Acta Cryst E CRYSTALLOGRAPHIC COMMUNICATIONS

ISSN 2056-9890

Crystal structure and Hirshfeld surface analysis of 7-ethoxy-5-methyl-2-(pyridin-3-yl)-11,12-dihydro-5,11-methano[1,2,4]triazolo[1,5-c][1,3,5]benzoxadiazocine

Ercan Aydemir,[a,b] Sevgi Kansiz,[c]* Mustafa Kemal Gumus,[b] Nikolay Yu. Gorobets[d,e] and Necmi Dege[c]

[a]T.R. Ministry of Forestry and Water Affairs, 11th Regional Directorate, 55030, Ilkadım, Samsun, Turkey, [b]Artvin Coruh University, Science-Technology Research and Application Center, Artvin 08000, Turkey, [c]Ondokuz Mayıs University, Faculty of Arts and Sciences, Department of Physics, 55139, Kurupelit, Samsun, Turkey, [d]SSI "Institute for Single Crystals" of National Academy of Sciences of Ukraine, 60 Nauky Ave., Kharkiv 61072, Ukraine, and [e]V.N. Karazin Kharkiv National University, Svobody sq. 4, Kharkiv 61077, Ukraine. *Correspondence e-mail: sevgi.koroglu@omu.edu.tr

Received 30 January 2018
Accepted 13 February 2018

Edited by D.-J. Xu, Zhejiang University (Yuquan Campus), China

Keywords: crystal structure; Biginelli condensation; benzoxadiazocine; Hirshfeld surfaces.

CCDC reference: 1820439

Supporting information: this article has supporting information at journals.iucr.org/e

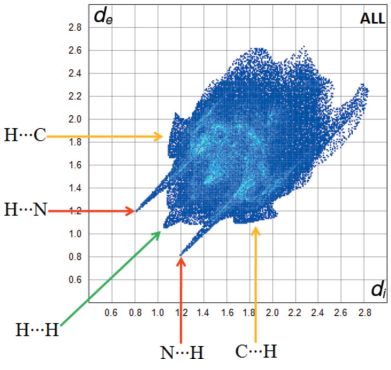
The title compound, $C_{19}H_{19}N_5O_2$, was prepared by the reaction of 3-amino-5-(pyridin-3-yl)-1,2,4-triazole with acetone and 2-hydroxy-3-ethoxybenzaldehyde. It crystallizes from ethanol in a tetragonal space group, with one molecule in the asymmetric unit. The 1,2,4-triazole five-membered ring is planar (maximum deviation = 0.0028 Å). The pyridine and phenyl rings are also planar with maximum deviations of 0.0091 and 0.0094 Å, respectively. In the crystal, N—H···N hydrogen bonds link the molecules into supramolecular chains propagating along the *c*-axis direction. Hirshfeld surface analysis and two-dimensional fingerprint plots have been used to analyse the intermolecular interactions present in the crystal.

1. Chemical context

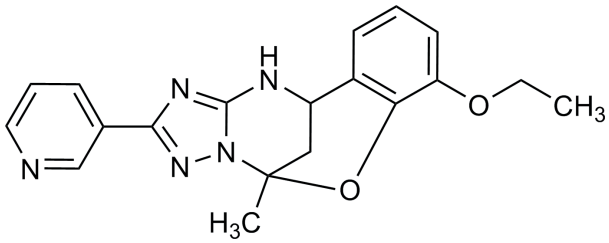
The title compound represents a conformationally restricted analogue of so-called Biginelli compounds known to exhibit multiple pharmacological activities. It was selected for a single-crystal X-ray analysis in order to probe the chemical and spatial requirements of some kinds of activity. 4-Aryl-3,4-dihydropyrimidine-2(1*H*)-ones and -thiones, known as Biginelli compounds, display a wide spectrum of significant pharmacological activities (Kappe, 2000). For example, these pyrimidine derivatives were assayed as antihypertensive agents, selective α_{1a}-adrenergic receptor antagonists, neuropeptide Y antagonists and were used as a lead for the development of anticancer drugs (Kappe, 2000). The Biginelli products have also been found to be potent hepatitis B replication inhibitors (Deres *et al.*, 2003).

Recently, the ability of oxygen-bridged azolopyrimidine derivatives to inhibit Eg5 activity has been examined (Svetlík *et al.*, 2010). As each of the above activities originates from stereo-selective binding of the drug molecule to its specific receptor, it is of interest to design a conformationally restricted probe molecule in order to examine geometric requirements of the given receptor binding site.

Since we had previously synthesized such a rigid type of oxygen-bridged triazolo-pyrimidine derivative, (I) (Gümüş *et al.*, 2017), we decided to examine the structure of this heterocyclic system by X-ray analysis. A novel Biginelli-like assembly of 3-amino-5-(pyridin-3-yl)-1,2,4-triazole with



acetone and 2-hydroxy-3-ethoxybenzaldehyde has been developed to enable easy access to 7-ethoxy-5-methyl-2-(pyridin-3-yl)-11,12-dihydro-5,11-methano[1,2,4]triazolo-[1,5-*c*][1,3,5]benzoxadiazocine compounds as representatives of a new class of heterocycles.



2. Structural commentary

The asymmetric unit of the title compound contains one independent molecule (Fig. 1). In the 1,2,4-triazole ring, the average C=N and C—N bond lengths are 1.324 and 1.355 Å, respectively, while the N—N bond is 1.389 (4) Å. These values consistent with literature values (Şen *et al.*, 2017*a*,*b*; Atalay *et al.*, 2004). The 1,2,4-triazole ring is planar with a maximum deviation of 0.0028 Å. The N1/C1–C5 and C12–C17 rings are also planar with maximum deviations of 0.0091 and 0.0094 Å, respectively. The dihedral angle between the N1/C1–C5 and C6/N2/N3/C7/N4 rings is 13.1 (2)°, while the latter ring is inclined to the N3/C10–C8/N5/C7 plane by 6.87 (15)°. The C12–C17 and N3/C10–C8/N5/C7 planes form dihedral angles of 7.8 (2) and 88.82 (12)°, respectively, with the C9/C10/O1/C12/C13 plane.

3. Supramolecular features

In the crystal, the N—H···N hydrogen bonds link the molecules, forming the supramolecular chains propagating along the *c*-axis direction (Table 1, Fig. 2).

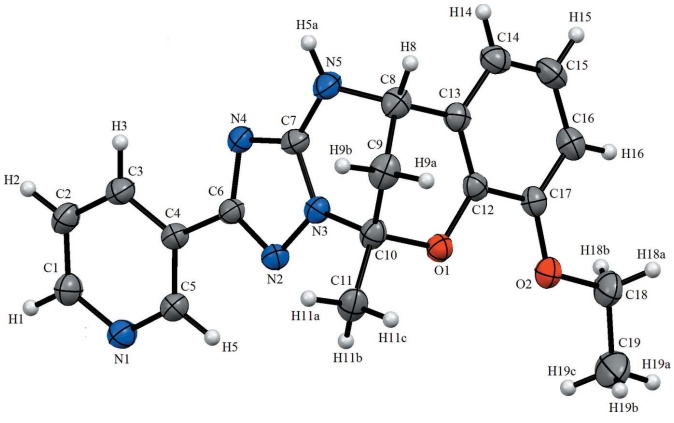


Figure 1
The molecular structure of the title compound, showing the atom labelling. Displacement ellipsoids are drawn at the 30% probability level.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N5—H5*A*···N1^{i}	0.86	2.13	2.907 (4)	149

Symmetry code: (i) $-x+\frac{1}{2}, -y+\frac{3}{2}, z-\frac{1}{2}$.

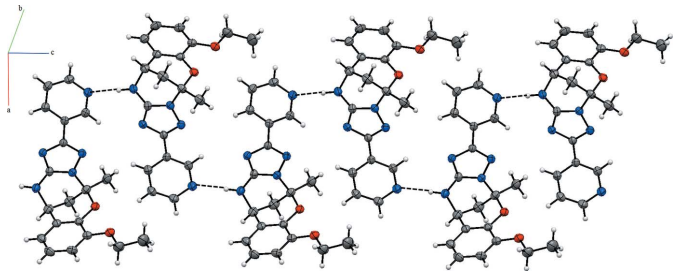


Figure 2
A partial view of the crystal packing of the title compound. Dashed lines denote the intermolecular N—H···N hydrogen bonds.

4. Hirshfeld surface analysis

Crystal Explorer17.5 (Turner *et al.*, 2017) was used to analyse the interactions in the crystal; fingerprint plots mapped over d_{norm} (Figs. 3 and 4) were generated. The molecular Hirshfeld surfaces were obtained using a standard (high) surface resolution with the three-dimentional d_{norm} surfaces mapped over a fixed colour scale of −0.484 (red) to 1.652 (blue). There are two red spots in the d_{norm} surface (Fig. 3), which are on the N-acceptor atoms involved in the interactions listed in Table 1. The red spots indicate the regions of donor–acceptor interactions (Kansiz *et al.*, 2018; Şen *et al.*, 2017*a*,*b*, 2018; Yaman *et al.*, 2018).

The intermolecular interactions of the title compound are shown in the 2D fingerprint plots shown in Fig. 5. The H···H interactions appear in the middle of the scattered points in the

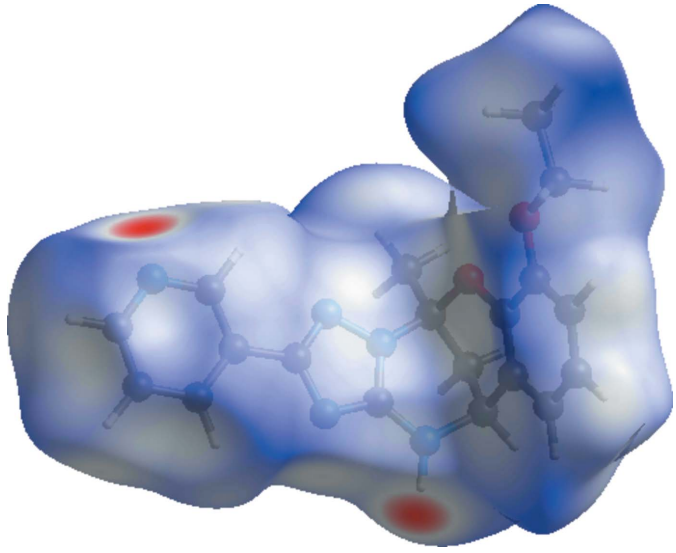


Figure 3
The Hirshfeld surface of $C_{19}H_{19}N_5O_2$ mapped with d_{norm}.

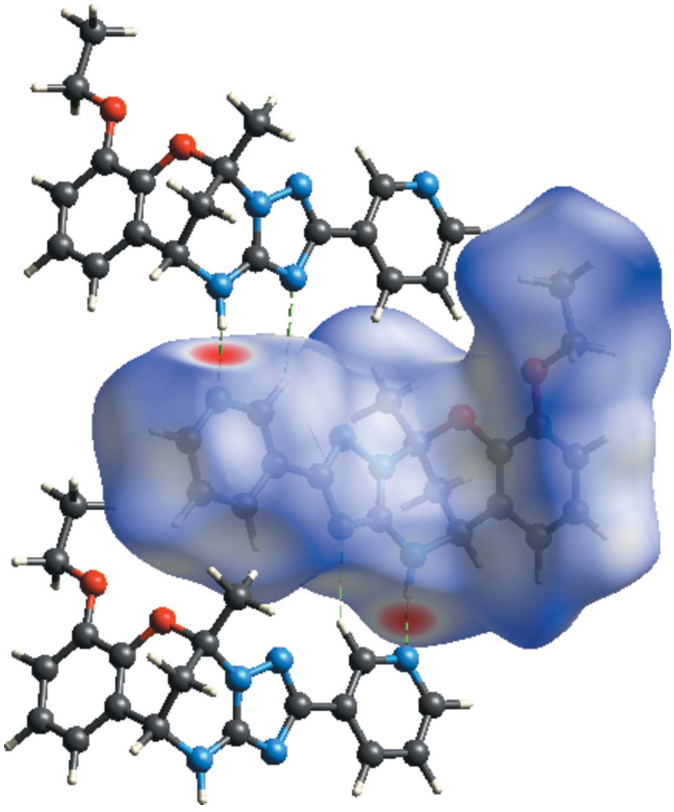


Figure 4
d_{norm} mapped on Hirshfeld surfaces to visualize the intermolecular interactions of $C_{19}H_{19}N_5O_2$.

two-dimensional fingerprint plots with a contribution to the overall Hirshfeld surface of 52.6% (Fig. 6). The contribution from the N···H/H···N contacts, corresponding to the N—H···N interaction, is represented by a pair of sharp spikes characteristic of a strong hydrogen-bond interaction (16.3%).

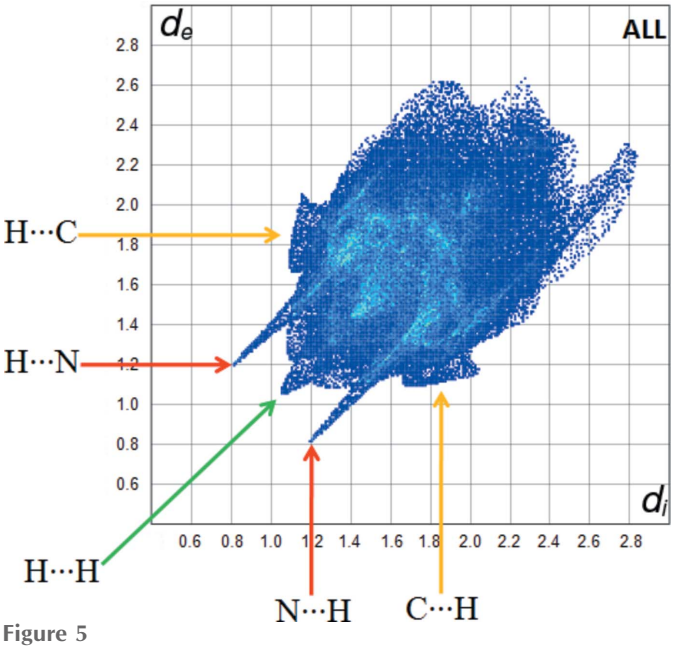


Figure 5
Fingerprint plot of the title compound.

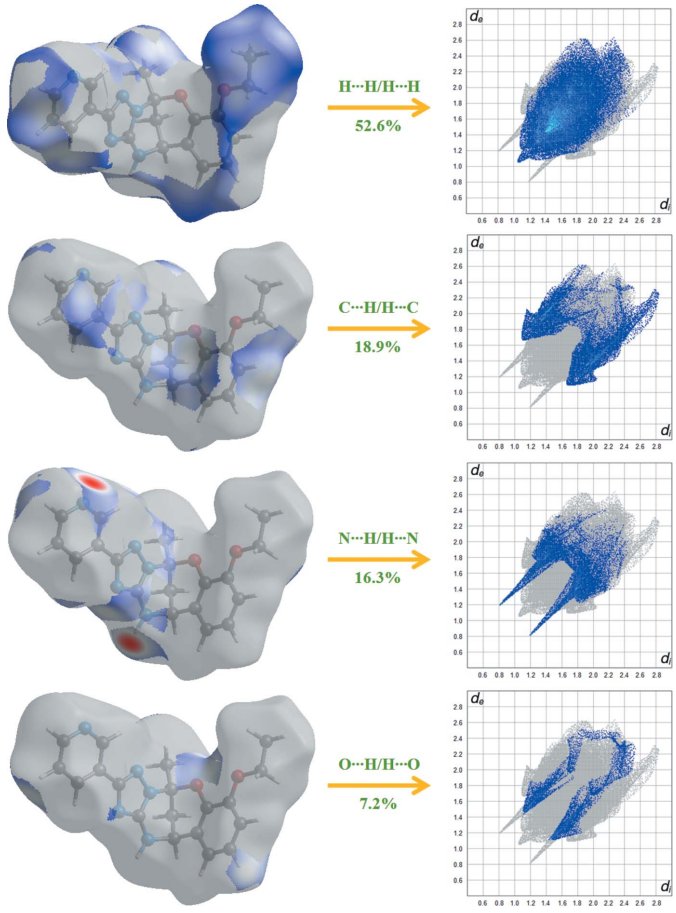


Figure 6
Two-dimensional fingerprint plots with a d_{norm} view of the H···H/H···H (52.6%), C···H/H···C (18.9%), N···H/H···N (16.3%) and O···H/H···O (7.2%) contacts in the title compound.

The whole fingerprint region and all other interactions, which are a combination of d_e and d_i, are displayed in Fig. 6.

5. Database survey

There are no direct precedents for the structure of (I) in the crystallographic literature (CSD Version 5.38; Groom *et al.*, 2016). However, there are several precedents for the triazolobenzoxadiazocines, including the structures of 5-(2-hydroxyphenyl)-7-methyl-4,5,6,7-tetrahydro[1,2,4]triazolo-[1,5-*a*]pyrimidin-7-ol (Gorobets *et al.*, 2010), ethyl 7-chloromethyl-5-(2-chlorophenyl)-7-hydroxy-2-methylsulfanyl-4,5,6,7-tetrahydro-1,2,4-triazolo[1,5-*a*]pyrimidine-6-carboxylate (Huang, 2009) and methyl 5′-(2-hydroxyphenyl)-5′,6′-dihydro-4′*H*-spiro[chromene-2,7′-[1,2,4]triazolo[1,5-*a*]pyrimidine]-3-carboxylate (Kettmann & Světlík, 2011).

6. Synthesis and crystallization

The synthesis of the title compound (Fig. 7) was described by Gümüş *et al.* (2017). 3-Amino-5-(pyridin-3-yl)-1,2,4-triazole(1.0 mmol), 2-hydroxy-3-ethoxybenzaldehyde (1.0 mmol), acetone (0.22 mL, 3.0 mmol), and abs. EtOH (2.0 mL) were

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{19}H_{19}N_5O_2$
M_r	349.39
Crystal system, space group	Tetragonal, $I\overline{4}$
Temperature (K)	293
a, c (Å)	17.1509 (8), 11.9033 (7)
V (Å^3)	3501.4 (4)
Z	8
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.09
Crystal size (mm)	0.54 × 0.34 × 0.16
Data collection	
Diffractometer	Stoe IPDS 2
Absorption correction	Integration (*X-RED32*; Stoe & Cie, 2002)
T_{min}, T_{max}	0.959, 0.984
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	8018, 3629, 2449
R_{int}	0.053
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.628
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.042, 0.088, 0.90
No. of reflections	3629
No. of parameters	236
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.15, −0.12
Absolute structure	Refined as an inversion twin.
Absolute structure parameter	−3 (2)

Computer programs: *X-AREA* and *X-RED* (Stoe & Cie, 2002), *SHELXL2017* (Sheldrick, 2008), *ORTEP-3 for Windows* and *WinGX* (Farrugia, 2012) and *PLATON* (Spek, 2009).

mixed in a microwave process vial, and then a 4 *N* solution of HCl in dioxane (0.07 mL, 0.3 mmol) was added. The mixture was irradiated at 423 K for 30 min. The reaction mixture was cooled by an air flow and stirred for 24 h at room temperature for complete precipitation of the product. The precipitate was filtered off, washed with EtOH (1.0 mL) and Et_2O (3 × 1.0 mL), and dried. Compound (I) was obtained in the form of a white solid. It was recrystallized from ethanol.

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. H atoms were positioned geometrically [N—H = 0.86 Å, C—H = 0.93 (aromatic), 0.96 (methyl) and 0.97 (methylene) Å] and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(N, C) and 1.5U_{eq}(methyl C).

Acknowledgements

The authors acknowledge the Faculty of Arts and Sciences, Ondokuz Mayıs University, Turkey, for the use of the Stoe IPDS 2 diffractometer (purchased under grant F.279 of the University Research Fund).

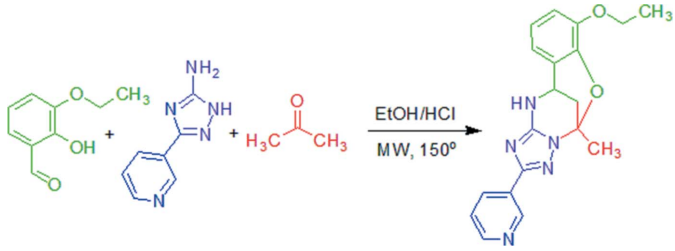


Figure 7
Synthesis of the title compound.

References

Atalay, Ş., Yavuz, M., Kahveci, B., Ağar, E. & Şaşmaz, S. (2004). *Acta Cryst.* E**60**, o2119–o2121.
Deres, K., Schröder, C. H., Paessens, A., Goldmann, S., Hacker, H. J., Weber, O., Krämer, T., Niewöhner, U., Pleiss, U., Stoltefuss, J., Graef, E., Koletzki, D., Masantschek, R. N. A., Reimann, A., Jaeger, R., Gross, R., Beckermann, B., Schlemmer, K. H., Haebich, D. & Rübsamen-Waigmann, H. (2003). *Science*, **299**, 893–896.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Gorobets, N. Y., Sedash, Y. V., Ostras, K. S., Zaremba, O. V., Shishkina, S. V., Baumer, V. N., Shishkin, O. V., Kovalenko, S. M., Desenko, S. M. & Van der Eycken, E. V. (2010). *Tetrahedron Lett.* **51**, 2095–2098.
Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* B**72**, 171–179.
Gümüş, M. K., Gorobets, N. Y., Sedash, Y. V., Chebanov, V. A. & Desenko, S. M. (2017). *Chem. Heterocycl. Compd*, **53**, 1261–1267.
Huang, S. (2009). *Acta Cryst.* E**65**, o2671.
Kansiz, S., Almarhoon, Z. M. & Dege, N. (2018). *Acta Cryst.* E**74**, 217–220.
Kappe, C. O. (2000). *Eur. J. Med. Chem.* **35**, 1043–1052.
Kettmann, V. & Světlík, J. (2011). *Acta Cryst.* E**67**, o92.
Şen, F., Çapan, İ., Dinçer, M. & Çukurovalı, A. (2018). *J. Mol. Struct.* **1155**, 278–287.
Şen, F., Dinçer, M., Yilmaz, I. & Cukurovali, A. (2017*a*). *J. Mol. Struct.* **1137**, 193–205.
Şen, F., Kansiz, S. & Uçar, İ. (2017*b*). *Acta Cryst.* C**73**, 517–524.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Stoe & Cie (2002). *IPDS 2 User Manual*. Stoe & Cie GmbH, Darmstadt, Germany.
Svetlík, J., Veizerová, L., Mayer, T. U. & Catarinella, M. (2010). *Bioorg. Med. Chem. Lett.* **20**, 4073–4076.
Turner, M. J., MacKinnon, J. J., Wolff, S. K., Grimwood, D. J., Spackman, P. R., Jayatilaka, D. & Spackman, M. A. (2017). *Crystal Explorer17.5*. University of Western Avustralia.
Yaman, M., Almarhoon, Z. M., Çakmak, Ş., Kütük, H., Meral, G. & Dege, N. (2018). *Acta Cryst.* E**74**, 41–44.

supporting information

Acta Cryst. (2018). E**74**, 367-370 [https://doi.org/10.1107/S2056989018002621]

Crystal structure and Hirshfeld surface analysis of 7-ethoxy-5-methyl-2-(pyridin-3-yl)-11,12-dihydro-5,11-methano[1,2,4]triazolo[1,5-*c*][1,3,5]benzoxadiazocine

Ercan Aydemir, Sevgi Kansiz, Mustafa Kemal Gumus, Nikolay Yu. Gorobets and Necmi Dege

Computing details

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA* (Stoe & Cie, 2002); data reduction: *X-RED* (Stoe & Cie, 2002); program(s) used to solve structure: *WinGX* (Farrugia, 2012); program(s) used to refine structure: *SHELXL2017* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *PLATON* (Spek, 2009).

7-Ethoxy-5-methyl-2-(pyridin-3-yl)-11,12-dihydro-5,11-methano-1,2,4]-triazolo[1,5-*c*][1,3,5]benzoxadiazocine

Crystal data

$C_{19}H_{19}N_5O_2$
$M_r = 349.39$
Tetragonal, $I\bar{4}$
$a = 17.1509$ (8) Å
$c = 11.9033$ (7) Å
$V = 3501.4$ (4) Å^3
$Z = 8$
$F(000) = 1472$
$D_x = 1.326$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 8727 reflections
$\theta = 1.7$–$27.6°$
$\mu = 0.09$ mm^{-1}
$T = 293$ K
Prism, colorless
0.54 × 0.34 × 0.16 mm

Data collection

Stoe IPDS 2 diffractometer
Radiation source: sealed X-ray tube, 12 x 0.4 mm long-fine focus
Detector resolution: 6.67 pixels mm^{-1}
rotation method scans
Absorption correction: integration (X-RED32; Stoe & Cie, 2002)
$T_{min} = 0.959$, $T_{max} = 0.984$
8018 measured reflections
3629 independent reflections
2449 reflections with $I > 2\sigma(I)$
$R_{int} = 0.053$
$\theta_{max} = 26.5°$, $\theta_{min} = 1.7°$
$h = -20\rightarrow21$
$k = -21\rightarrow21$
$l = -14\rightarrow13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.088$
$S = 0.90$
3629 reflections
236 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0372P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.15$ e Å^{-3}
$\Delta\rho_{min} = -0.12$ e Å^{-3}

Absolute structure: Refined as an inversion twin.

Absolute structure parameter: −3 (2)

Special details

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

Refinement. Refined as a 2-component inversion twin.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.0390 (2)	0.7259 (2)	0.4925 (3)	0.0581 (10)
H1	−0.014021	0.716332	0.501878	0.070*
C2	0.0662 (2)	0.7372 (2)	0.3863 (3)	0.0597 (10)
H2	0.032912	0.733641	0.324888	0.072*
C3	0.1444 (2)	0.7541 (2)	0.3716 (3)	0.0558 (9)
H3	0.164641	0.761427	0.299904	0.067*
C4	0.19186 (19)	0.76002 (19)	0.4641 (3)	0.0451 (8)
C5	0.1594 (2)	0.7450 (2)	0.5683 (3)	0.0524 (9)
H5	0.191757	0.746889	0.630959	0.063*
C6	0.27446 (19)	0.78337 (18)	0.4538 (3)	0.0433 (8)
C7	0.3820 (2)	0.81015 (19)	0.3807 (3)	0.0452 (8)
C8	0.5188 (2)	0.8326 (2)	0.3714 (3)	0.0530 (9)
H8	0.557656	0.853590	0.319160	0.064*
C9	0.5062 (2)	0.8898 (2)	0.4669 (4)	0.0575 (10)
H9A	0.555879	0.903724	0.500282	0.069*
H9B	0.481787	0.936913	0.438785	0.069*
C10	0.4545 (2)	0.85180 (19)	0.5536 (3)	0.0484 (8)
C11	0.4295 (2)	0.9041 (2)	0.6485 (4)	0.0620 (11)
H11A	0.401451	0.947929	0.618728	0.093*
H11B	0.396382	0.875548	0.698823	0.093*
H11C	0.474656	0.922184	0.688324	0.093*
C12	0.53691 (18)	0.74016 (18)	0.5322 (3)	0.0432 (8)
C13	0.54834 (18)	0.75667 (19)	0.4207 (3)	0.0460 (8)
C14	0.5883 (2)	0.7029 (2)	0.3536 (3)	0.0557 (10)
H14	0.596039	0.713003	0.277630	0.067*
C15	0.6162 (2)	0.6352 (2)	0.4004 (4)	0.0631 (11)
H15	0.642215	0.599352	0.355389	0.076*
C16	0.6062 (2)	0.6195 (2)	0.5133 (3)	0.0576 (10)
H16	0.625672	0.573495	0.543568	0.069*
C17	0.56737 (19)	0.6719 (2)	0.5813 (3)	0.0471 (8)
C18	0.5850 (3)	0.5943 (3)	0.7453 (4)	0.0716 (12)
H18A	0.641332	0.593704	0.738080	0.086*
H18B	0.564269	0.547848	0.709738	0.086*
C19	0.5622 (3)	0.5965 (3)	0.8665 (4)	0.1006 (17)
H19A	0.582492	0.551306	0.903941	0.151*

H19B	0.583133	0.642681	0.900739	0.151*
H19C	0.506407	0.597026	0.872521	0.151*
N1	0.08383 (17)	0.72774 (19)	0.5838 (3)	0.0572 (8)
N2	0.31602 (16)	0.80437 (15)	0.5418 (2)	0.0458 (7)
N3	0.38723 (15)	0.82148 (16)	0.4920 (2)	0.0442 (7)
N4	0.31108 (15)	0.78598 (17)	0.3519 (2)	0.0467 (7)
N5	0.44380 (16)	0.82375 (18)	0.3130 (2)	0.0542 (8)
H5A	0.439554	0.826988	0.241203	0.065*
O1	0.49588 (12)	0.78802 (13)	0.60565 (18)	0.0468 (6)
O2	0.55363 (14)	0.66252 (14)	0.6933 (2)	0.0567 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.045 (2)	0.071 (3)	0.058 (3)	−0.0032 (17)	−0.0068 (19)	−0.002 (2)
C2	0.055 (2)	0.077 (3)	0.048 (2)	−0.0014 (19)	−0.0144 (18)	−0.001 (2)
C3	0.055 (2)	0.070 (2)	0.042 (2)	0.0003 (18)	−0.0024 (18)	0.0043 (18)
C4	0.0452 (19)	0.051 (2)	0.0389 (19)	0.0027 (15)	−0.0019 (16)	−0.0025 (16)
C5	0.050 (2)	0.065 (2)	0.043 (2)	0.0048 (17)	−0.0047 (17)	−0.0016 (18)
C6	0.0474 (19)	0.0454 (19)	0.0370 (19)	0.0039 (14)	−0.0035 (16)	−0.0014 (16)
C7	0.052 (2)	0.046 (2)	0.037 (2)	0.0000 (16)	−0.0011 (17)	0.0026 (16)
C8	0.050 (2)	0.054 (2)	0.055 (2)	−0.0108 (17)	−0.0034 (18)	0.0068 (19)
C9	0.062 (2)	0.0416 (19)	0.069 (3)	−0.0079 (16)	−0.012 (2)	0.0020 (19)
C10	0.052 (2)	0.0433 (18)	0.050 (2)	0.0020 (15)	−0.0100 (17)	−0.0025 (17)
C11	0.070 (3)	0.051 (2)	0.064 (3)	0.0125 (18)	−0.020 (2)	−0.0181 (19)
C12	0.0350 (17)	0.0465 (19)	0.048 (2)	−0.0012 (14)	−0.0029 (15)	−0.0062 (17)
C13	0.0388 (19)	0.050 (2)	0.049 (2)	−0.0059 (15)	−0.0008 (16)	−0.0023 (17)
C14	0.048 (2)	0.066 (2)	0.054 (2)	−0.0040 (18)	0.0081 (18)	−0.0043 (19)
C15	0.056 (2)	0.065 (3)	0.068 (3)	0.0102 (19)	0.014 (2)	−0.013 (2)
C16	0.048 (2)	0.056 (2)	0.069 (3)	0.0077 (17)	0.0038 (19)	−0.001 (2)
C17	0.0395 (19)	0.048 (2)	0.053 (2)	0.0002 (15)	−0.0013 (16)	0.0001 (17)
C18	0.070 (3)	0.072 (3)	0.073 (3)	0.020 (2)	−0.002 (2)	0.020 (2)
C19	0.101 (4)	0.119 (4)	0.082 (3)	0.032 (3)	0.009 (3)	0.039 (3)
N1	0.0495 (18)	0.075 (2)	0.0473 (19)	−0.0042 (15)	−0.0009 (15)	0.0003 (16)
N2	0.0471 (17)	0.0498 (16)	0.0404 (17)	0.0034 (12)	−0.0027 (14)	−0.0044 (13)
N3	0.0414 (16)	0.0491 (16)	0.0422 (18)	−0.0011 (12)	−0.0038 (13)	−0.0030 (13)
N4	0.0436 (17)	0.0571 (18)	0.0393 (17)	0.0005 (14)	−0.0050 (13)	0.0033 (13)
N5	0.0486 (18)	0.073 (2)	0.0410 (16)	−0.0050 (15)	−0.0034 (14)	0.0104 (16)
O1	0.0494 (13)	0.0459 (13)	0.0451 (13)	0.0082 (10)	−0.0054 (11)	−0.0044 (11)
O2	0.0557 (15)	0.0585 (16)	0.0559 (16)	0.0140 (12)	−0.0017 (13)	0.0087 (13)

Geometric parameters (Å, º)

C1—N1	1.331 (5)	C10—C11	1.504 (5)
C1—C2	1.362 (5)	C11—H11A	0.9600
C1—H1	0.9300	C11—H11B	0.9600
C2—C3	1.384 (5)	C11—H11C	0.9600
C2—H2	0.9300	C12—C13	1.372 (5)

C3—C4	1.373 (5)	C12—O1	1.390 (4)
C3—H3	0.9300	C12—C17	1.409 (5)
C4—C5	1.383 (5)	C13—C14	1.399 (5)
C4—C6	1.477 (4)	C14—C15	1.374 (5)
C5—N1	1.343 (4)	C14—H14	0.9300
C5—H5	0.9300	C15—C16	1.380 (6)
C6—N2	1.317 (4)	C15—H15	0.9300
C6—N4	1.367 (4)	C16—C17	1.380 (5)
C7—N4	1.330 (4)	C16—H16	0.9300
C7—N3	1.342 (4)	C17—O2	1.364 (4)
C7—N5	1.352 (4)	C18—O2	1.428 (4)
C8—N5	1.469 (4)	C18—C19	1.495 (6)
C8—C13	1.515 (5)	C18—H18A	0.9700
C8—C9	1.517 (5)	C18—H18B	0.9700
C8—H8	0.9800	C19—H19A	0.9600
C9—C10	1.509 (5)	C19—H19B	0.9600
C9—H9A	0.9700	C19—H19C	0.9600
C9—H9B	0.9700	N2—N3	1.389 (4)
C10—O1	1.443 (4)	N5—H5A	0.8600
C10—N3	1.463 (4)		

N1—C1—C2	123.7 (3)	H11A—C11—H11C	109.5
N1—C1—H1	118.1	H11B—C11—H11C	109.5
C2—C1—H1	118.1	C13—C12—O1	124.0 (3)
C1—C2—C3	118.7 (4)	C13—C12—C17	121.3 (3)
C1—C2—H2	120.7	O1—C12—C17	114.7 (3)
C3—C2—H2	120.7	C12—C13—C14	119.1 (3)
C4—C3—C2	119.2 (3)	C12—C13—C8	120.3 (3)
C4—C3—H3	120.4	C14—C13—C8	120.6 (3)
C2—C3—H3	120.4	C15—C14—C13	119.7 (4)
C3—C4—C5	117.8 (3)	C15—C14—H14	120.1
C3—C4—C6	121.4 (3)	C13—C14—H14	120.1
C5—C4—C6	120.7 (3)	C14—C15—C16	121.1 (4)
N1—C5—C4	123.5 (3)	C14—C15—H15	119.5
N1—C5—H5	118.2	C16—C15—H15	119.5
C4—C5—H5	118.2	C17—C16—C15	120.3 (4)
N2—C6—N4	116.6 (3)	C17—C16—H16	119.9
N2—C6—C4	121.8 (3)	C15—C16—H16	119.9
N4—C6—C4	121.5 (3)	O2—C17—C16	125.5 (3)
N4—C7—N3	111.2 (3)	O2—C17—C12	116.0 (3)
N4—C7—N5	128.1 (3)	C16—C17—C12	118.5 (3)
N3—C7—N5	120.7 (3)	O2—C18—C19	107.4 (4)
N5—C8—C13	112.8 (3)	O2—C18—H18A	110.2
N5—C8—C9	107.2 (3)	C19—C18—H18A	110.2
C13—C8—C9	108.2 (3)	O2—C18—H18B	110.2
N5—C8—H8	109.5	C19—C18—H18B	110.2
C13—C8—H8	109.5	H18A—C18—H18B	108.5
C9—C8—H8	109.5	C18—C19—H19A	109.5

C10—C9—C8	108.5 (3)	C18—C19—H19B	109.5
C10—C9—H9A	110.0	H19A—C19—H19B	109.5
C8—C9—H9A	110.0	C18—C19—H19C	109.5
C10—C9—H9B	110.0	H19A—C19—H19C	109.5
C8—C9—H9B	110.0	H19B—C19—H19C	109.5
H9A—C9—H9B	108.4	C1—N1—C5	116.8 (3)
O1—C10—N3	109.5 (3)	C6—N2—N3	101.2 (3)
O1—C10—C11	105.7 (3)	C7—N3—N2	109.4 (3)
N3—C10—C11	111.3 (3)	C7—N3—C10	126.8 (3)
O1—C10—C9	109.4 (3)	N2—N3—C10	123.7 (3)
N3—C10—C9	105.9 (3)	C7—N4—C6	101.6 (3)
C11—C10—C9	115.1 (3)	C7—N5—C8	115.0 (3)
C10—C11—H11A	109.5	C7—N5—H5A	122.5
C10—C11—H11B	109.5	C8—N5—H5A	122.5
H11A—C11—H11B	109.5	C12—O1—C10	115.3 (2)
C10—C11—H11C	109.5	C17—O2—C18	117.1 (3)

N1—C1—C2—C3	−2.2 (6)	C2—C1—N1—C5	2.8 (6)
C1—C2—C3—C4	−0.9 (6)	C4—C5—N1—C1	−0.4 (5)
C2—C3—C4—C5	3.0 (5)	N4—C6—N2—N3	−0.8 (3)
C2—C3—C4—C6	−175.7 (3)	C4—C6—N2—N3	−179.3 (3)
C3—C4—C5—N1	−2.5 (5)	N4—C7—N3—N2	−0.2 (4)
C6—C4—C5—N1	176.2 (3)	N5—C7—N3—N2	179.3 (3)
C3—C4—C6—N2	166.1 (3)	N4—C7—N3—C10	−176.5 (3)
C5—C4—C6—N2	−12.5 (5)	N5—C7—N3—C10	3.0 (5)
C3—C4—C6—N4	−12.4 (5)	C6—N2—N3—C7	0.5 (3)
C5—C4—C6—N4	169.0 (3)	C6—N2—N3—C10	177.0 (3)
N5—C8—C9—C10	68.7 (3)	O1—C10—N3—C7	−101.2 (4)
C13—C8—C9—C10	−53.3 (4)	C11—C10—N3—C7	142.3 (4)
C8—C9—C10—O1	67.4 (3)	C9—C10—N3—C7	16.6 (4)
C8—C9—C10—N3	−50.5 (3)	O1—C10—N3—N2	83.0 (4)
C8—C9—C10—C11	−173.8 (3)	C11—C10—N3—N2	−33.5 (4)
O1—C12—C13—C14	−177.8 (3)	C9—C10—N3—N2	−159.2 (3)
C17—C12—C13—C14	2.6 (5)	N3—C7—N4—C6	−0.2 (4)
O1—C12—C13—C8	2.9 (5)	N5—C7—N4—C6	−179.7 (3)
C17—C12—C13—C8	−176.7 (3)	N2—C6—N4—C7	0.7 (4)
N5—C8—C13—C12	−98.3 (4)	C4—C6—N4—C7	179.2 (3)
C9—C8—C13—C12	20.1 (4)	N4—C7—N5—C8	−166.6 (3)
N5—C8—C13—C14	82.4 (4)	N3—C7—N5—C8	14.0 (5)
C9—C8—C13—C14	−159.2 (3)	C13—C8—N5—C7	70.4 (4)
C12—C13—C14—C15	−0.7 (5)	C9—C8—N5—C7	−48.7 (4)
C8—C13—C14—C15	178.6 (3)	C13—C12—O1—C10	9.4 (4)
C13—C14—C15—C16	−0.7 (6)	C17—C12—O1—C10	−171.0 (3)
C14—C15—C16—C17	0.3 (6)	N3—C10—O1—C12	71.7 (3)
C15—C16—C17—O2	179.6 (4)	C11—C10—O1—C12	−168.4 (3)
C15—C16—C17—C12	1.5 (5)	C9—C10—O1—C12	−43.9 (4)
C13—C12—C17—O2	178.7 (3)	C16—C17—O2—C18	2.8 (5)
O1—C12—C17—O2	−0.9 (4)	C12—C17—O2—C18	−179.0 (3)

C13—C12—C17—C16	−3.0 (5)	C19—C18—O2—C17	−179.6 (4)
O1—C12—C17—C16	177.4 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N5—H5*A*···N1^{i}	0.86	2.13	2.907 (4)	149

Symmetry code: (i) −*x*+1/2, −*y*+3/2, *z*−1/2.