SHORT COMMUNICATIONS

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Crystal data of *o*-phenanthroline hydrate.

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The space group of *o*-phenanthroline hydrate has been determined as either $P3_1$ or the enantiomorphic $P3_2$; these differ from $P31m$ deduced by Sen [*Acta Cryst.* (1974). B30, 556]. Cell dimensions were redetermined as: $a=17{\cdot}7978$ (6), $c=8{\cdot}5214$ (3) Å.

Recently Sen (1974) reported the space group of *o*-phenanthroline hydrate as $P31m$, but we have arrived at a different space group in the course of our structure analysis, as described below.

o-Phenanthroline hydrate, certified grade from Wako Pure Chemical Industries Ltd, Japan, was recrystallized from a benzene solution as a colourless prism elongated along the *c* axis, m.p. 93–94 °C. By a careful examination of the Weissenberg photographs, and of the counter data obtained later, the Laue symmetry was determined to be $\bar{3}$, which was inconsistent with $\bar{3}m$ reported by Donnay, Donnay & Harding (1965). The absence of the *m* symmetry was evidenced by the observation of a clearly discernible inequality of *hkil* and *khil*, though the inequality was slight.

In order to identify the space group the 000*l* reflexions were carefully investigated, first on a Weissenberg photograph, secondly by counter techniques. An 0*kl* Weissenberg photograph of 70 h exposure with Ni-filtered Cu $K\alpha$ radiation at 30 kV and 18 mA showed a weak but sharp spot at the position corresponding to the 0001 reflexion, but its shape was different from that of other spots. The 0003 and 0006 reflexions were definitely observed.

Next, a precise measurement of the 0001 reflexion was attempted using the counter technique. The crystal was mounted on a Rigaku four-circle goniometer with the *c* axis parallel to the φ axis. The arcs of the goniometer head were adjusted so that the intensity of the 0003 reflexion was unchanged throughout the full 360° rotation of the φ axis at $\chi=90°$. When the crystal was rotated around the 0001 scattering vector, or the φ axis, keeping the diffraction condition for 0001, a number of peaks of various intensities were observed. These peaks should be Renninger reflexions or simultaneous reflexions. Thus the 0001 spot observed on the Weissenberg photograph can be ascribed to a Renninger reflexion. The sharp shape of the spot supports this conclusion. The crystal was readjusted in order to reduce the influence of the Renninger effect on the 0001 reflexion, and the intensity was measured using the θ–2θ step-scan technique with a step of 0·02° (in 2θ), the respective counting time being 120 s. The scan width was 1·60° (in 2θ) with background counts of 1944 s on either side of the scan. We obtained the values of 94933(308), 19890(141) and 18538(136) counts for the total counts during the scan, and background counts before and after the scan, respectively. The estimated standard deviations are given in parentheses. We thus have intensity $I=-1137$ with standard deviation $\sigma(I)=579$ by simple computations. The negative value would be due to inadequate subtraction of the background, and should be read as zero. Thus it can be stated with certainty that the 0001 reflexion is definitely absent. In conclusion the space group of *o*-phenanthroline hydrate is $P3_1$ or enantiomorphous $P3_2$.

The cell parameters were redetermined by a least-squares fit of 18 reflexions in the range $70°<2\theta<85°$ (Cu $K\alpha_1$, $\lambda=1{\cdot}54051$ Å) on the diffractometer. Table 1 summarizes the crystal data. Specimens recrystallized from a hot aqueous solution were also examined and the same conclusion was reached. We have completed the three-dimensional data collection and the structure analysis is in progress.

Table 1. *Crystal data of o-phenanthroline hydrate*

	Present work	Donnay *et al.* (1965)	Sen (1974)
Laue symmetry	$\bar{3}$	$\bar{3}m$	
Space group	$P3_1$, $P3_2$	$P31m$ (most probable)	$P31m$
a (Å)	17·7978 (6)	17·67 (5)	17·67
c (Å)	8·5214 (3)	8·55 (3)	8·55
U (Å^3)	2337·6 (2)	2312	2312
D_m (g cm^{-3})	1·27	1·25 (5)	1·24
D_x (g cm^{-3})	1·267	1·28	1·28

References

DONNAY, G., DONNAY, J. D. H. & HARDING, M. J. C. (1965). *Acta Cryst.* **19**, 688–689.

SEN, M. (1974). *Acta Cryst.* B30, 556.