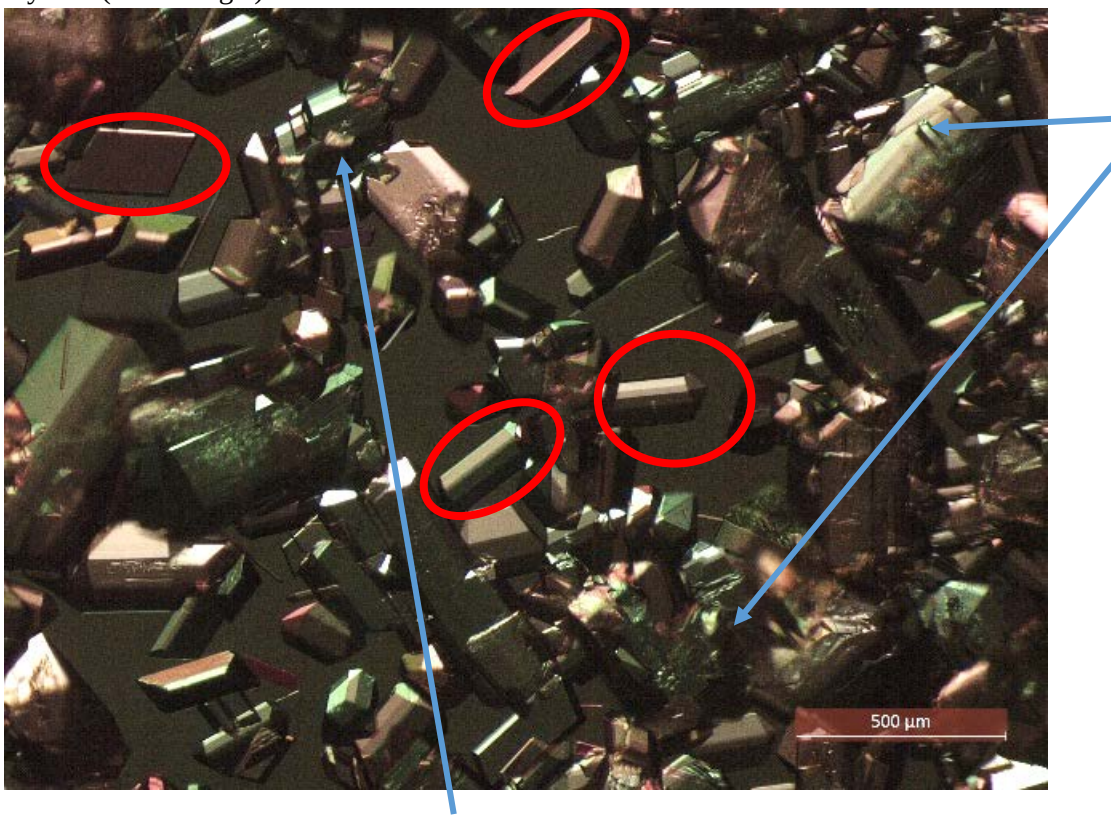
Choosing a Crystal

In order to get good diffraction from a crystal you must choose a good quality, single crystal to mount onto the diffractometer. A good crystal:

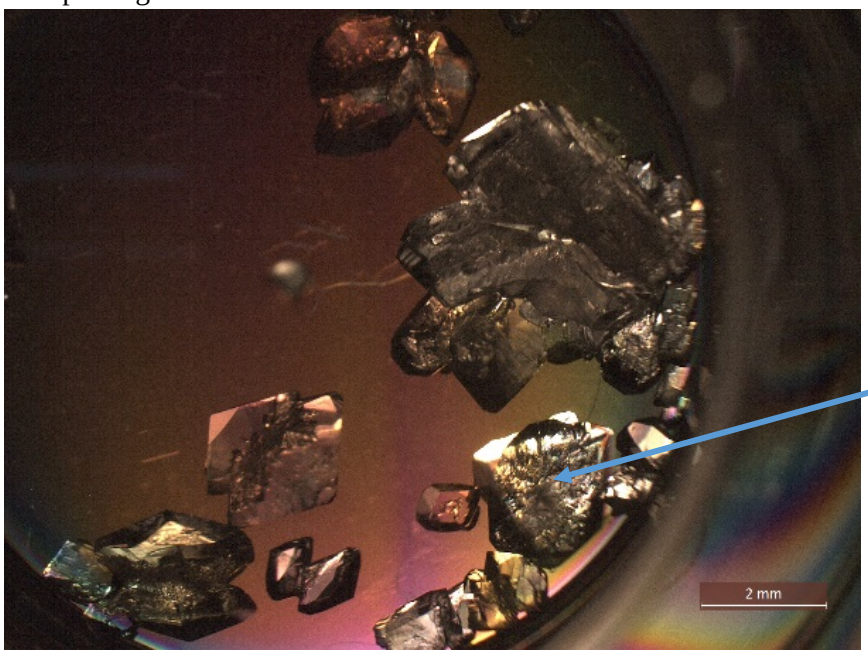
- Has sharp, well-defined crystal faces free from defects
- Has a regular geometry (if possible)
- Is clear (rather than opaque)
- Has an even colour (no rainbow/multi-coloured faces under cross polars)
- And is SINGLE, ie no passengers, intergrowths or shared growth planes

Using a cross polarizer, most crystals will extinguish light as the polarizer is rotated through 90° (although there are exceptions for very high symmetry cells). This can also help to determine if a crystal is single or twinned - more than one attached together or growth in separate directions from a common plane. The crystal should ideally be smaller than the size of the x-ray beam, so that it is entirely bathed in the rays produced and the amount of irradiated matter remains constant throughout the experiment, irrespective of the orientation of the crystal. If the crystals are stuck together or too large, a scalpel or razor blade can be used to prize them apart or reduce the size to get the best possible crystal for mounting.

Example of a sample of crystals including nice single crystals with clearly defined edges which could be used for diffraction (circled in red) and also some marked or defected crystals and clusters of crystals (ie not single) not as suitable for diffraction.



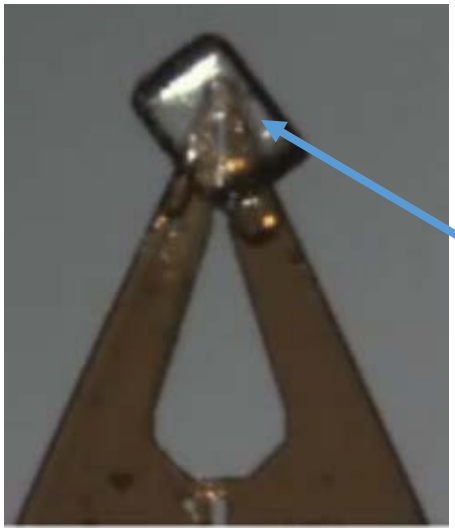
These crystals below are poorer quality as the surfaces are marked or defected and the crystals are clumped together or twinned.



Clear marking or defects on the surfaces

Note the size of the crystal (dimensions) as these will be entered into the software when collecting the diffraction pattern. Ideal crystals are approximately 0.1-0.3mm in size, ideally in all three dimensions. You should place a little oil on a slide then, with a spatula, remove some crystals from the vial/container and place into the oil. (Ensure you do not have too many crystals on the slide as this can make it much harder to manipulate your selected crystal). The oil helps coat and protect the crystals if they are air, moisture or solvent-loss sensitive. It also helps to separate and clean them. Under the microscope assess the crystals you have, and if you feel there may be better ones still in the sample then you can take more onto the slide. Use the tools to manipulate the crystals and try to get your selected crystal on its own, away from others. If it is too large or stuck to another, use the blade to reduce the size or to separate it from its twin then move it out of the oil as much as possible as too much oil will affect the diffraction.

You will then need to mount the crystal onto a 'pip'. Take care these are very fragile. You should aim to get the crystal at the top point of the diamond over the loop and sticking out in a fairly central position if possible. This is fiddly and takes time so have patience.



Crystal at top of pip