Variable Temperature Data Collections with CrysAlisPro

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The variable temperature data collection features in CrysAlisPro coupled with AutoChem allow rapid data acquisition, data reduction and structure solution.

- The data in this example were collected with a SuperNova molybdenum micro-focus source with Atlas CCD detector coupled with the Cryojet.
- The software also functions with the other Agilent XRD products; Xcalibur, Gemini, and Nova.
- The variable temperature software is compatible with a range of cryo-devices including the Cryojet, Cryostream, and Cobra.
- A sample with a phase transition at ca. 150 K was analysed at temperatures between 200 K and 100 K in 5K steps (a total of 21 data collections). See step 4.
- A movie of the crystal was taken at each temperature, and the cryo-device was given 10 minutes in which to stabilise at each temperature (Step 4).
- Automatic ramping and switching off of the cryo-device at the end of the experiment.
- In total, the full data collection was 16 hours; a convenient overnight experiment!
- Automatic data reduction and the use of AutoChem means very little user input is required to produce finished results for the 21 data sets.

How to set up the experiment:
- Run a pre-experiment
- Calculate the strategy and open the Temperature Strategy window
- Select temperatures required and import strategy
- Start experiment

Results:
- The crystal is clearly observed to become a darker green colour with decreasing temperature. Beyond 150 K, further colour changes were not observed due to the intensity of the crystal colour.
- The length of the c- and a-axes and the volume are displayed as a function of temperature in Graphs 1-3, respectively.
- Analysis of the individual cell parameters shows that the a axis decreases rapidly around the phase transition, while the c axis increases.

Graph 1. c-axis vs. temperature
Graph 2. a-axis vs temperature
Graph 3. Cell volume vs temperature