Sulphur crystals

1 INTRODUCTION

Sulphur is an element that can be found in nature in its pure form, typically in areas of volcanic activity, or in minerals as a sulphide or sulphate e.g. H_2S in sour natural gas, pyrite (FeS_2) and gypsum ($CaS0_4$)[1].

Figure 1: Sulphur mineral from the El Desierto mine, Potosi, Bolivia

The name sulphur is derived from the Sanskrit name 'Shulbary' which translates to 'enemy of copper' as sulphur destroys the metallic property of copper.

In the laboratory sulphur is purified by a recrystallization with carbon disulphide (CS_2). The crystals thus formed are polluted with the solvents used such as H_2S and SO_2. As these are chemicals that are preferably not used in a home laboratory other solvents are to be used.
In the past I used paraffinic oil (lamp oil) to recrystallize sulphur[2]. However, I found the use of lamp oil to be less suitable for preparing micro crystals for microscopic investigations. Recently, I read an article where toluene and xylene were used as a solvent[3]. At home, most people will not have these chemicals available in their pure form but I remembered that 'thinner' contains similar chemicals (aromatics) i.e. the thinner used in this experiment contains xylene, ethylbenzene and n-butanol as main components. Note that the composition of thinner can vary as it is vendor dependent.

The aim of this experiment is to investigate whether conducting a recrystallization with thinner is a relatively fast method for generating microcrystals suitable for microscopic investigations.

2 MATERIALS

- Paint thinner
- Sulphur powder
- Heating plate
- Test tube with stopper
- Scale
- Polarisation microscope (Euromex ML2000)
- Microscope camera (Lucky Zoom USB5M)
- Filter paper

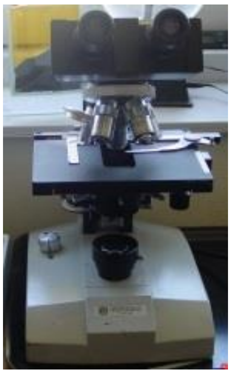

Figure 2: Euromex ML2000 microscope

- Funnel
- Glass slides
- Cover glasses
- Bottle with screw cap
- Stirring rod
- Dissecting Needle
- Spatula
- Graduated cylinder

Figure 3: Lucky Zoom 5Mp camera

3 METHOD

- With the graduated cylinder pour about 20 ml thinner into the bottle.
- Add about 1 g sulphur powder and mix well.
- Place the bottle with a loose cap on the heating plate and switch the heating plate on.
- Mix regularly
- Once the solution has reached a temperature of about 100 °C filtrate a part of it off in the test tube.
- With the aid of the stirring rod put a drop of the hot solution on the glass slide.
- Let de solvent evaporate.
- Place a cover glass
- Observe under the polarisation microscope.

Figure 4: Heating the mixture

Thinner has a rather strong smell. Conduct this experiment in a well-ventilated room.

4 RESULTS

As can be seen in Figure 5, small needle like crystals form almost immediately in the test tube during filtration.

The pictures below were taken with the Euromex ML2000 polarization microscope and the Lucky Zoom 5Mp camera. The captions refer to the objective lens used when taking the picture.

Figure 5: Crystallization in the test tube

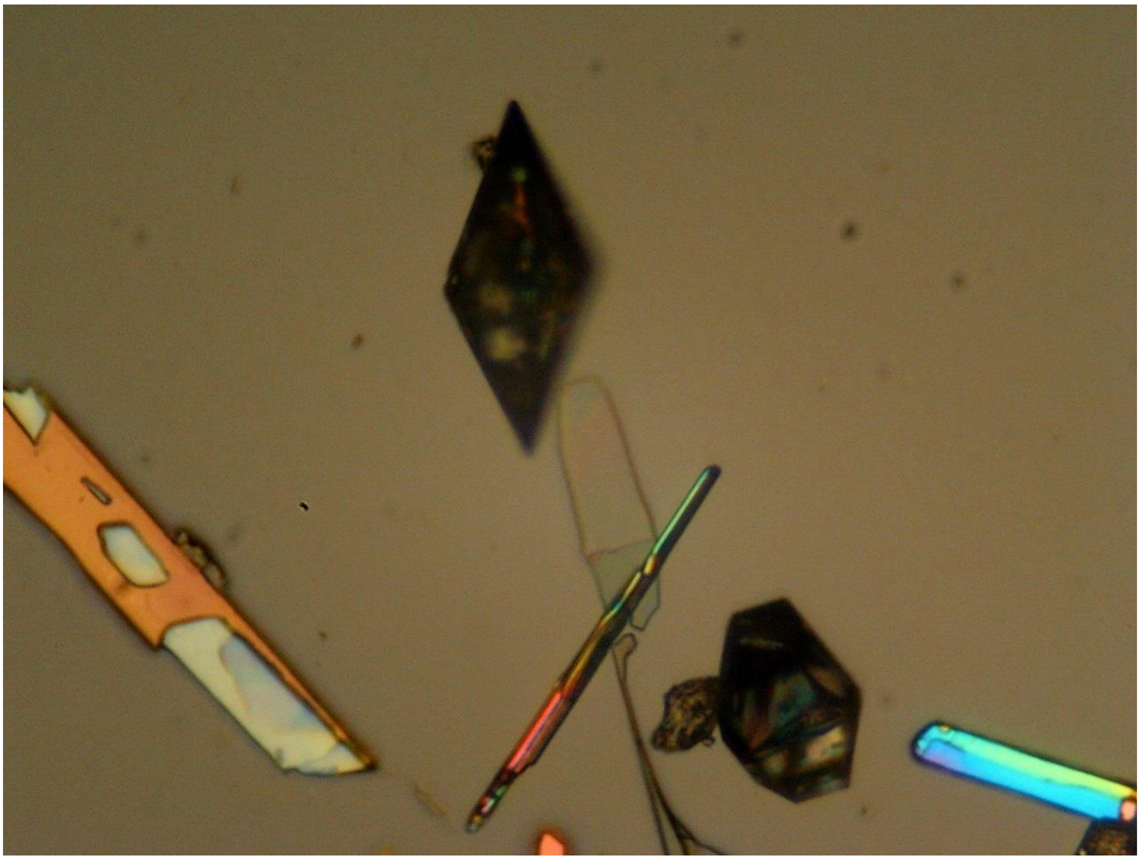

Figure 6: Sulphur crystals, Euromex Phase DM 20X / 0.40 DIN

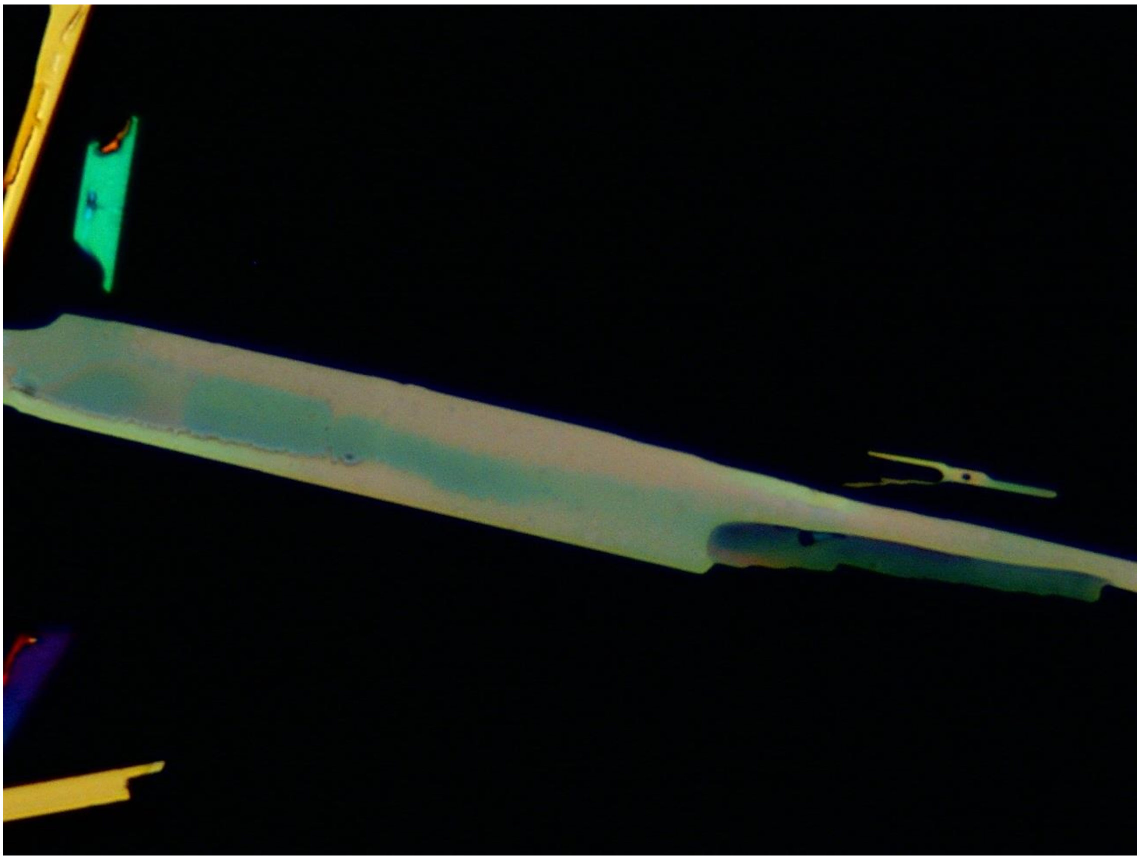

Figure 7: Sulphur crystals, Euromex Phase DM 20X / 0.40 DIN

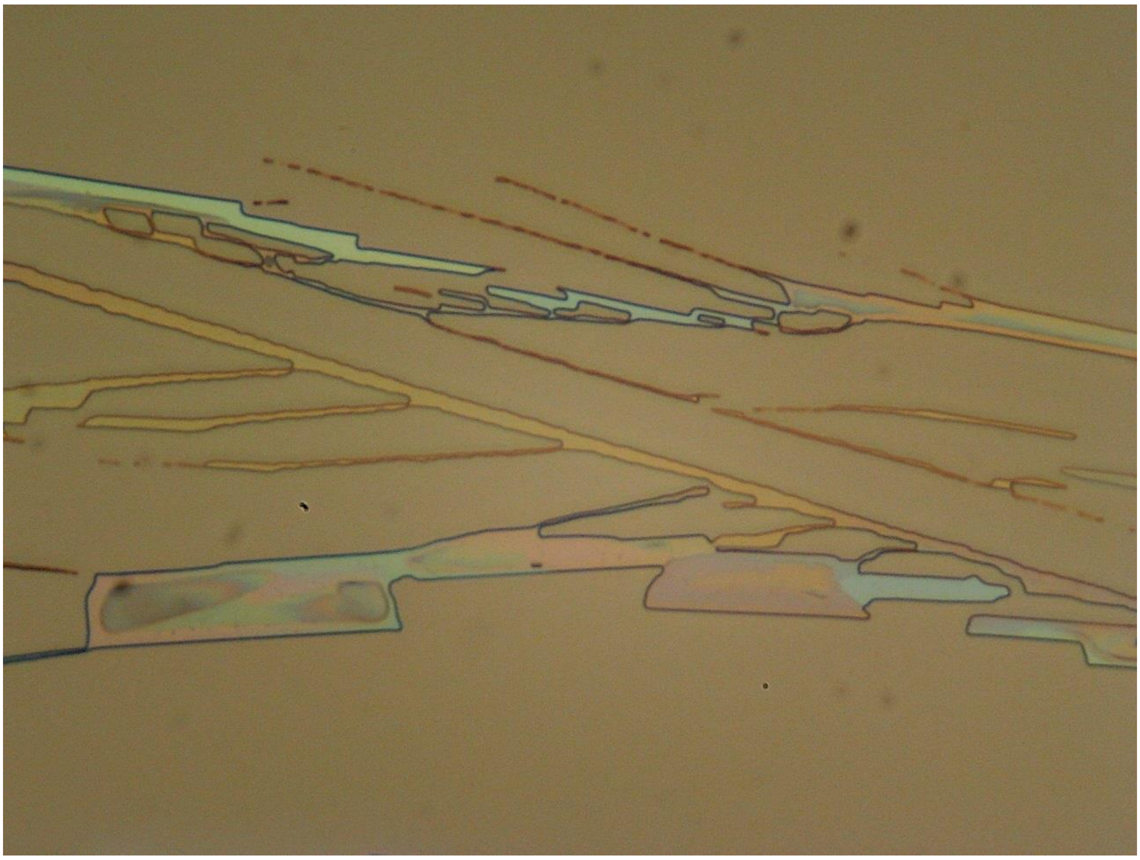

Figure 8: Sulphur crystals, Euromex Phase DM 20X / 0.40 DIN

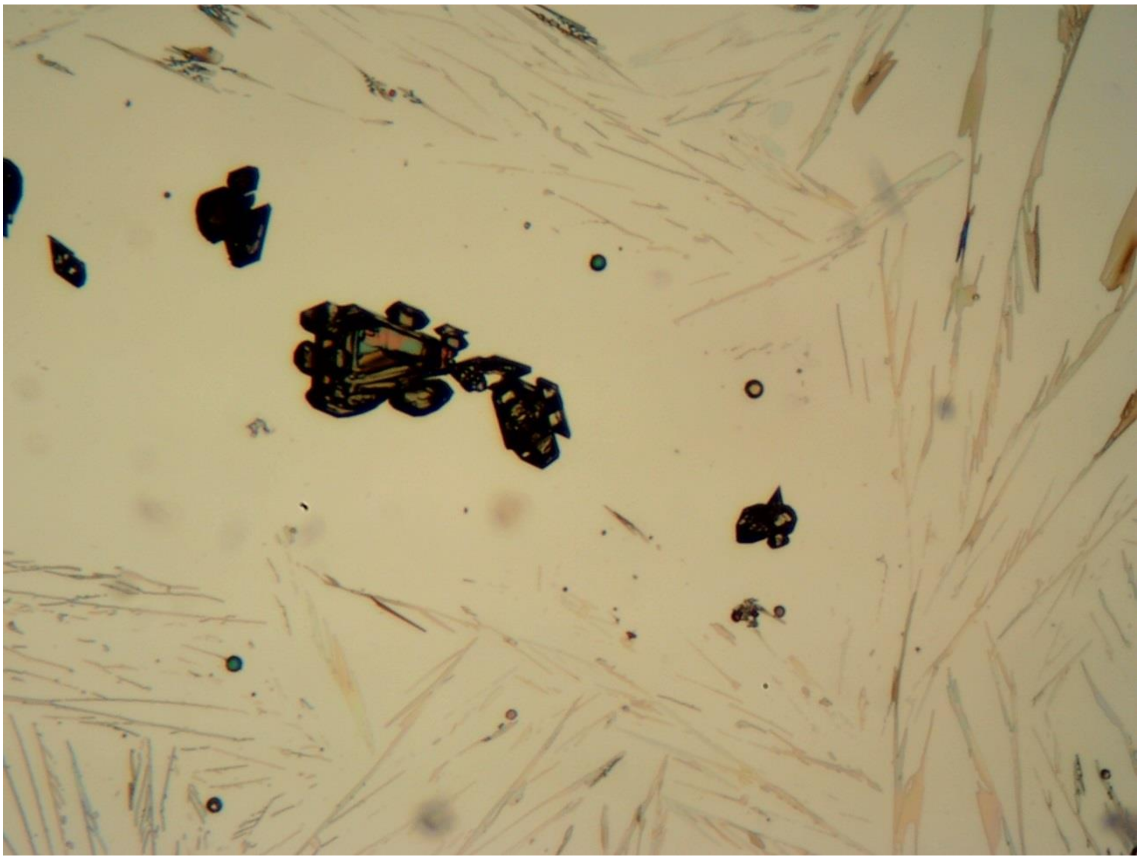

Figure 9: Sulphur crystals, Euromex S. Flat Field 10 0.25 – 160 0.17 DIN

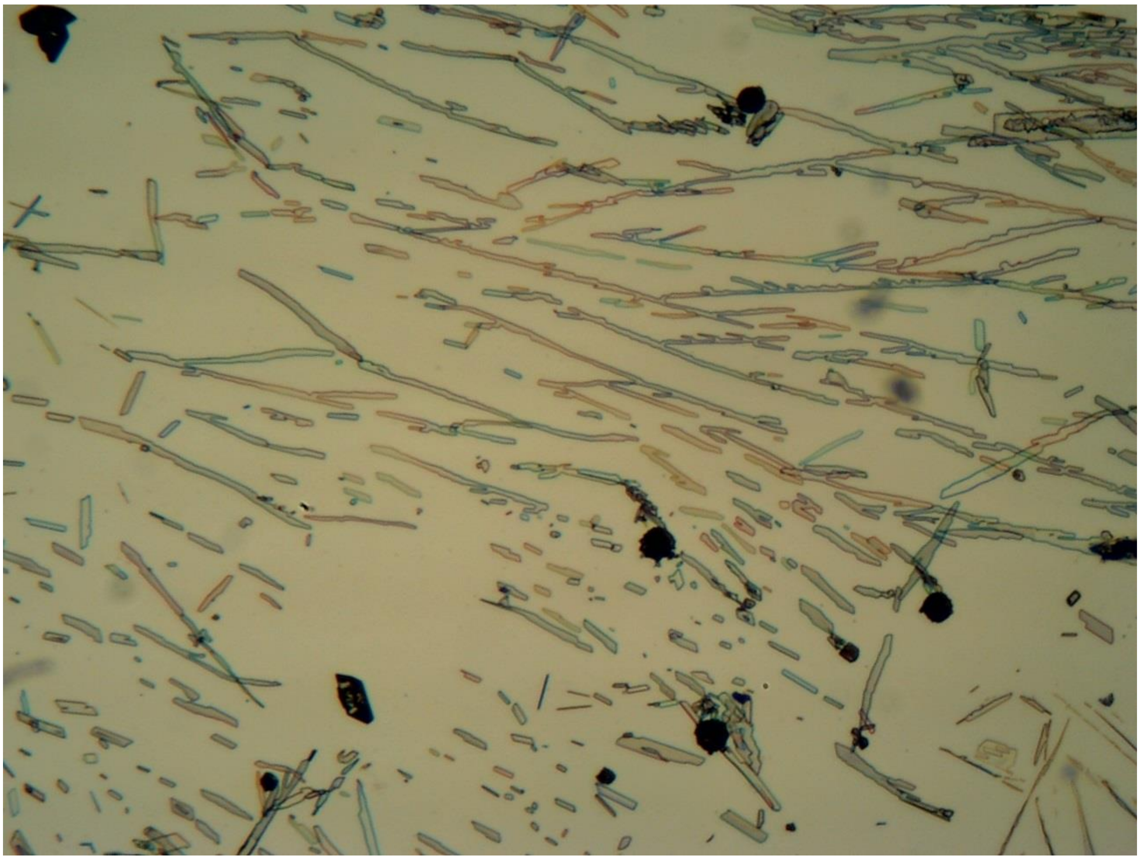

Figure 10: Sulphur crystals¸ Meiji S. Flat Field 4/0.10 – 160 0.17 DIN

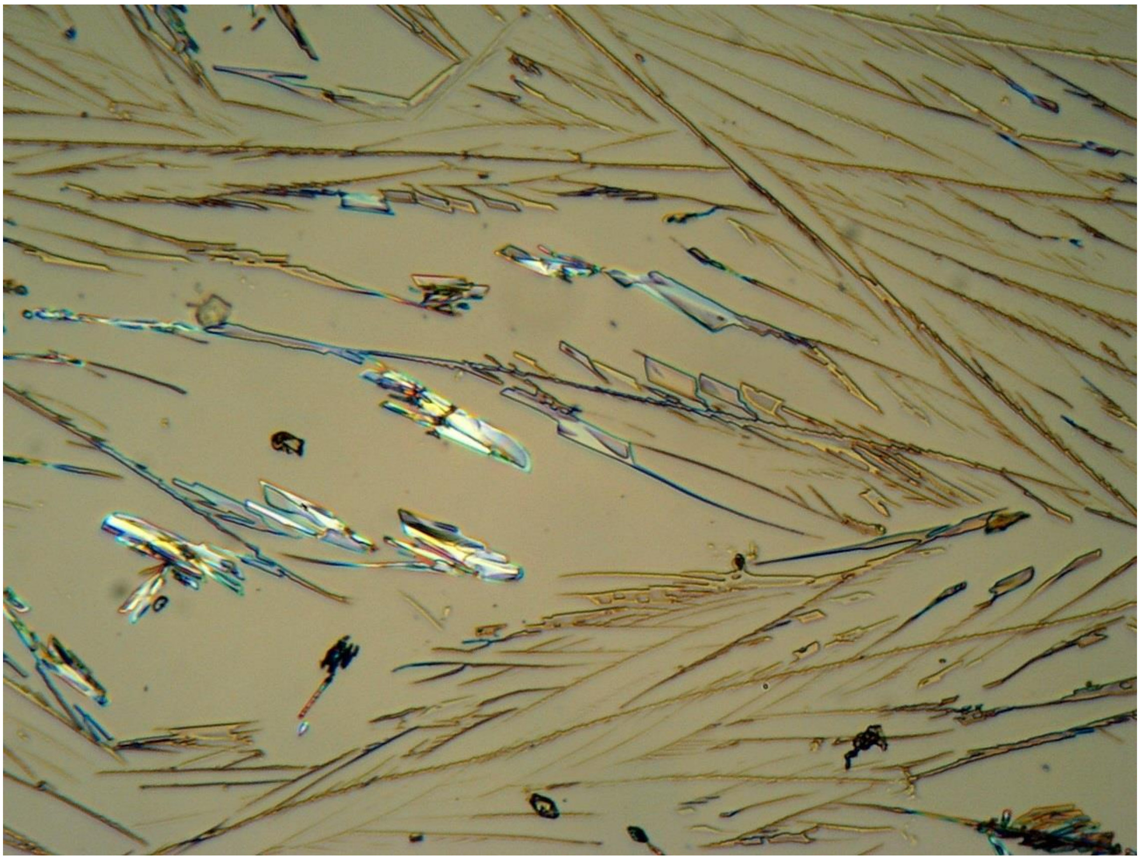

Figure 11: Sulphur crystals, Meiji S. Flat Field 4/0.10 – 160 0.17 DIN

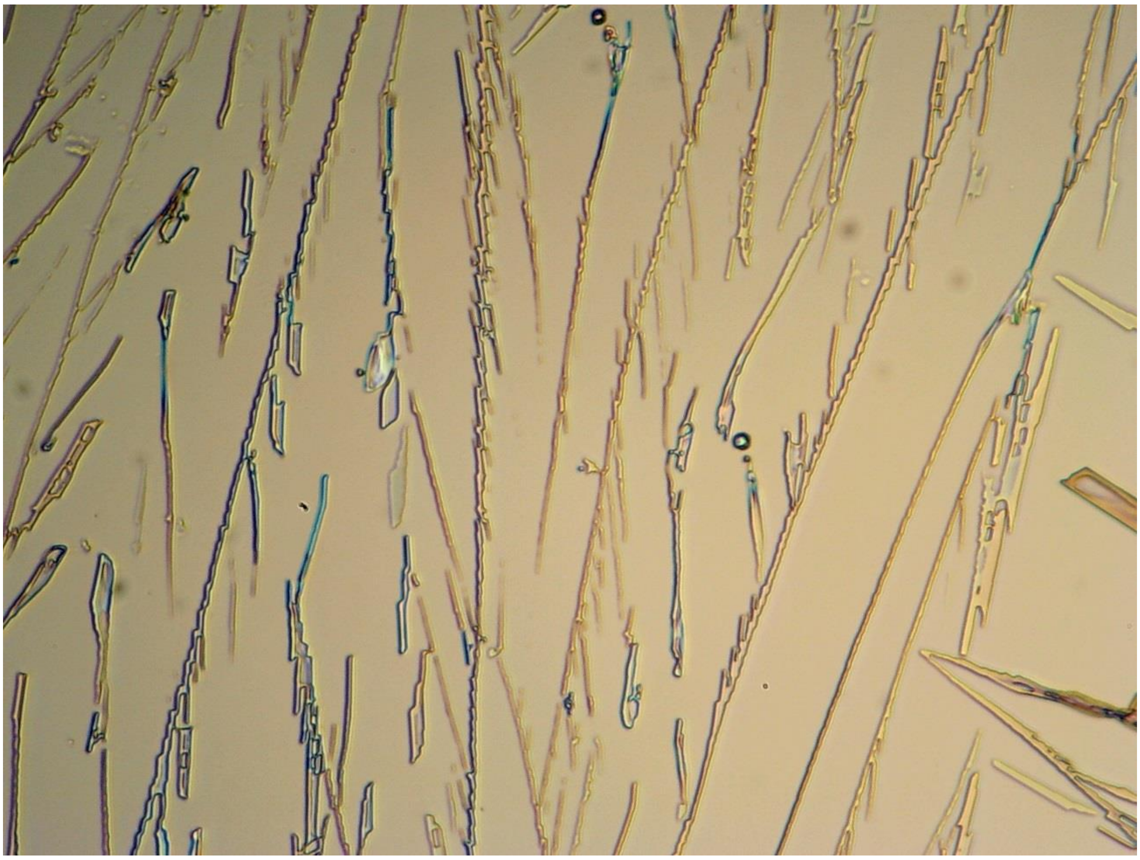

Figure 12: Sulphur crystals, Meiji S. Flat Field 4/0.10 – 160 0.17 DIN

Figure 13: Sulphur crystals, Meiji S. Flat Field 4/0.10 – 160 0.17 DIN

5 DISCUSSION

The typical sulphur molecule consists of an eight ring in zig zag form.

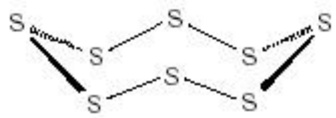

Figure 14: S_8- ring

Sulphur is found in two modifications (both S_8). Below 95.5 °C rhombic and above this temperature monoclinic.At 119.5 °C the monoclinic sulphur will melt producing a light yellow liquid which is still composed of eight rings. The property that there are different structural forms of the same element is called allotropy. As the rhombic sulphur is stable at temperatures below 95.5°C, rhombic sulphur is the allotropic form normally found in nature. Monoclinic sulphur is stable between 95.5°C up to 120°C. If monoclinic sulphur is cooled it transforms into rhombic sulphur at a temperature below 95.5°C. When molten sulphur is cooled, it solidifies into monoclinic sulphur. At room temperature all forms of sulphur are gradually transformed into rhombic sulphur.

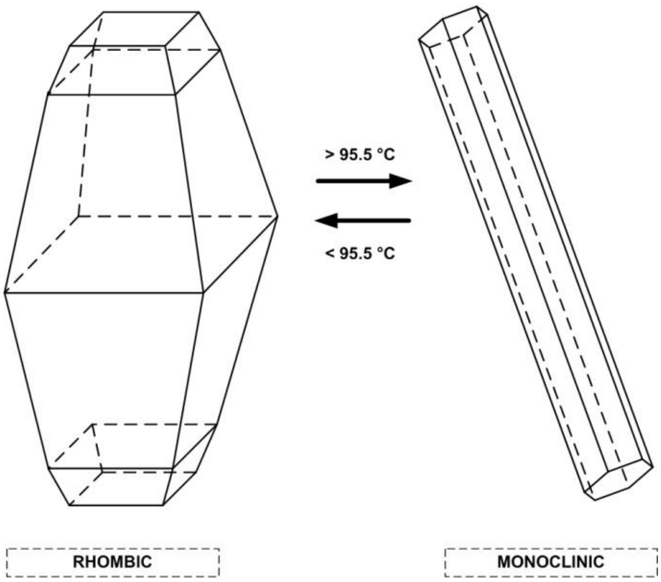

Figure 15: Rhombic and monoclinic sulphur

This information helps explaining the visual observations made during the experiment. The crystallization that can be observed in the test tube is the formation of needle like crystals which suggests the formation of monoclinic sulphur. This observation appears to be confirmed by the microscopic images, whereby the majority of the pictures show needle like crystals. Only in a few pictures small rhombic like crystals can be observed (Figure 6, Figure 9 and Figure 10).

These results appear to be counterintuitive when taken the data presented above into account. However, the thinner and sulphur was heated to a temperature above 95.5 °C where monoclinic crystals can form. Subsequently the solution was filtrated and although the solution cools rapidly, monoclinic crystals can form above 95.5 °C. Furthermore, the conversion from monoclinic to rhombic sulphur will require some time and one can therefore assume that due to the rapid cooling the crystals are predominantly "frozen" into their monoclinic form.

This assumption appears to be confirmed when studying a recipe for preparing rhombic sulphur crystals[4]. Powdered sulphur was dissolved in carbon disulphide and recrystallization occurred at room temperature i.e. the solution was not heated.

In that respect it will be interesting to store these slides for a prolonged period of time and check whether the conversion from monoclinic to rhombic sulphur will continue on the slide itself.

6 LITERATURE

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4. Charles H. Stone; "Preparation of rhombic sulfur crystals"; *Journal of Chemical Education*; **1932** 5 9; p. 941.

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