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The twinned crystal structure of 3-indolylacetic acid

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.029
wR factor = 0.074
Data-to-parameter ratio = 13.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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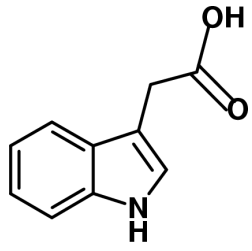
Crystals of the title compound, $C_{10}H_9NO_2$, whose structure has already been determined four times by different research groups, were found to be twinned. Satisfactory refinement is only possible if the twinning is taken into account. This kind of twinning in the monoclinic crystal system can, in principle, occur if the following condition is met: $a\cdot|\cos\beta| = c/2$, *i.e.* if the short diagonal of the *ac* plane is of the same length as *a* or *c* itself.

Comment

The structure of the title compound, (I), has already been determined (Karle *et al.*, 1964; Chandrasekhar & Raghunathan, 1982; Pfeiffer *et al.*, 1987; Nigović *et al.*, 2000), but none of the authors has described any twinning.



(I)

Surprisingly, we encountered a twinned crystal and satisfactory refinement was only possible when the correct twin law, (101/010/00$\bar{1}$), was applied. This case of twinning is characterized by the fact that one of the face diagonals is of the same length as one of the axes bordering the face. As a result, the face diagonal can be an axis of the twin unit cell, whereas the other axis is reversed. The present kind of twinning in the monoclinic crystal system can, in principle, occur if the following condition is met: $a\cdot|\cos\beta| = c/2$. The structure determinations of Chandrasekhar & Raghunathan (1982), Pfeiffer *et al.* (1987) and Nigović *et al.* (2000) have led to acceptable results. However, Karle *et al.* (1964), who had used equi-inclination Weissenberg photographs and zero-level precession photographs on a thin plate-like crystal to collect the diffracted intensities, ascribed the relatively high R value to the shape of the crystal and to the difficulty in estimating the density of the diffraction spots.

Experimental

The title compound was purchased from Fluka and recrystallized from diethyl ether.

Crystal data

$C_{10}H_9NO_2$
M_r = 175.18
Monoclinic, $P2_1/c$
a = 17.848 (2) Å
b = 5.2214 (7) Å
c = 9.568 (1) Å
β = 105.70 (1)°
V = 858.39 (17) Å^3
Z = 4
D_x = 1.356 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 10 779 reflections
θ = 1.7–26.5°
μ = 0.10 mm^{-1}
T = 100 (2) K
Block, colourless
0.40 × 0.30 × 0.20 mm

Data collection

Stoe IPDS–II two-circle diffractometer
ω scans
Absorption correction: none
11 341 measured reflections
1756 independent reflections
1476 reflections with $I > 2\sigma(I)$
R_{int} = 0.050
θ_{max} = 26.4°
$h = -22 \rightarrow 22$
$k = -6 \rightarrow 6$
$l = -11 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.074
S = 1.01
1756 reflections
128 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0454P)^2 + 0.0141P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.046 (5)

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O1^{i}	0.94 (3)	1.70 (3)	2.6372 (16)	172 (2)

Symmetry code: (i) $1-x, 1-y, 1-z$.

After having encountered severe problems during structure solution, anisotropic refinement remained stalled at $R1$ = 0.28. It was therefore assumed that the crystal was twinned and applying the twin law (101/010/00$\bar{1}$), which is equivalent to ($\bar{1}0\bar{1}$/010/001), eventually succeeded ($R1$ dropped below 0.1). All H atoms could now be located by difference Fourier synthesis. They were refined with fixed individual displacement parameters [U(H) = 1.2U_{eq}(C)] using a riding model with C—H(aromatic) = 0.95 Å or C—H(methylene) = 0.99 Å. H atoms bonded to N and O atoms were refined isotropically. The twin ratio refined to 0.416 (1)/0.584 (1).

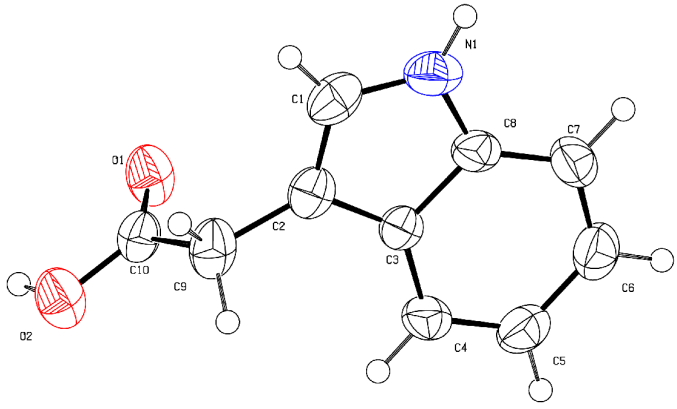


Figure 1
Perspective view of (I) with the atom numbering; displacement ellipsoids are at the 50% probability level.

Data collection: *X-Area* (Stoe & Cie, 2001); cell refinement: *X-Area*; data reduction: *X-Area*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 1990).

References

Chandrasekhar, K. & Raghunathan, S. (1982). *Acta Cryst.* B**38**, 2534–2535.
Karle, I. L., Britts, K. & Gum, P. (1964). *Acta Cryst.* **17**, 496–499.
Nigović, B., Antolic, S., Kojic Prodic, B., Kiralj, R., Magnus, V. & Salopek-Sondi, B. (2000). *Acta Cryst.* B**56**, 94–111.
Pfeiffer, D., Kutschabsky, L., Leibnitz, P. & Adam, G. (1987). *Cryst. Res. Technol.* **22**, K1–K4.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.
Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.
Stoe & Cie (2001). *X-Area*. Stoe & Cie, Darmstadt, Germany.