View metadata, citation and similar papers at core.ac.uk

brought to you by CORE

provided by AIR Universita degli studi di Milano

ARTICLE IN PRESS – Acta Cryst. E

Acta Cryst E STRUCTURE REPORTS

ISSN 1600-5368

Crystal structure of 3-(3-oxo-2,3,4,4a,5,6-hexahydrobenzo[*h*]cinnolin-2-yl)propionic acid

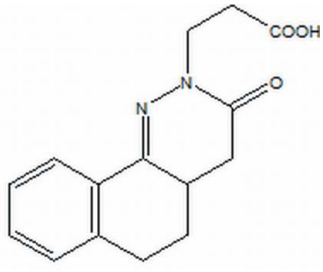
Proof instructions

Proof corrections should be returned by **6 September 2014**. After this period, the Editors reserve the right to publish your article with only the Managing Editor's corrections.

Please
(1) Read these proofs and assess whether any corrections are necessary.
(2) Check that any technical editing queries highlighted in **bold underlined** text have been answered.
(3) Send corrections by e-mail to **checkin@iucr.org**. Please describe corrections using plain text, where possible, giving the line numbers indicated in the proof. Please do not make corrections to the pdf file electronically and please do not return the pdf file. If no corrections are required please let us know.

If you wish to purchase printed offprints, please complete the attached order form and return it by e-mail as soon as possible.

Please check the following details for your article



Thumbnail image for contents page

Abbreviated author list: Meneghetti, F.; Masciocchi, D.; Gelain, A.; Villa, S.

How to cite your article in press

Your article has not yet been assigned page numbers, but may be cited using the doi:

Meneghetti, F., Masciocchi, D., Gelain, A. & Villa, S. (2014). *Acta Cryst.* E**70**, doi:10.1107/S1600536814019850.

You will be sent the full citation when your article is published and also given instructions on how to download an electronic reprint of your article.

Files: e/hb7233/hb7233.3d e/hb7233/hb7233.sgml **HB7233** HO IU-147/17(3)9 147/12(3)9 (70) HB7233 PROOFS E:HO:2014:70:1:0:O1–O

Acta Cryst E STRUCTURE REPORTS

OPEN ACCESS

ISSN 1600-5368

Crystal structure of 3-(3-oxo-2,3,4,4a,5,6-hexahydrobenzo[*h*]cinnolin-2-yl)propionic acid

Fiorella Meneghetti,* Daniela Masciocchi, Arianna Gelain and Stefania Villa

Department of Pharmaceutical Sciences, University of Milano, via L. Mangiagalli, 25, 20133-Milano, Italy. *Correspondence e-mail: fiorella.meneghetti@unimi.it

Received 21 May 2014; accepted 2 September 2014

Edited by W. T. A. Harrison, University of Aberdeen, Scotland

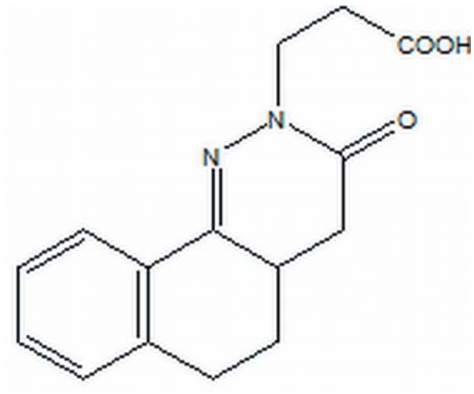
The asymmetric unit of the title compound, $C_{15}H_{16}N_2O_3$, contains two independent molecules, which present a different conformation of the carboxylic acid side chain [C—C—C—OH torsion angles = 65.3 (7) and −170.1 (5)°]. In both molecules, the dihydropyridazinone ring adopts a geometry intermediate between a twisted-boat and a half-chair conformation, while the central six-membered ring is almost in a half-boat conformation. In the crystal, molecules are linked by O—H···O_k (k = ketone) hydrogen bonds, generating [01$\bar{1}$] chains. Aromatic π–π stacking contacts between the benzene and the dihydropyridazinone rings [centroid–centroid distance [3.879 (9) Å] are also observed.

Keywords: crystal structure; pyridazinone moiety; stat3 inhibitor.

CCDC reference: 874435

1. Related literature

For background to the bioactivity of pyridazinone derivatives, see: Masciocchi *et al.* (2013). For structural and molecular modeling studies, see: Toma *et al.* (1990). For the chemistry of pyridazinone derivatives, see: Costantino *et al.* (1996).



2. Experimental

2.1. Crystal data

$C_{15}H_{16}N_2O_3$
M_r = 272.3
Triclinic, $P\bar{1}$
a = 11.217 (4) Å
b = 11.668 (4) Å
c = 12.110 (4) Å
α = 79.22 (1)°
β = 64.62 (1)°
γ = 68.630 (9)°
V = 1332.6 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 K
0.65 × 0.45 × 0.40 mm

2.2. Data collection

Enraf–Nonius TurboCAD-4 diffractometer
5412 measured reflections
4682 independent reflections
1412 reflections with $I > 2\sigma(I)$
R_{int} = 0.081
3 standard reflections every 120 min
intensity decay: 9%

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.205
S = 0.92
4682 reflections
368 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2A—H2A···O1B	0.92 (7)	1.78 (7)	2.651 (6)	158 (6)
O2B—H2B···O1A	0.90 (6)	1.75 (6)	2.598 (7)	157 (5)

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *WinGX* (Farrugia, 2012).

Acknowledgements

This study was supported by funds from PRIN 2010–2011.

Files: e/hb7233/hb7233.3d e/hb7233/hb7233.sgml **HB7233** HO IU-147/17(3)9 147/12(3)9 (70) HB7233 PROOFS E:HO:2014:70:1:0:O1–O

Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7233).

References

Altomare, A., Burla, M. C., Camalli, M., Cascarano, G., Giacovazzo, C., Gagliardi, A. & Polidori, G. (1994). *J. Appl. Cryst.* **27**, 435.

Costantino, L., Rastelli, G., Vescovili, K., Cignarella, G., Vianello, P., Del Corso, A., Cappiello, M., Mura, U. & Barlocco, D. (1996). *J. Med. Chem.* **39**, 4396–4405.

Enraf–Nonius (1994). *CAD-4 EXPRESS*. Enraf–Nonius, Delft, The Netherlands.

Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.

Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.

Masciocchi, D., Gelain, A., Porta, F., Meneghetti, F., Pedretti, A., Celentano, G., Barlocco, D., Legnani, L., Toma, L., Kwon, B.-M., Asai, A. & Villa, S. (2013). *Med. Chem. Commun.* **4**, 1181–1188.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Toma, L., Cignarella, G., Barlocco, D. & Ronchetti, F. (1990). *J. Med. Chem.* **33**, 1591–1594.

Files: e/hb7233/hb7233.3d e/hb7233/hb7233.sgml **HB7233** HO IU-147/17(3)9 147/12(3)9 (70) HB7233 PROOFS E:HO:2014:70:1:0:O1–O

supporting information

Crystal structure of 3-(3-oxo-2,3,4,4a,5,6-hexahydrobenzo[*h*]cinnolin-2-yl)propionic acid

Fiorella Meneghetti,* Daniela Masciocchi, Arianna Gelain and Stefania Villa

S1. Structural commentary

In our previous researches focused on the discovery of new inhibitors targeting aberrant STAT3 signaling for the treatment of human cancers, we evidenced that several pyridazinone derivatives were able to interfer within the STAT3 pathway (Masciocchi *et al.*, 2013). As the size of the central ring plays a main role in determining the conformational properties for this class of compounds, we investigated the extent of planarity of the phenyl with respect to the other cycles by crystallographic analysis determining the molecular structure of the title compound. The asymmetric unit of the title compound (Fig. 1) is characterized by two crystallographically independent molecules (*a* and *b*). The values obtained for the bond length and angles of the two independent molecules are in accordance with each other, whilst at the same time presenting a different conformation of the carboxylic chain linked to N1. This difference is best evidenced by the torsion angles N2—N1—C13—C14 of 76 (1)°[-100 (1)°] and O2—C15—C14—C13 of 65 (1)°[-170 (1)°] (the values in the square brackets refer to the *b* labeled molecule). The tricyclic skeleton of the compound consists of three fused rings slightly twisted with respect to each other. The dihedral angles between their least-square planes α (C1/N1/N2/C4/C3/C2), β (C3/C4/C5/C6/C7/C8) and γ (C5/C6/C12/C11/C10/C9) are: α-β = 5.0 (1)°[3.0 (1)°], α-γ = 11.5 (1)°[10.3 (1)°], β-γ = 6.6 (1)°[7.3 (1)°], respectively. In detail, the dihydropyridazinone ring adopts a geometry intermediate between a twisted-boat and a half-chair conformation quantitatively defined by the parameters QT = 0.321 (6)Å[0.242 (6)Å], φ = -87 (1)°[-83 (1)°] and θ = 113.2 (9)°[114 (1)°], while the central six-membered ring is almost in a half-boat conformation, characterized by the puckering parameters QT = 0.372 (7)Å[0.334 (7)Å], φ = -64 (1)°[-58 (1)°] and θ = 57 (1)°[59 (1)°], with the flap atom C8 out of the best mean plane calculated over the other five carbons by 0.512 (6)Å[0.462 (6)Å]. In the crystal, the *a* and *b* molecules are flattened and lay on planes deviated from that containing the *a* and *c* axes by about 30°. The two conformers interact through π-π contacts between the benzene and the dihydropyridazinone rings, at a centroid-centroid distance of 3.879 (9)Å. In addition, *a* and *b* molecules are interconnected through hydrogen bonds, where the carboxylic oxygen O2 is donor of a proton to the ketonic oxygen O1 of the partner molecule (Fig. 2). The intermolecular contacts involve O2a which is linked to a centrosymmetrically related molecule of *b* (O2a—H···O1b(i) at a distance of 1.78 (7)Å and angle of 158 (6)° [symmetry code: (i) -*x*, 1 - *y*, 1 - *z*)] and O2b that hydrogen bonds a molecule related to *a* by a crystallographic inversion centre (O2b–H···O1a(ii) at a distance of 1.75 (6)Å and angle of 157 (5)° [symmetry code:(ii) at -*x*, 2 - *y*, - *z*)].

S2. Crystallization

After many attempts weakly diffracting yellow prisms were grown by slow evaporation of a 30:70 water/methanol solution.

S3. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. All non-H-atoms were refined anisotropically. The H-atoms positions bonded to heteroatoms were obtained by a close examination of a final difference Fourier, while the remaining ones were introduced at calculated positions and refined with fixed isotropic thermal parameters (1.2 Ueq of the parent atom).

fig1.tif

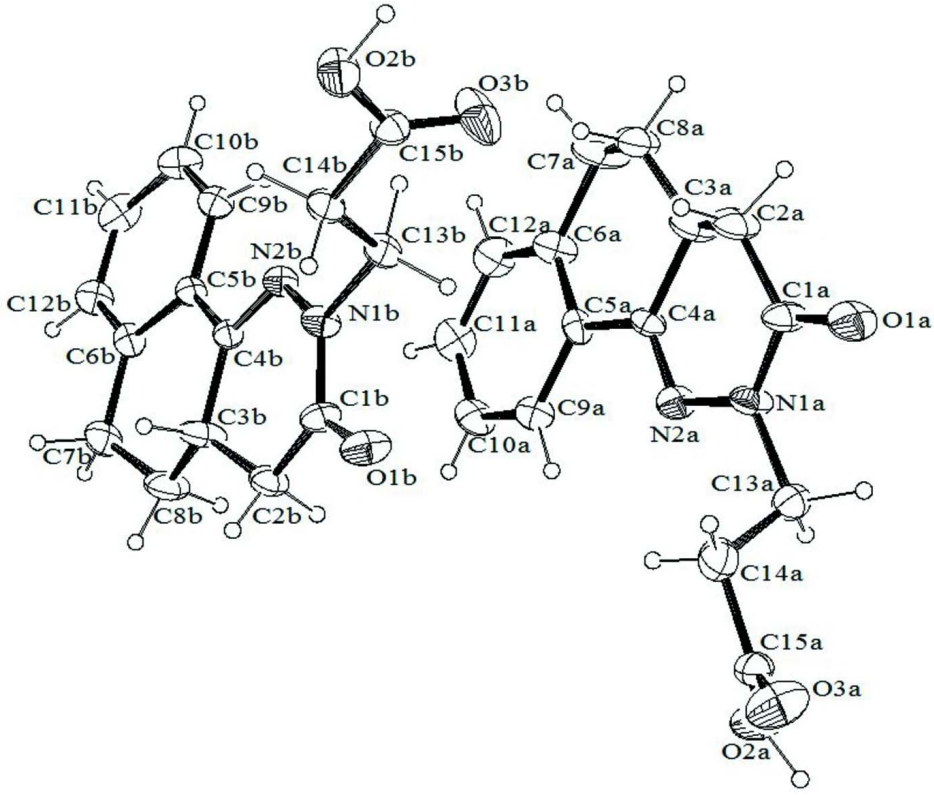


Figure 1

The molecular structure of the asymmetric unit of the title compound, showing displacement ellipsoids for non-H atoms at the 40% probability level.

fig2.tif

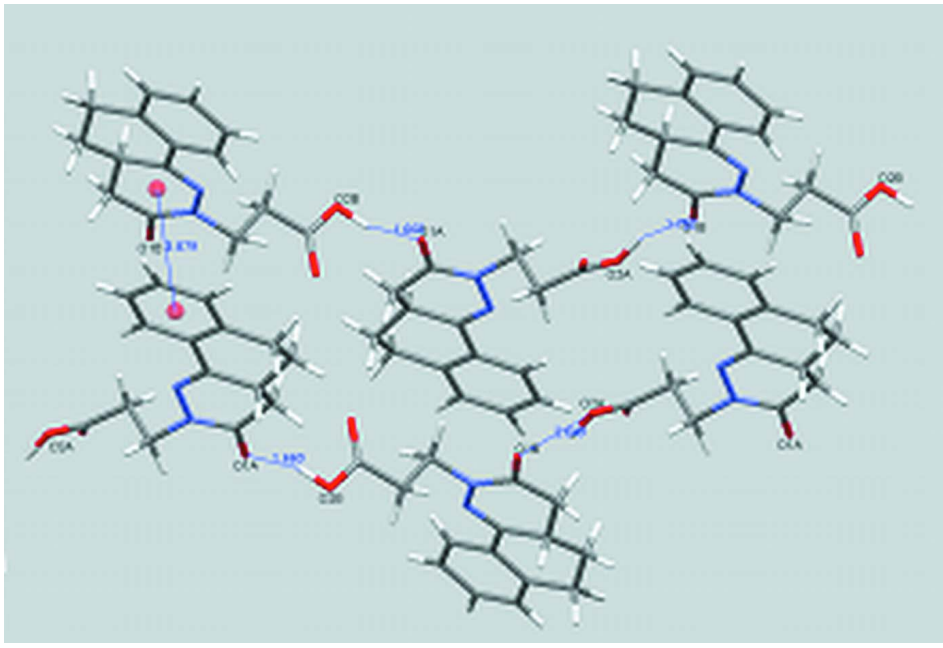


Figure 2

Intermolecular interactions of the title compound. Hydrogen bonds are shown as dashed lines.

3-(3-Oxo-2,3,4,4a,5,6-hexahydrobenzo[*h*]cinnolin-2-yl)propionic acid

Crystal data

$C_{15}H_{16}N_2O_3$	$Z = 4$
$M_r = 272.3$	$F(000) = 576$
Triclinic, $P\overline{1}$	$D_x = 1.357$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 11.217$ (4) Å	Cell parameters from 25 reflections
$b = 11.668$ (4) Å	$\theta = 9$–$10°$
$c = 12.110$ (4) Å	$\mu = 0.10$ mm^{-1}
$\alpha = 79.22$ (1)°	$T = 293$ K
$\beta = 64.62$ (1)°	Prism, yellow
$\gamma = 68.630$ (9)°	0.65 × 0.45 × 0.40 mm
$V = 1332.6$ (8) Å^3	

Data collection

Enraf–Nonius TurboCAD-4 diffractometer	$R_{int} = 0.081$
Radiation source: fine-focus sealed tube	$\theta_{max} = 25.0°$, $\theta_{min} = 2.1°$
Graphite monochromator	$h = -12 \rightarrow 13$
non–profiled $\omega/2\theta$ scans	$k = -13 \rightarrow 13$
5412 measured reflections	$l = -1 \rightarrow 14$
4682 independent reflections	3 standard reflections every 120 min
1412 reflections with $I > 2\sigma(I)$	intensity decay: 9%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.059$
$wR(F^2) = 0.205$
$S = 0.92$
4682 reflections
368 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0932P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.28$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1A	0.0119 (4)	0.6619 (4)	0.0134 (4)	0.0688 (13)
O2A	−0.0208 (4)	0.3563 (4)	0.4257 (4)	0.0671 (14)
H2A	−0.067 (6)	0.299 (6)	0.451 (6)	0.08 (2)*
O3A	−0.2262 (5)	0.4797 (4)	0.4325 (5)	0.0882 (16)
N1A	0.1447 (4)	0.5851 (4)	0.1231 (4)	0.0496 (13)
N2A	0.2481 (5)	0.5828 (4)	0.1591 (4)	0.0471 (13)
C1A	0.1087 (6)	0.6616 (5)	0.0356 (6)	0.0530 (17)
C2A	0.1924 (6)	0.7439 (6)	−0.0252 (6)	0.068 (2)
H2A1	0.2002	0.7583	−0.1092	0.081*
H2A2	0.1415	0.8224	0.0140	0.081*
C3A	0.3308 (6)	0.7041 (6)	−0.0269 (6)	0.0621 (18)
H3A	0.3821	0.6377	−0.0852	0.075*
C4A	0.3353 (6)	0.6383 (5)	0.0923 (5)	0.0383 (14)
C5A	0.4477 (6)	0.6302 (5)	0.1270 (5)	0.0408 (14)
C6A	0.5553 (5)	0.6744 (5)	0.0508 (5)	0.0520 (17)
C7A	0.5541 (6)	0.7381 (7)	−0.0711 (6)	0.081 (2)
H7A1	0.5920	0.8051	−0.0879	0.097*
H7A2	0.6149	0.6797	−0.1354	0.097*
C8A	0.4166 (6)	0.7870 (6)	−0.0752 (6)	0.067 (2)
H8A1	0.4281	0.8098	−0.1595	0.080*
H8A2	0.3654	0.8618	−0.0294	0.080*
C9A	0.4508 (6)	0.5720 (5)	0.2382 (5)	0.0484 (16)
H9A	0.3789	0.5420	0.2913	0.058*
C10A	0.5593 (6)	0.5589 (5)	0.2699 (6)	0.0576 (18)

H10A	0.5600	0.5200	0.3442	0.069*
C11A	0.6657 (6)	0.6025 (6)	0.1934 (7)	0.068 (2)
H11A	0.7392	0.5929	0.2148	0.081*
C12A	0.6632 (6)	0.6610 (6)	0.0840 (6)	0.0662 (19)
H12A	0.7350	0.6917	0.0320	0.079*
C13A	0.0675 (6)	0.4990 (5)	0.1958 (6)	0.0529 (16)
H13A	0.1326	0.4197	0.2051	0.063*
H13B	0.0167	0.4871	0.1534	0.063*
C14A	−0.0311 (6)	0.5484 (5)	0.3179 (6)	0.0610 (18)
H14A	−0.0990	0.6255	0.3081	0.073*
H14B	0.0194	0.5652	0.3576	0.073*
C15A	−0.1058 (7)	0.4600 (5)	0.3977 (6)	0.0529 (16)
O1B	0.1349 (4)	0.8168 (3)	0.4536 (4)	0.0599 (12)
O2B	0.0424 (5)	1.2275 (4)	0.1748 (4)	0.0815 (16)
H2B	0.038 (6)	1.246 (5)	0.101 (5)	0.070*
O3B	0.1379 (6)	1.0503 (4)	0.0884 (5)	0.0973 (19)
N1B	0.3133 (4)	0.8868 (4)	0.3469 (4)	0.0422 (12)
N2B	0.4445 (4)	0.8964 (4)	0.3189 (4)	0.0374 (11)
C1B	0.2460 (6)	0.8252 (5)	0.4432 (5)	0.0435 (15)
C2B	0.3141 (6)	0.7674 (5)	0.5298 (5)	0.0506 (16)
H2B1	0.2426	0.7792	0.6121	0.061*
H2B2	0.3534	0.6795	0.5173	0.061*
C3B	0.4248 (6)	0.8110 (6)	0.5232 (5)	0.0609 (19)
H3B	0.3709	0.8868	0.5689	0.073*
C4B	0.4978 (5)	0.8586 (4)	0.3997 (5)	0.0347 (13)
C5B	0.6346 (5)	0.8694 (4)	0.3670 (5)	0.0355 (14)
C6B	0.6924 (5)	0.8414 (5)	0.4544 (5)	0.0425 (15)
C7B	0.6210 (6)	0.7916 (5)	0.5799 (5)	0.0535 (17)
H7B1	0.6902	0.7264	0.6035	0.064*
H7B2	0.5783	0.8566	0.6376	0.064*
C8B	0.5143 (7)	0.7434 (6)	0.5873 (6)	0.069 (2)
H8B1	0.5608	0.6606	0.5571	0.083*
H8B2	0.4553	0.7367	0.6731	0.083*
C9B	0.7096 (5)	0.9085 (5)	0.2506 (5)	0.0453 (15)
H9B	0.6733	0.9244	0.1912	0.054*
C10B	0.8366 (6)	0.9246 (5)	0.2198 (6)	0.0570 (18)
H10B	0.8844	0.9527	0.1418	0.068*
C11B	0.8898 (6)	0.8975 (5)	0.3091 (6)	0.0593 (19)
H11B	0.9754	0.9066	0.2904	0.071*
C12B	0.8196 (6)	0.8580 (5)	0.4235 (6)	0.0489 (16)
H12B	0.8572	0.8418	0.4820	0.059*
C13B	0.2663 (5)	0.9335 (5)	0.2464 (5)	0.0463 (15)
H13C	0.2232	0.8791	0.2377	0.056*
H13D	0.3459	0.9344	0.1705	0.056*
C14B	0.1637 (6)	1.0613 (5)	0.2699 (5)	0.0510 (16)
H14C	0.2069	1.1154	0.2791	0.061*
H14D	0.0842	1.0601	0.3458	0.061*
C15B	0.1141 (6)	1.1115 (6)	0.1669 (6)	0.0479 (15)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1A	0.069 (3)	0.075 (3)	0.081 (3)	−0.027 (2)	−0.057 (3)	0.031 (2)
O2A	0.051 (3)	0.069 (3)	0.083 (3)	−0.025 (3)	−0.037 (3)	0.031 (3)
O3A	0.063 (3)	0.064 (3)	0.130 (5)	−0.018 (3)	−0.044 (3)	0.025 (3)
N1A	0.044 (3)	0.063 (3)	0.046 (3)	−0.024 (3)	−0.025 (3)	0.022 (3)
N2A	0.046 (3)	0.051 (3)	0.057 (3)	−0.017 (3)	−0.037 (3)	0.017 (3)
C1A	0.055 (4)	0.046 (4)	0.055 (4)	−0.016 (3)	−0.028 (4)	0.021 (3)
C2A	0.073 (5)	0.080 (5)	0.065 (5)	−0.035 (4)	−0.049 (4)	0.039 (4)
C3A	0.056 (4)	0.076 (4)	0.052 (4)	−0.021 (4)	−0.031 (4)	0.026 (4)
C4A	0.040 (3)	0.039 (3)	0.032 (3)	−0.008 (3)	−0.019 (3)	0.009 (3)
C5A	0.043 (3)	0.036 (3)	0.043 (4)	0.000 (3)	−0.026 (3)	−0.006 (3)
C6A	0.030 (3)	0.067 (4)	0.048 (4)	−0.011 (3)	−0.014 (3)	0.012 (3)
C7A	0.049 (4)	0.117 (6)	0.060 (5)	−0.039 (4)	−0.013 (4)	0.038 (4)
C8A	0.078 (5)	0.078 (5)	0.055 (4)	−0.041 (4)	−0.036 (4)	0.033 (4)
C9A	0.041 (4)	0.044 (3)	0.049 (4)	−0.002 (3)	−0.020 (3)	0.004 (3)
C10A	0.061 (4)	0.054 (4)	0.059 (5)	−0.001 (3)	−0.042 (4)	0.006 (3)
C11A	0.046 (4)	0.077 (5)	0.081 (6)	−0.008 (4)	−0.032 (4)	−0.010 (4)
C12A	0.044 (4)	0.087 (5)	0.067 (5)	−0.025 (4)	−0.019 (4)	−0.001 (4)
C13A	0.051 (4)	0.050 (4)	0.064 (5)	−0.019 (3)	−0.029 (4)	0.007 (3)
C14A	0.067 (4)	0.056 (4)	0.063 (5)	−0.019 (4)	−0.027 (4)	−0.006 (4)
C15A	0.066 (4)	0.054 (4)	0.052 (4)	−0.030 (4)	−0.033 (4)	0.016 (3)
O1B	0.046 (2)	0.061 (3)	0.078 (3)	−0.023 (2)	−0.036 (2)	0.026 (2)
O2B	0.128 (4)	0.046 (3)	0.085 (4)	0.002 (3)	−0.082 (4)	0.004 (3)
O3B	0.139 (5)	0.066 (3)	0.081 (4)	0.026 (3)	−0.081 (4)	−0.018 (3)
N1B	0.032 (3)	0.052 (3)	0.044 (3)	−0.009 (2)	−0.026 (2)	0.012 (3)
N2B	0.031 (3)	0.038 (3)	0.040 (3)	−0.008 (2)	−0.017 (2)	0.004 (2)
C1B	0.037 (4)	0.039 (3)	0.055 (4)	−0.012 (3)	−0.025 (3)	0.016 (3)
C2B	0.057 (4)	0.060 (4)	0.042 (4)	−0.025 (3)	−0.029 (3)	0.018 (3)
C3B	0.056 (4)	0.079 (5)	0.050 (4)	−0.032 (4)	−0.029 (4)	0.035 (4)
C4B	0.040 (3)	0.028 (3)	0.037 (4)	−0.006 (2)	−0.020 (3)	0.001 (3)
C5B	0.036 (3)	0.034 (3)	0.039 (4)	−0.006 (3)	−0.023 (3)	0.003 (3)
C6B	0.040 (4)	0.038 (3)	0.046 (4)	−0.001 (3)	−0.023 (3)	−0.003 (3)
C7B	0.054 (4)	0.058 (4)	0.053 (4)	−0.010 (3)	−0.036 (3)	0.007 (3)
C8B	0.090 (5)	0.083 (5)	0.055 (5)	−0.047 (4)	−0.047 (4)	0.037 (4)
C9B	0.040 (4)	0.042 (3)	0.047 (4)	−0.003 (3)	−0.023 (3)	0.006 (3)
C10B	0.036 (3)	0.056 (4)	0.062 (5)	−0.012 (3)	−0.011 (3)	0.012 (3)
C11B	0.037 (4)	0.056 (4)	0.085 (6)	−0.010 (3)	−0.031 (4)	0.004 (4)
C12B	0.039 (4)	0.051 (4)	0.058 (5)	−0.003 (3)	−0.032 (4)	0.002 (3)
C13B	0.037 (3)	0.055 (4)	0.046 (4)	−0.009 (3)	−0.023 (3)	0.004 (3)
C14B	0.057 (4)	0.051 (4)	0.047 (4)	−0.011 (3)	−0.032 (3)	0.009 (3)
C15B	0.052 (4)	0.039 (4)	0.049 (4)	−0.005 (3)	−0.026 (3)	0.003 (3)

Geometric parameters (Å, º)

O1A—C1A	1.225 (6)	O1B—C1B	1.236 (6)
O2A—C15A	1.330 (7)	O2B—C15B	1.295 (7)

197	O2A—H2A	0.92 (6)	O2B—H2B	0.90 (6)
198	O3A—C15A	1.174 (6)	O3B—C15B	1.175 (6)
199	N1A—C1A	1.353 (7)	N1B—C1B	1.332 (6)
200	N1A—N2A	1.395 (5)	N1B—N2B	1.403 (5)
201	N1A—C13A	1.477 (6)	N1B—C13B	1.466 (6)
202	N2A—C4A	1.272 (6)	N2B—C4B	1.287 (6)
203	C1A—C2A	1.465 (8)	C1B—C2B	1.478 (7)
204	C2A—C3A	1.441 (7)	C2B—C3B	1.472 (7)
205	C2A—H2A1	0.9700	C2B—H2B1	0.9700
206	C2A—H2A2	0.9700	C2B—H2B2	0.9700
207	C3A—C8A	1.480 (8)	C3B—C8B	1.448 (7)
208	C3A—C4A	1.515 (7)	C3B—C4B	1.478 (7)
209	C3A—H3A	0.9800	C3B—H3B	0.9800
210	C4A—C5A	1.459 (7)	C4B—C5B	1.459 (7)
211	C5A—C6A	1.377 (7)	C5B—C9B	1.388 (7)
212	C5A—C9A	1.399 (7)	C5B—C6B	1.397 (7)
213	C6A—C12A	1.380 (7)	C6B—C12B	1.389 (7)
214	C6A—C7A	1.526 (8)	C6B—C7B	1.501 (7)
215	C7A—C8A	1.457 (7)	C7B—C8B	1.461 (7)
216	C7A—H7A1	0.9700	C7B—H7B1	0.9700
217	C7A—H7A2	0.9700	C7B—H7B2	0.9700
218	C8A—H8A1	0.9700	C8B—H8B1	0.9700
219	C8A—H8A2	0.9700	C8B—H8B2	0.9700
220	C9A—C10A	1.376 (7)	C9B—C10B	1.384 (7)
221	C9A—H9A	0.9300	C9B—H9B	0.9300
222	C10A—C11A	1.365 (8)	C10B—C11B	1.384 (8)
223	C10A—H10A	0.9300	C10B—H10B	0.9300
224	C11A—C12A	1.378 (8)	C11B—C12B	1.357 (8)
225	C11A—H11A	0.9300	C11B—H11B	0.9300
226	C12A—H12A	0.9300	C12B—H12B	0.9300
227	C13A—C14A	1.481 (8)	C13B—C14B	1.503 (7)
228	C13A—H13A	0.9700	C13B—H13C	0.9700
229	C13A—H13B	0.9700	C13B—H13D	0.9700
230	C14A—C15A	1.504 (8)	C14B—C15B	1.514 (7)
231	C14A—H14A	0.9700	C14B—H14C	0.9700
232	C14A—H14B	0.9700	C14B—H14D	0.9700
233				
234	C15A—O2A—H2A	107 (4)	C15B—O2B—H2B	102 (4)
235	C1A—N1A—N2A	125.5 (5)	C1B—N1B—N2B	125.6 (4)
236	C1A—N1A—C13A	120.9 (5)	C1B—N1B—C13B	121.2 (4)
237	N2A—N1A—C13A	113.5 (4)	N2B—N1B—C13B	112.4 (4)
238	C4A—N2A—N1A	119.2 (4)	C4B—N2B—N1B	118.6 (4)
239	O1A—C1A—N1A	121.5 (5)	O1B—C1B—N1B	119.5 (5)
240	O1A—C1A—C2A	124.3 (5)	O1B—C1B—C2B	124.2 (5)
241	N1A—C1A—C2A	114.2 (5)	N1B—C1B—C2B	116.2 (5)
242	C3A—C2A—C1A	117.4 (5)	C3B—C2B—C1B	117.4 (5)
243	C3A—C2A—H2A1	107.9	C3B—C2B—H2B1	108.0
244	C1A—C2A—H2A1	107.9	C1B—C2B—H2B1	108.0

245	C3A—C2A—H2A2	107.9	C3B—C2B—H2B2	108.0
246	C1A—C2A—H2A2	107.9	C1B—C2B—H2B2	108.0
247	H2A1—C2A—H2A2	107.2	H2B1—C2B—H2B2	107.2
248	C2A—C3A—C8A	120.7 (5)	C8B—C3B—C2B	120.4 (5)
249	C2A—C3A—C4A	111.5 (5)	C8B—C3B—C4B	114.2 (5)
250	C8A—C3A—C4A	112.6 (5)	C2B—C3B—C4B	112.9 (5)
251	C2A—C3A—H3A	103.2	C8B—C3B—H3B	101.9
252	C8A—C3A—H3A	103.2	C2B—C3B—H3B	101.9
253	C4A—C3A—H3A	103.2	C4B—C3B—H3B	101.9
254	N2A—C4A—C5A	119.0 (5)	N2B—C4B—C5B	117.1 (5)
255	N2A—C4A—C3A	121.2 (5)	N2B—C4B—C3B	123.2 (5)
256	C5A—C4A—C3A	119.6 (5)	C5B—C4B—C3B	119.7 (5)
257	C6A—C5A—C9A	118.3 (5)	C9B—C5B—C6B	118.4 (5)
258	C6A—C5A—C4A	121.7 (5)	C9B—C5B—C4B	121.7 (5)
259	C9A—C5A—C4A	119.9 (5)	C6B—C5B—C4B	119.9 (5)
260	C5A—C6A—C12A	120.4 (5)	C12B—C6B—C5B	119.1 (5)
261	C5A—C6A—C7A	119.2 (5)	C12B—C6B—C7B	119.6 (5)
262	C12A—C6A—C7A	120.4 (6)	C5B—C6B—C7B	121.3 (5)
263	C8A—C7A—C6A	114.2 (5)	C8B—C7B—C6B	113.1 (5)
264	C8A—C7A—H7A1	108.7	C8B—C7B—H7B1	109.0
265	C6A—C7A—H7A1	108.7	C6B—C7B—H7B1	109.0
266	C8A—C7A—H7A2	108.7	C8B—C7B—H7B2	109.0
267	C6A—C7A—H7A2	108.7	C6B—C7B—H7B2	109.0
268	H7A1—C7A—H7A2	107.6	H7B1—C7B—H7B2	107.8
269	C7A—C8A—C3A	116.2 (5)	C3B—C8B—C7B	118.4 (5)
270	C7A—C8A—H8A1	108.2	C3B—C8B—H8B1	107.7
271	C3A—C8A—H8A1	108.2	C7B—C8B—H8B1	107.7
272	C7A—C8A—H8A2	108.2	C3B—C8B—H8B2	107.7
273	C3A—C8A—H8A2	108.2	C7B—C8B—H8B2	107.7
274	H8A1—C8A—H8A2	107.4	H8B1—C8B—H8B2	107.1
275	C10A—C9A—C5A	120.6 (6)	C10B—C9B—C5B	122.4 (5)
276	C10A—C9A—H9A	119.7	C10B—C9B—H9B	118.8
277	C5A—C9A—H9A	119.7	C5B—C9B—H9B	118.8
278	C11A—C10A—C9A	120.5 (6)	C11B—C10B—C9B	117.6 (6)
279	C11A—C10A—H10A	119.7	C11B—C10B—H10B	121.2
280	C9A—C10A—H10A	119.7	C9B—C10B—H10B	121.2
281	C10A—C11A—C12A	119.4 (6)	C12B—C11B—C10B	121.4 (6)
282	C10A—C11A—H11A	120.3	C12B—C11B—H11B	119.3
283	C12A—C11A—H11A	120.3	C10B—C11B—H11B	119.3
284	C11A—C12A—C6A	120.8 (6)	C11B—C12B—C6B	121.0 (5)
285	C11A—C12A—H12A	119.6	C11B—C12B—H12B	119.5
286	C6A—C12A—H12A	119.6	C6B—C12B—H12B	119.5
287	N1A—C13A—C14A	110.2 (5)	N1B—C13B—C14B	110.8 (4)
288	N1A—C13A—H13A	109.6	N1B—C13B—H13C	109.5
289	C14A—C13A—H13A	109.6	C14B—C13B—H13C	109.5
290	N1A—C13A—H13B	109.6	N1B—C13B—H13D	109.5
291	C14A—C13A—H13B	109.6	C14B—C13B—H13D	109.5
292	H13A—C13A—H13B	108.1	H13C—C13B—H13D	108.1

C13A—C14A—C15A	111.9 (5)		C13B—C14B—C15B	111.7 (5)
C13A—C14A—H14A	109.2		C13B—C14B—H14C	109.3
C15A—C14A—H14A	109.2		C15B—C14B—H14C	109.3
C13A—C14A—H14B	109.2		C13B—C14B—H14D	109.3
C15A—C14A—H14B	109.2		C15B—C14B—H14D	109.3
H14A—C14A—H14B	107.9		H14C—C14B—H14D	107.9
O3A—C15A—O2A	123.1 (6)		O3B—C15B—O2B	123.7 (6)
O3A—C15A—C14A	124.0 (6)		O3B—C15B—C14B	122.9 (6)
O2A—C15A—C14A	112.9 (6)		O2B—C15B—C14B	113.3 (6)
C1A—N1A—N2A—C4A	−14.3 (8)		C1B—N1B—N2B—C4B	−10.7 (7)
C13A—N1A—N2A—C4A	169.3 (5)		C13B—N1B—N2B—C4B	179.9 (4)
N2A—N1A—C1A—O1A	−176.4 (5)		N2B—N1B—C1B—O1B	−175.2 (5)
C13A—N1A—C1A—O1A	−0.3 (9)		C13B—N1B—C1B—O1B	−6.7 (8)
N2A—N1A—C1A—C2A	2.0 (8)		N2B—N1B—C1B—C2B	3.0 (8)
C13A—N1A—C1A—C2A	178.1 (5)		C13B—N1B—C1B—C2B	171.5 (5)
O1A—C1A—C2A—C3A	−156.2 (7)		O1B—C1B—C2B—C3B	−164.7 (6)
N1A—C1A—C2A—C3A	25.5 (9)		N1B—C1B—C2B—C3B	17.2 (8)
C1A—C2A—C3A—C8A	−173.7 (6)		C1B—C2B—C3B—C8B	−167.9 (6)
C1A—C2A—C3A—C4A	−38.1 (9)		C1B—C2B—C3B—C4B	−28.0 (8)
N1A—N2A—C4A—C5A	−176.8 (5)		N1B—N2B—C4B—C5B	179.7 (4)
N1A—N2A—C4A—C3A	−1.5 (8)		N1B—N2B—C4B—C3B	−3.0 (7)
C2A—C3A—C4A—N2A	26.7 (8)		C8B—C3B—C4B—N2B	164.1 (5)
C8A—C3A—C4A—N2A	166.0 (5)		C2B—C3B—C4B—N2B	21.7 (8)
C2A—C3A—C4A—C5A	−158.1 (6)		C8B—C3B—C4B—C5B	−18.6 (8)
C8A—C3A—C4A—C5A	−18.7 (8)		C2B—C3B—C4B—C5B	−161.0 (5)
N2A—C4A—C5A—C6A	171.5 (5)		N2B—C4B—C5B—C9B	−5.4 (7)
C3A—C4A—C5A—C6A	−3.9 (8)		C3B—C4B—C5B—C9B	177.1 (5)
N2A—C4A—C5A—C9A	−6.1 (8)		N2B—C4B—C5B—C6B	173.8 (5)
C3A—C4A—C5A—C9A	178.6 (5)		C3B—C4B—C5B—C6B	−3.7 (7)
C9A—C5A—C6A—C12A	0.2 (8)		C9B—C5B—C6B—C12B	2.4 (8)
C4A—C5A—C6A—C12A	−177.4 (5)		C4B—C5B—C6B—C12B	−176.8 (5)
C9A—C5A—C6A—C7A	179.9 (5)		C9B—C5B—C6B—C7B	−176.4 (5)
C4A—C5A—C6A—C7A	2.4 (8)		C4B—C5B—C6B—C7B	4.4 (7)
C5A—C6A—C7A—C8A	22.2 (9)		C12B—C6B—C7B—C8B	−162.0 (5)
C12A—C6A—C7A—C8A	−158.0 (6)		C5B—C6B—C7B—C8B	16.8 (8)
C6A—C7A—C8A—C3A	−46.2 (9)		C2B—C3B—C8B—C7B	−178.9 (6)
C2A—C3A—C8A—C7A	179.2 (7)		C4B—C3B—C8B—C7B	41.7 (8)
C4A—C3A—C8A—C7A	44.0 (8)		C6B—C7B—C8B—C3B	−40.6 (8)
C6A—C5A—C9A—C10A	−0.4 (8)		C6B—C5B—C9B—C10B	−2.3 (8)
C4A—C5A—C9A—C10A	177.2 (5)		C4B—C5B—C9B—C10B	176.9 (5)
C5A—C9A—C10A—C11A	0.1 (9)		C5B—C9B—C10B—C11B	1.5 (8)
C9A—C10A—C11A—C12A	0.5 (10)		C9B—C10B—C11B—C12B	−0.8 (9)
C10A—C11A—C12A—C6A	−0.7 (10)		C10B—C11B—C12B—C6B	1.0 (9)
C5A—C6A—C12A—C11A	0.4 (10)		C5B—C6B—C12B—C11B	−1.8 (8)
C7A—C6A—C12A—C11A	−179.4 (6)		C7B—C6B—C12B—C11B	177.0 (5)
C1A—N1A—C13A—C14A	−101.3 (6)		C1B—N1B—C13B—C14B	89.7 (6)
N2A—N1A—C13A—C14A	75.3 (6)		N2B—N1B—C13B—C14B	−100.4 (5)

N1A—C13A—C14A—C15A	−176.6 (5)		N1B—C13B—C14B—C15B	179.8 (4)
C13A—C14A—C15A—O3A	−115.0 (7)		C13B—C14B—C15B—O3B	10.4 (9)
C13A—C14A—C15A—O2A	65.3 (7)		C13B—C14B—C15B—O2B	−170.1 (5)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2*A*—H2*A*···O1*B*	0.92 (7)	1.78 (7)	2.651 (6)	158 (6)
O2*B*—H2*B*···O1*A*	0.90 (6)	1.75 (6)	2.598 (7)	157 (5)

ORDER FORM

Acta Cryst E STRUCTURE REPORTS

ISSN 1600-5368

YOU WILL AUTOMATICALLY BE SENT DETAILS OF HOW TO DOWNLOAD
AN ELECTRONIC REPRINT OF YOUR PAPER, FREE OF CHARGE.
PRINTED REPRINTS MAY BE PURCHASED USING THIS FORM.

Please scan your order and send to checkin@iucr.org

INTERNATIONAL UNION OF CRYSTALLOGRAPHY
5 Abbey Square
Chester CH1 2HU, England.

VAT No. GB 161 9034 76

Article No.: E141985-HB7233

Title of article Crystal structure of 3-(3-oxo-2,3,4,4a,5,6-hexahydrobenzo[*h*]cinnolin-2-yl)propionic acid

Name Fiorella Meneghetti

Address Department of Pharmaceutical Sciences, University of Milano, via L. Mangiagalli, 25, 20133-Milano, Italy

E-mail address (for electronic reprints) fiorella.meneghetti@unimi.it

DIGITAL PRINTED REPRINTS

I wish to order . . . . . paid reprints

These reprints will be sent to the address given above. If the above address or e-mail address is not correct, please indicate an alternative:

PAYMENT

Charge for reprints . . . . . USD Total charge . . . . . USD

☐ A cheque for . . . . . USD payable to **INTERNATIONAL UNION OF CRYSTALLOGRAPHY** is enclosed

☐ An official purchase order made out to **INTERNATIONAL UNION OF CRYSTALLOGRAPHY** ☐ is enclosed ☐ will follow

Purchase order No.

☐ Please invoice me

☐ I wish to pay by credit card

Date | Signature

DIGITAL PRINTED REPRINTS

An electronic reprint is supplied free of charge.

Printed reprints without limit of number may be purchased at the prices given in the table below. The requirements of all joint authors, if any, and of their laboratories should be included in a single order, specifically ordered on the form overleaf. All orders for reprints must be submitted promptly.

Prices for reprints are given below in **United States dollars** and include postage.

	Size of paper (in printed pages)				
Number of reprints required	1–2	3–4	5–8	9–16	Additional 8's
50	184	268	372	560	246
100	278	402	556	842	370
150	368	534	740	1122	490
200	456	664	920	1400	610
Additional 50's	86	128	178	276	116

PAYMENT AND ORDERING

Cheques should be in **United States dollars** payable to **INTERNATIONAL UNION OF CRYSTALLOGRAPHY**. Official purchase orders should be made out to **INTERNATIONAL UNION OF CRYSTALLOGRAPHY**.

Orders should be returned by email to checkin@iucr.org

ENQUIRIES

Enquiries concerning reprints should be sent to support@iucr.org.