

LOW TEMPERATURE MEASUREMENTS OF KCN AND NH₄Br WITH AN OXFORD CRYOSYSTEMS COBRA ON A STOE STADI P ESSENTIALS

SETUP

Powder Samples of KCN and NH₄Br were measured using capillaries (0.3mm, 0.5mm TYPE 14) in Debye-Scherrer setup on a STOE STADI P ESSENTIALS diffractometer equipped with pure Cu K α ₁ radiation and a 320 μ DECTRIS MYTHEN2 1K 450. The temperature was controlled by an Oxford Cryosystems Cobra closed-cycle He cryostat (c.f. Figure 1).

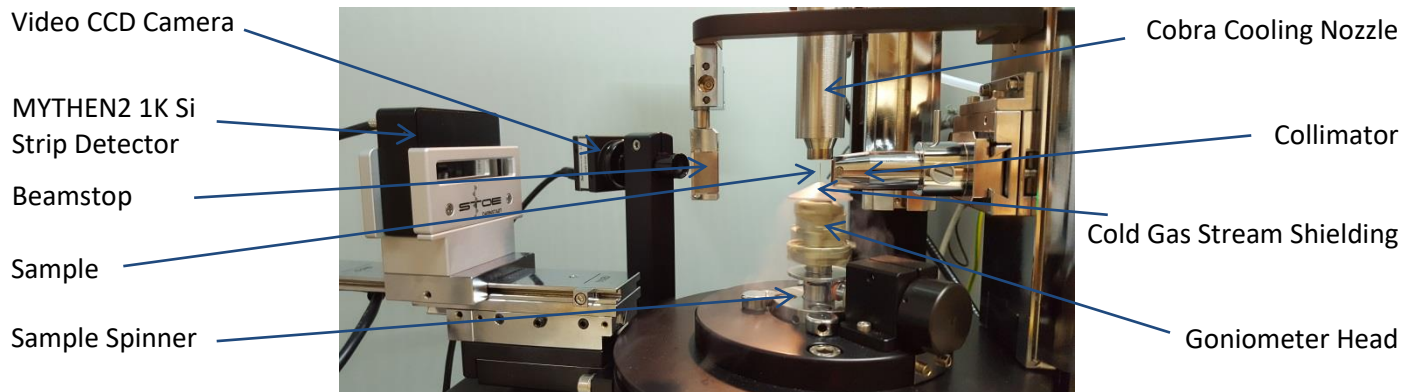


Figure 1: Setup of the STOE STADI P ESSENTIALS goniometer equipped with an Oxford Cryosystems Cobra

RESULTS

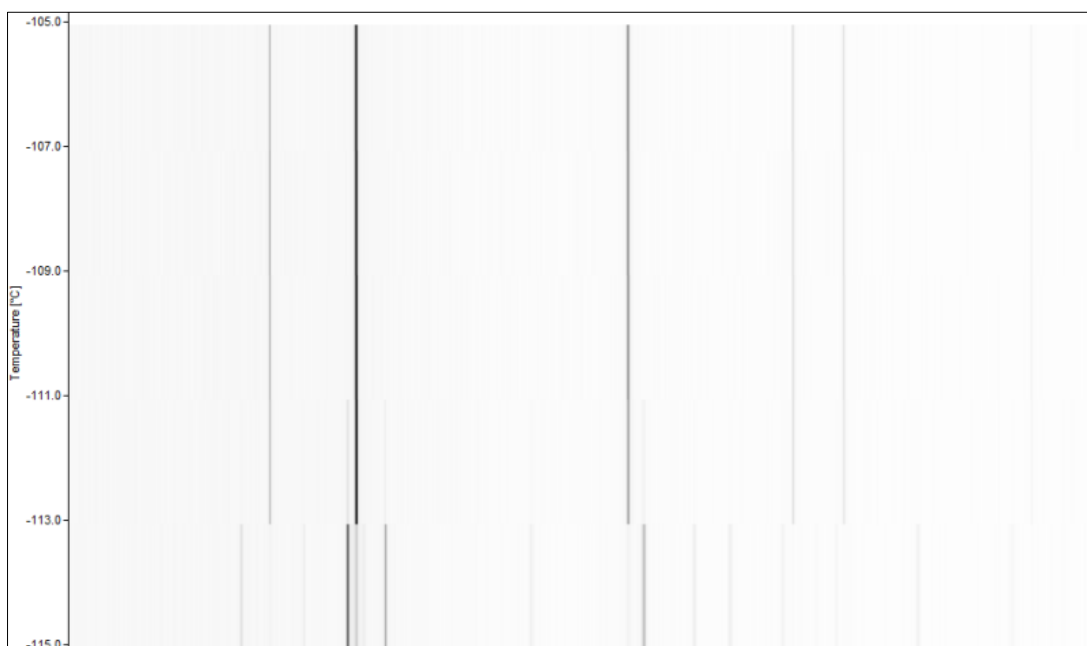


Figure 2: Guinier Plot of KCN between 15° to 60° 2 θ and -105°C to -115°C

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A first phase transition in KCN ($Fm\bar{3}m \rightarrow Immm$) is reported to appear at 168 K ($\sim -105^\circ\text{C}$) and a second ($Immm \rightarrow Pmmn$) at 83 K ($\sim -190^\circ\text{C}$).^[1] KCN was measured between 293 K and 83 K (20°C to -190°C) in 2°C steps. Figure 2 shows a Guinier-Plot between -105°C and -115°C (168K – 158K) where a phase transition at -113°C (160K) is clearly visible. The difference of 8K between the transition temperature reported in [1] and the measurement are in an expected range for such a setup and temperature region.

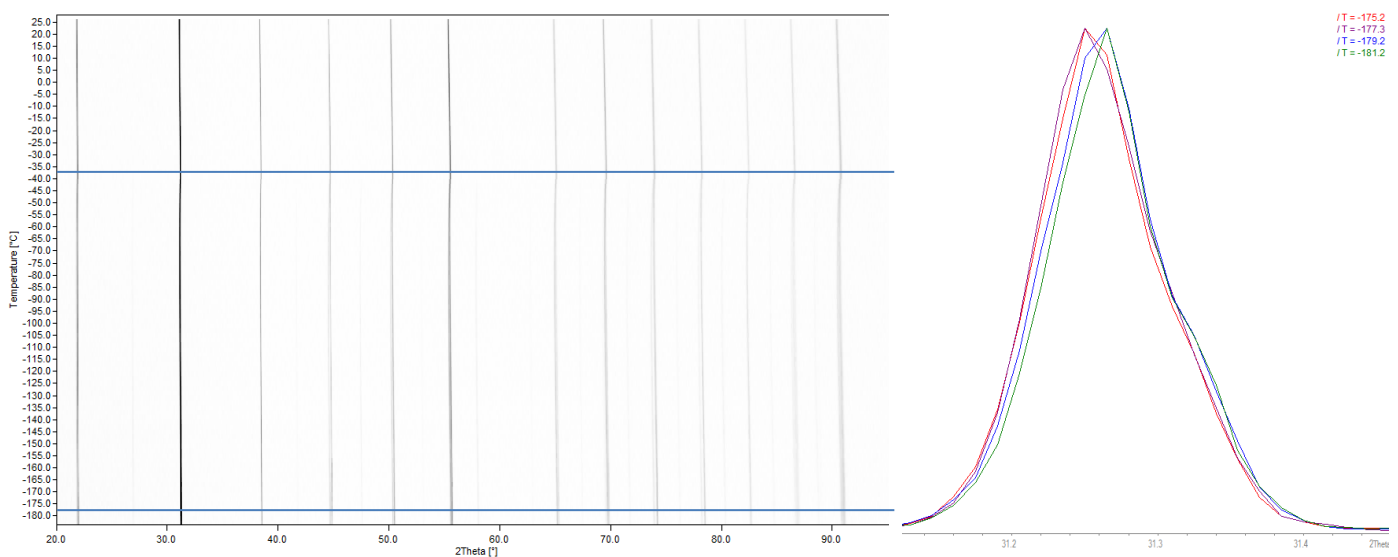


Figure 3: Guinier Plot from 25°C to -181°C ($\sim 298\text{K}$ to $\sim 90\text{K}$) between 20° and 95° 2θ of NH₄Br (left). The blue lines indicate two phase transitions at -38°C (235K) and -178°C (95K); Close-up at $\sim 31.25^\circ$ 2θ between -175°C (red) and -181°C (green) which emphasizes the second phase transition at -178°C (right)

NH₄Br exhibits two phase transitions at 235K (-38°C ; $Pm\bar{3}m \rightarrow P4/nmm$) and at 107K (-166°C ; $P4/nmm \rightarrow P\bar{4}3m$)^[2], which are both clearly visible in the diffraction patterns (c.f. Figure 3, left). The 2nd phase transition is characterized only by a small shift in the 2θ position of the reflections (c.f. Figure 3, right). The expected and measured temperature of the 1st phase transition match very well, whereas there is a deviation of $\sim 11^\circ\text{C}$ at the 2nd phase transition.

CONCLUSION

The combination of an Oxford Cryosystems Cobra with an STOE STADI P ESSENTIALS diffractometer is a very sophisticated setup, which enables one to measure high resolution powder diffraction patterns in a temperature range from 400 K to ~ 80 K. The samples chosen here cannot be seen as standard samples in terms of absolute temperature but are comparable to regular laboratory samples. Due to the high resolution of the STOE STADI P diffractometer it is possible to identify the phase transition of NH₄Br at 107K, which is only due to a small shift in the 2θ position of the reflections (c.f. Figure 3, right).

[1] D. Fontaine, *C.R. Acad. Sci Set.* **1975**, B281, 443.

[2] N. G. Personage, L. A. K. Staveley, *Disorder in crystals* **1978**, Clarendon Press, Oxford.