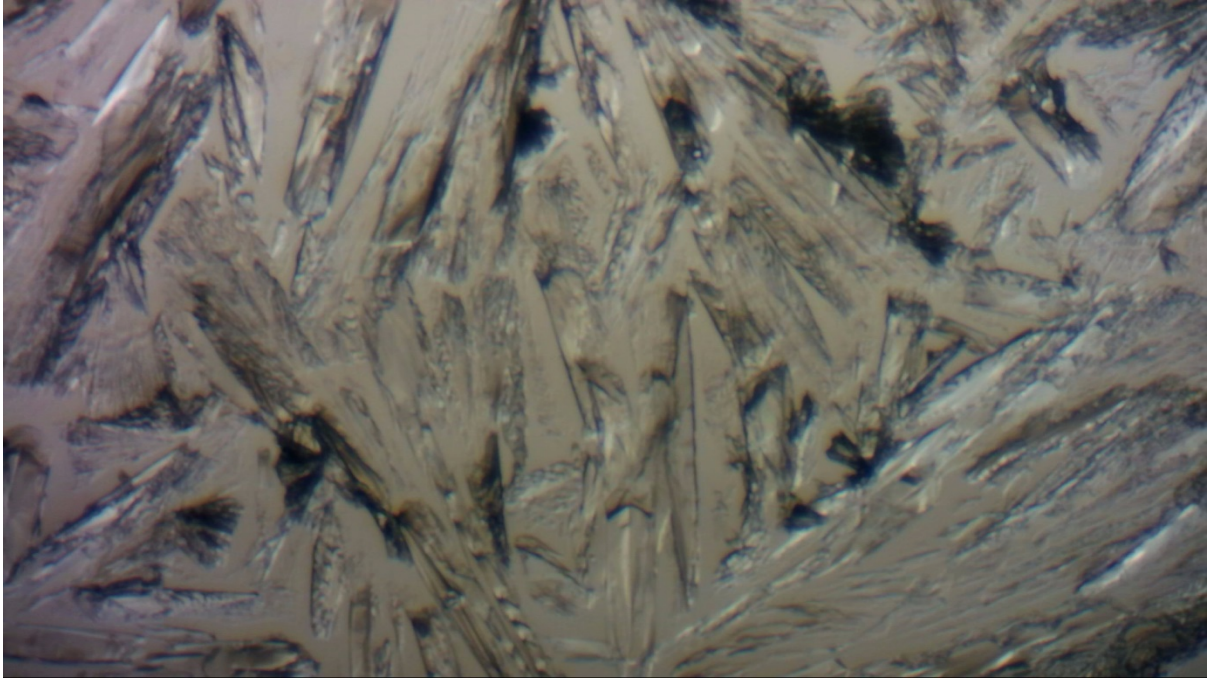
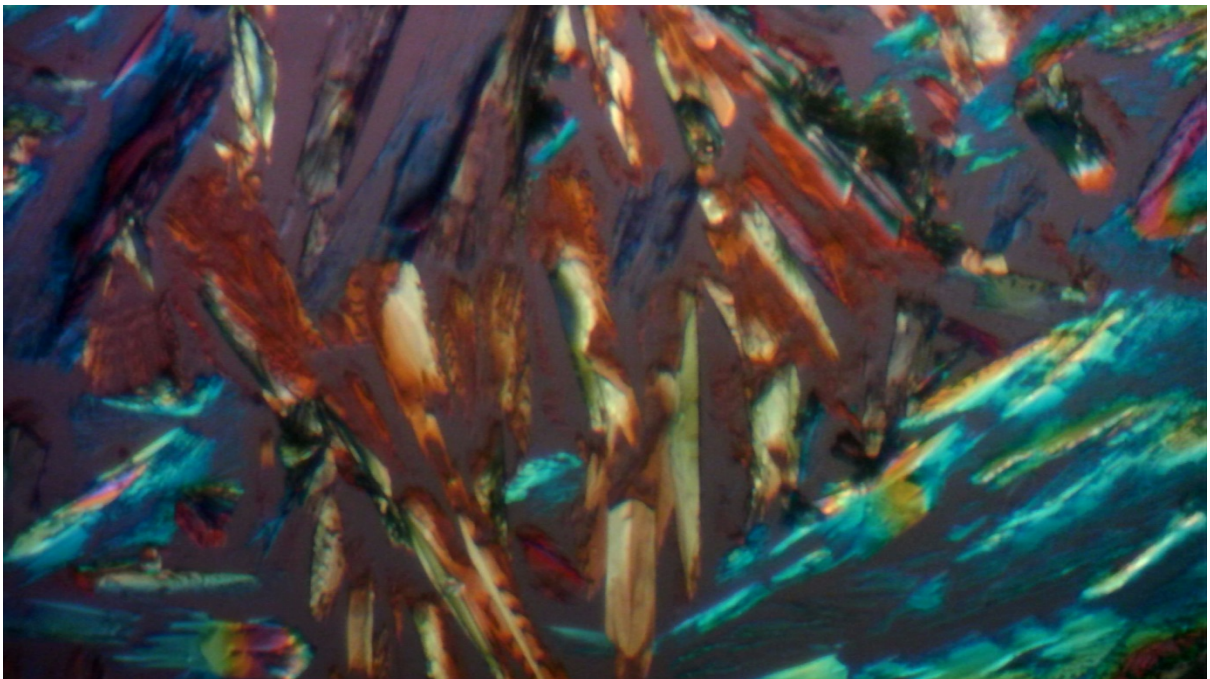


Image 2



Boots Brand

Image 1

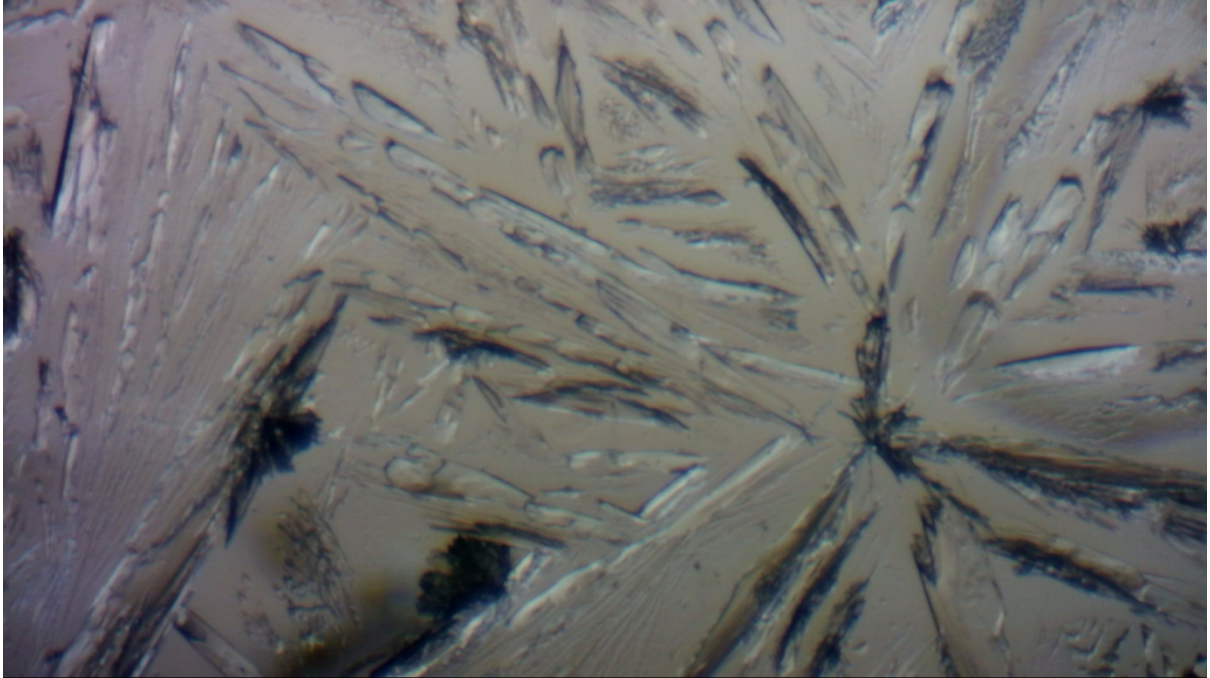
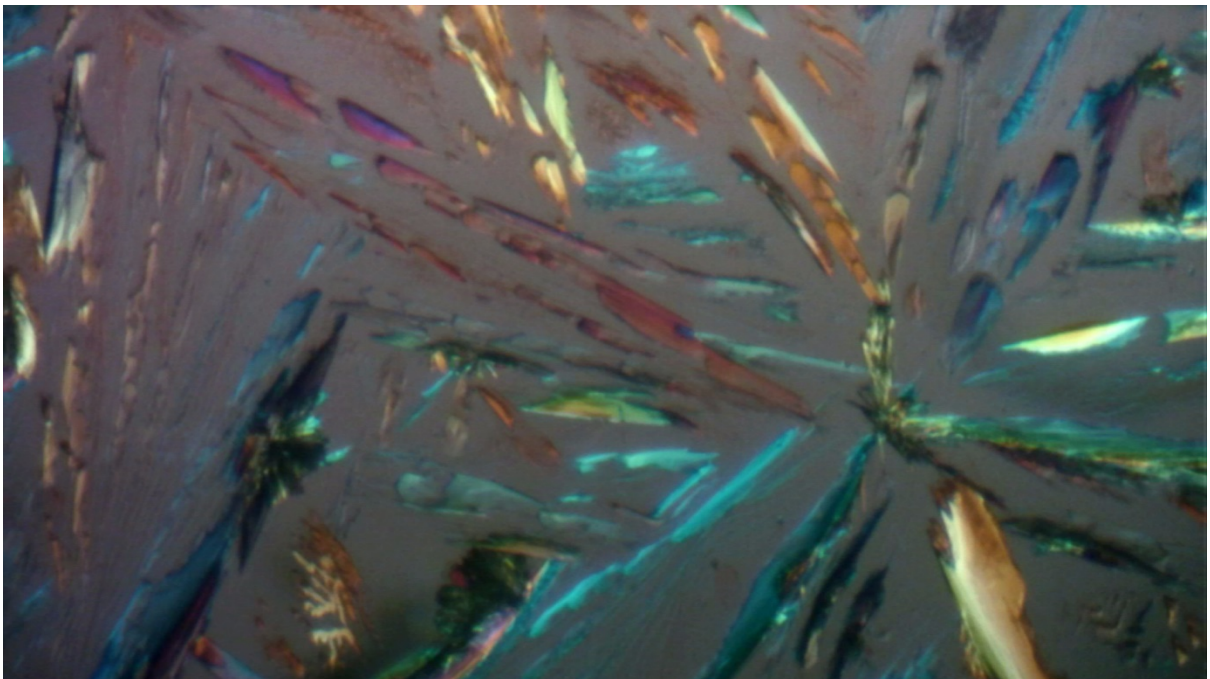


Image 2



Morrisons Brand

Image 1

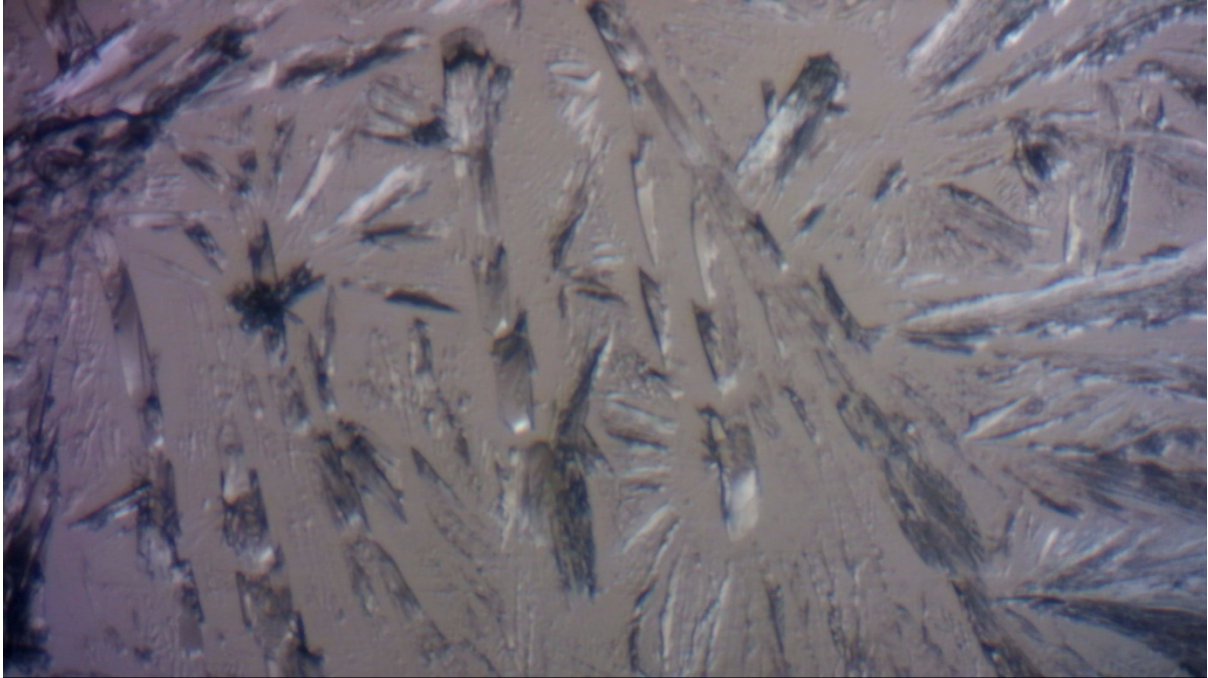
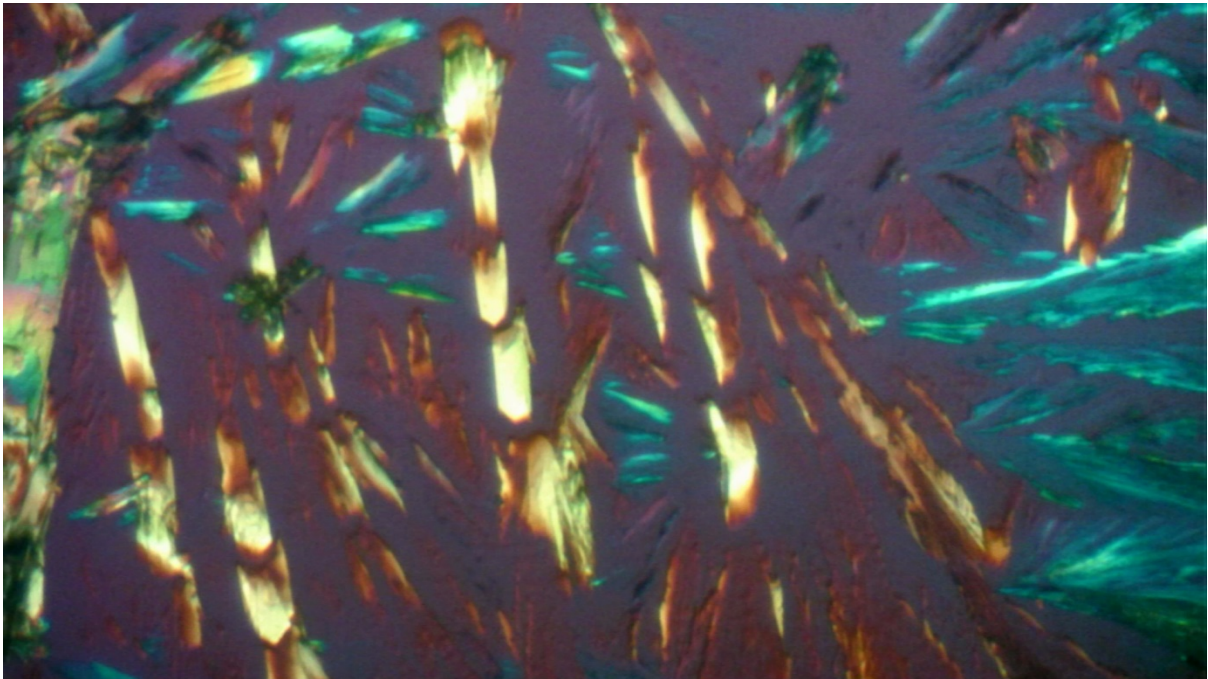


Image 2



Tesco Brand

Image 1

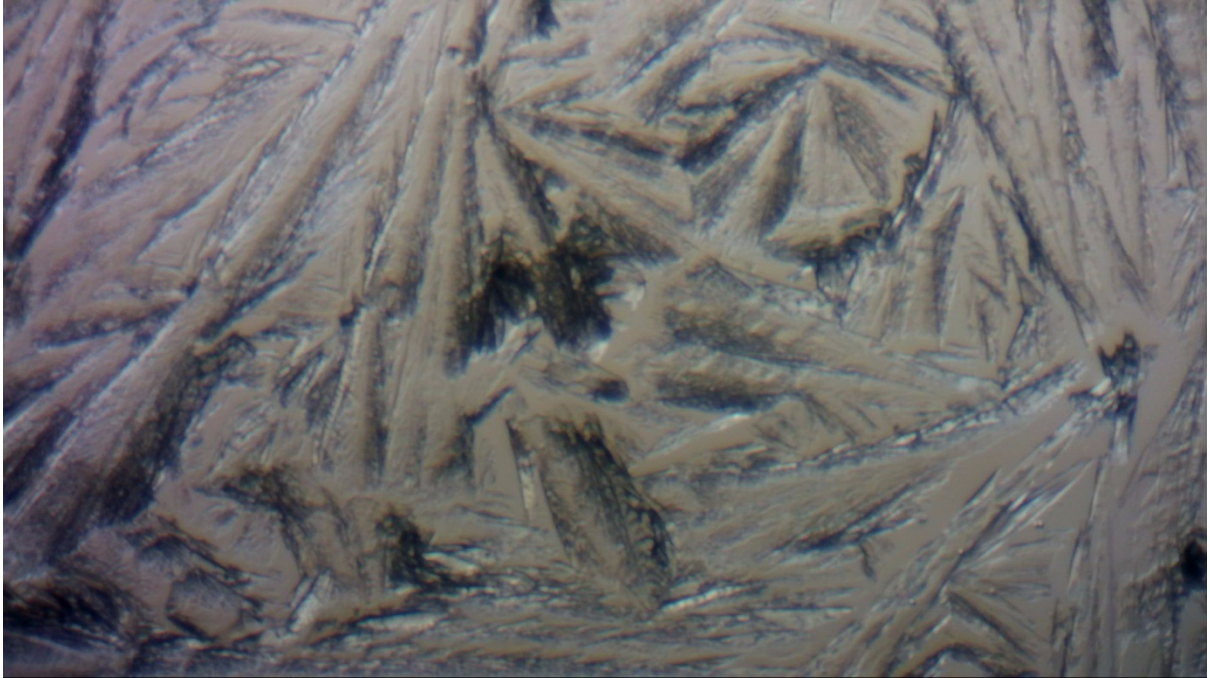
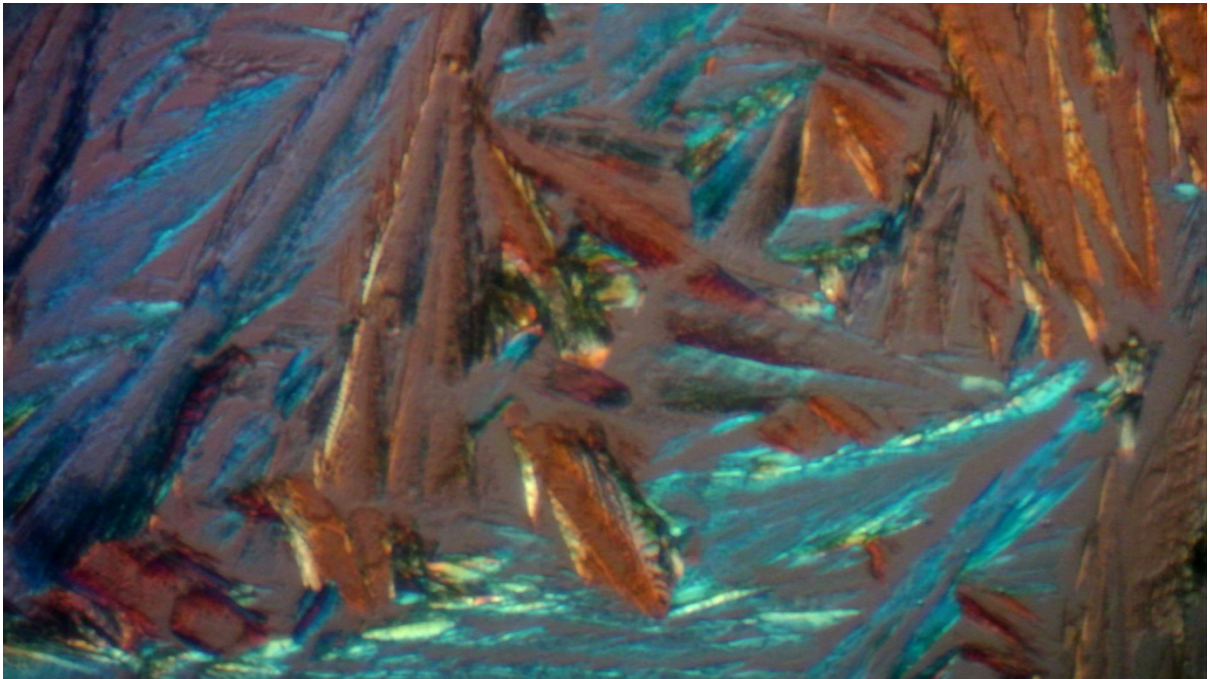


Image 2



Wilko Brand

Image 1

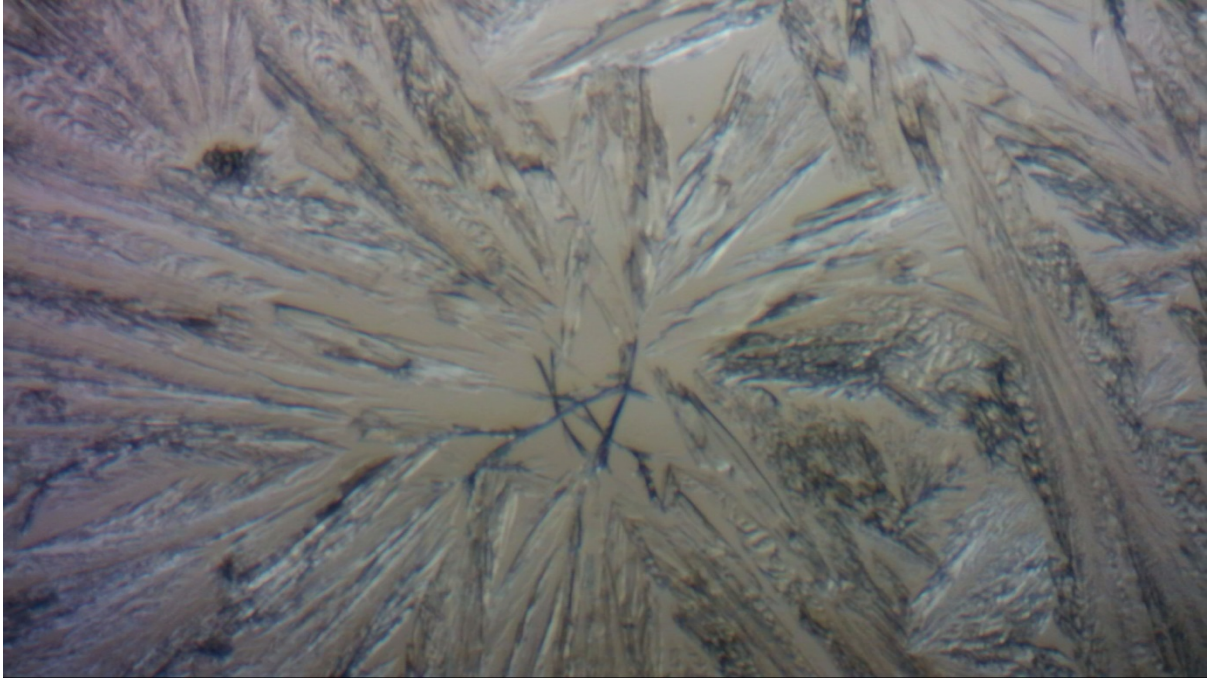
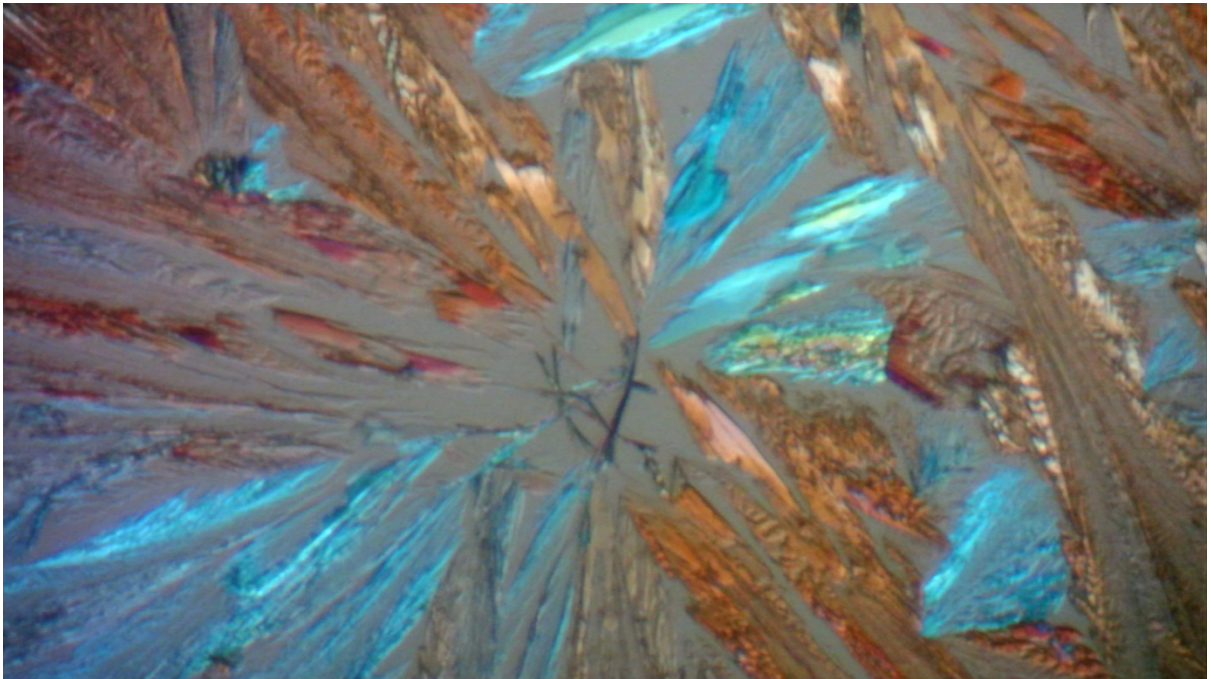


Image 2



Conclusions

The anisotropic nature and resulting birefringence of aspirin and caffeine crystals render the crystals ideal for observation under polarised light and result in the spectacular colours evident in image 2 for each brand. The obvious difference in the crystal structure of Anadin Original from those of the other brands is likely to be attributable to the inclusion of caffeine in addition to aspirin in the Anadin tablet formulation. It is suggested that this leads to the co-crystallization of the two compounds during the procedure used in the investigation and thence the observed crystal structure.

As well as acetylsalicylic acid, aspirin is sold both as sodium acetylsalicylate and also as calcium acetylsalicylate (ref iii) which may account for the differences between the other brands.

Further work needs to be undertaken to evaluate the effect of the inclusion of caffeine on the crystal structures of the brands which do not contain it: also the crystallographic differences between sodium and calcium acetylsalicylates should be investigated.

References

- i) CRC Handbook of Chemistry and Physics CD ROM 2005.
- ii) CRC Handbook of Chemistry and Physics 58th Edition 1977-1978.
- iii) Electronic Supplementary Material (ESI) for CrystEngComm. © The Royal Society of Chemistry 2015.

Interpret this simple experiment as you will, but as we say here in Cumbria:

‘Ave a go yersel’!

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