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Crystal structure and molecular docking study of diethyl 2,2′-({[(1*E*,1′*E*)-(hydrazine-1,2-diylidene)-bis(methanylylidene)]bis(4,1-phenylene)}bis(oxy))-diacetate

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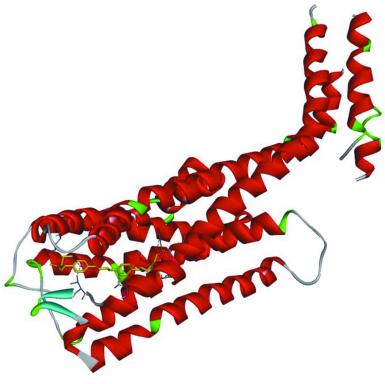
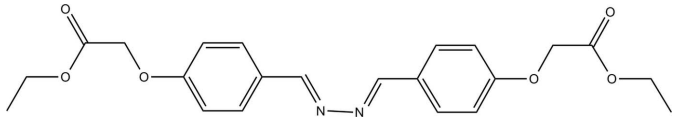
The title Schiff base, $C_{22}H_{24}N_2O_6$, adopts an *E* configuration. The molecule is planar, the mean planes of the phenyl ring system (r.m.s deviation = 0.0059 Å) forms a dihedral angle of 0.96 (4)° with the mean plane of the phenyl ring moiety (r.m.s deviation = 0.0076 Å). In the crystal, molecules are linked by weak intermolecular C—H···O and C—H···N hydrogen bonds into chains extending along the *c*-axis and *b*-axis directions, respectively. A molecular docking study between the title molecule and 5-HT2C, which is a G protein receptor and ligand-gated ion channels found in nervous systems (PDB ID: 6BQH) was executed. The experiment shows that it is a good potential agent because of its affinity and ability to stick to the active sites of the receptor.

1. Chemical context

Compounds with an azomethine group (–C═N–) are known as Schiff bases, which are usually synthesized from the condensation of active carbonyl groups and primary amines (Yang *et al.*, 2001). Furthermore, these derivatives represent an important class of organic compounds, especially in the medicinal and pharmaceutical fields (Murtaza *et al.*, 2014). It is well known from the literature that Schiff bases display excellent biological properties, such as antioxidant and analgesic (Karrouchi *et al.*, 2016), antibacterial and cytotoxic (Maaref *et al.*, 2020), antidiabetic (Karrouchi *et al.*, 2022) and anti-inflammatory activities (Rana *et al.*, 2012). These derivatives are also used as corrosion inhibitors, which relies on their ability to spontaneously form a monolayer on the surface being protected (El Arrouji *et al.*, 2020). In this study, the title compound, diethyl 2,2′-({[(1*E*,1′*E*)-(hydrazine-1,2-diylidene)-bis(methanylylidene)]bis(4,1-phenylene)}bis(oxy))diacetate, was characterized by single crystal X-ray and studied by Hirshfeld surface analysis.





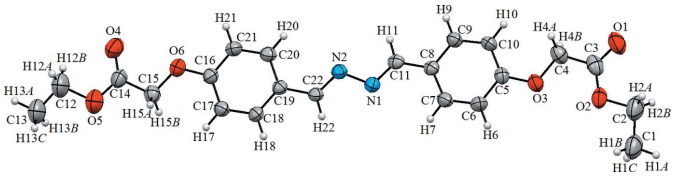


Figure 1
The molecular structure of the title compound, with atom labelling. Displacement ellipsoids are drawn at the 40% probability level.

2. Structural commentary

The molecular structure of the title compound is illustrated in Fig. 1. The asymmetric unit contains one independent molecule, which is planar, the mean plane of the C5–C10 phenyl ring (r.m.s deviation = 0.006 Å) forms a dihedral angle of 0.96 (4)° with the mean plane of the C16–C20 phenyl ring (r.m.s deviation = 0.008 Å). The C3—O1 and C14—O4 bond lengths in the molecule are 1.213 (8) and 1.212 (8) Å, respectively, while the C11—N1 and C22—N2 bond lengths are 1.274 (7) and 1.275 (7) Å, respectively (Table 1). These results suggest a double-bond character for the C=O and C=N bonds. The N1—N2 bond distance, 1.419 (7) Å, is compatible with 1.411 Å (Manawar *et al.*, 2019; Kansiz *et al.*, 2021). These results suggest a single bond character for N—N, as expected from hydrazine structures. The exocyclic angles C4—C3—O2 [115.4 (6)°], O1—C3—O2 [125.4 (8)°], C15—C14—O4 [125.5 (7)°] and C15—C14—O5 [111.9 (6)°] deviate significantly from the normal value of 120°; this may be due to steric repulsion (H4*A*···H10 = 2.22 Å and H15*B*···H17 = 2.32 Å). Bond lengths and angles are within normal ranges and are comparable to those observed in related structures (see *Database survey* section).

3. Supramolecular features

In the crystal, there are two intermolecular hydrogen bonds. The C6—H6···O4^{i} hydrogen bond links the molecules to each other along the *c*-axis direction while the C4—H4*B*···N1^{ii} hydrogen bond links the molecules to each other along the *b*-axis direction (symmetry codes as in Table 1). A view of the crystal packing is shown in Fig. 2.

4. Database survey

A search of the Cambridge Structural Database (CSD, version 5.42, update of May 2021; Groom *et al.*, 2016) for the ethyl 2-(*p*-tolyloxy)acetate skeleton revealed seven similar compounds, *viz.*: ethyl 4-[1-(4-bromophenyl)-3-methyl-5-oxo-4,5-dihydro-1*H*-1,2,4-triazol-4-yliminomethyl]phenoxyacetate (EKEYEY; Thamotharan *et al.*, 2003), di[3-fluoro-6-methoxy-4-(ethoxycarbonylmethoxy)benzyl] ether (HIGLEP; Wallner *et al.*, 2007), ethyl (2-fluoro-4-hydroxymethyl-5-methoxyphenoxy)acetate (HIGLIT; Wallner *et al.*, 2007), diethyl 3,3-bis{3-[4-(2-ethoxy-2-oxoethoxy)-3-methoxyphenyl]acryloyl}-pentanedioate (JUMJEI; Xu *et al.*, 2015), ethyl (4-{3-[2,4-bis(2-ethoxy-2-oxoethoxy)phenyl]-3-oxoprop-1-en-1-yl}phenoxy)acetate (PIXWAW; Liu, 2014), ethyl [(2-oxo-2*H*-chromen-7-yl)oxy]acetate (WIHDEY; Fun *et al.*, 2013) and ethyl {4-[(*E*)-2-(3,4,5-trimethoxyphenyl)vinyl]phenoxy}acetate (XEWZIJ; Baolin *et al.*, 2007). In EKEYEY, the ethoxycarbonylmethoxy group is oriented at an angle of 29.42 (15)° with respect to the mean plane of the benzene ring. The mean plane of the 2*H*-chromene ring system (O1/C1–C9, r.m.s deviation = 0.026 Å) forms a dihedral angle of 81.71 (6)° with the mean plane of ethyl 2-hydroxyacetate moiety (O1/N3/C9/C10, r.m.s deviation = 0.026 Å) in WIHDEY. This dihedral angle for the title compound is smaller than in both EKEYEY and WIHDEY with a value of 4.38 (8)°. The C10—C11 bond distance of 1.516 (2) Å in WIHDEY, corresponding to a single bond, is slightly longer than observed for the title compound [C3—C4 = 1.498 (10) Å]. This bond length is also longer than in XEWZIJ [C18–C19 = 1.493 (3) Å; Baolin *et al.*, 2007)].

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···O4^{i}	0.93	2.57	3.483 (9)	169
C4—H4*B*···N1^{ii}	0.97	2.69	3.618 (10)	161

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

5. Molecular docking study

Molecular docking is a substantial process for finding the interactions between small molecules and macromolecules. Intermolecular bonds that occur between ligand and receptor are indicated by molecular docking. In this study, *AutoDockVina* (Trott & Olson, 2010) was used for predictive binding sites between the title molecule and the 5-HT2C receptor (Peng *et al.*, 2018). 6BQH is a serotonin receptor, which can be efficient for designing drugs to treat ailments

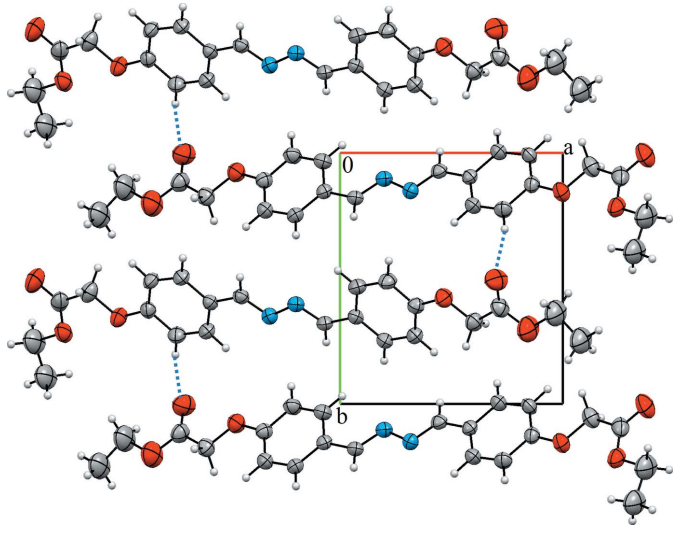


Figure 2
The crystal packing of the title compound with the intermolecular C—H···O hydrogen bonds shown as dashed lines.

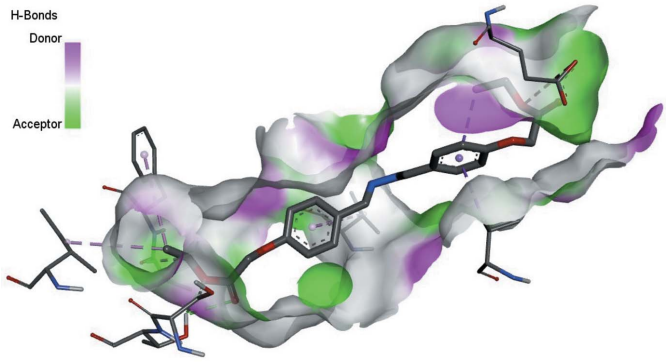


Figure 3
Three-dimensional visual of the intermolecular interactions for the best binding pose of the title compound docking with 6BQH.

such as anxiety, aggression, sleep disorders, and other psychological diseases. The three-dimensional structure of 6BQH was taken from the Protein Data Bank (PDB). Before the docking calculations, the receptor must be prepared for efficient insertion. For this reason, all water and ligand molecules were cleared on receptor active sites. According to these active sites, grid box dimensions were defined as 100 x 80 x 110 Å. In addition, $-x$, y, z centres were adjusted to be −40.569, 33.142, 45.392, respectively, and then the 5-HT2C receptor was saved in PDBQT format for the calculations. In the next step, rotatable angles for the coupling structure were determined and recorded in PDBQT format. *Discovery Studio Visualizer* (Biovia, 2017) was used for observations and preparations. All docking calculations were calculated with *AutoDockVina*. Twenty variable links were decided by *AutoDockVina* for the ligands connected to the receptor of the protein. The best affinity energy was observed in the first calculation, which is −6.2 kcal mol^{-1}. The bonding type of interaction is represented in Fig. 3. The 2D and 3D visuals of the intermolecular interactions for the best binding pose of the title compound docked into macromolecule 6BQH can be seen in Fig. 4. In addition, docking conformation can be seen in Fig. 5. Consequently, the title compound could be a possible molecule for drug design to treat psychological disorders, because its ability is suitable to stick to active sites of the receptor.

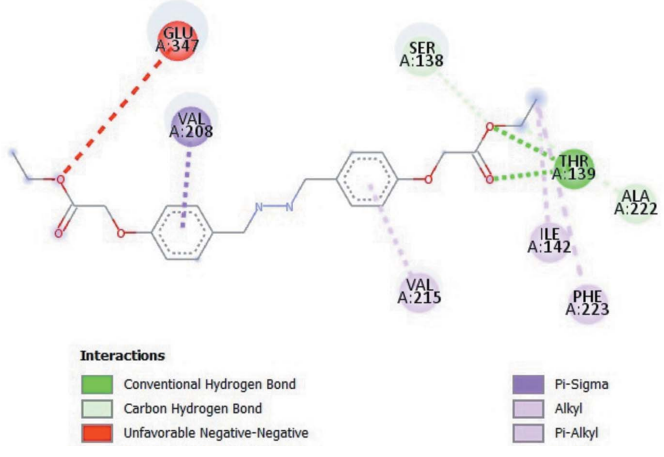


Figure 4
Two-dimensional visual of the intermolecular interactions for the best binding pose of the title compound docking with 6BQH.

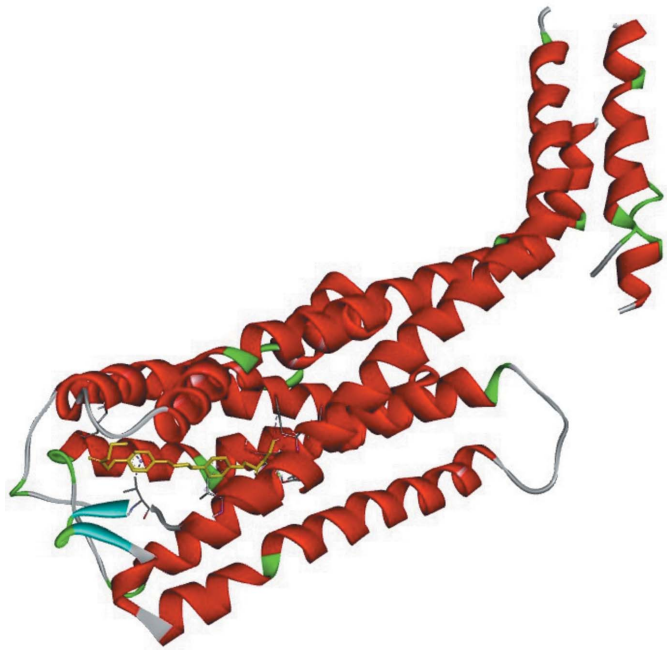


Figure 5
Three-dimensional conformation of the title compound with 6BQH.

6. Synthesis and crystallization

Hydrazine hydrate (0.013 g, 0.24 mmol) was added dropwise to a solution of ethyl 2-(4-formylphenoxy)acetate (0.5 g, 0.48 mmol) in ethanol (20 ml), and the mixture was refluxed for 4 h. After cooling, the solvent was removed under reduced pressure, and the residue was purified by recrystallization from ethanol to afford single crystals (yield 80%).

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. C-bound H atoms were positioned geometrically and refined using a riding model with C—H = 0.93–0.97 Å and U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms, and U_{iso}(H) = 1.2U_{eq}(C) for all other H atoms. The crystal studied was refined as a two-component inversion twin, but the absolute structure was indeterminate.

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Author contributions are as follows. Conceptualization, SD, SK, and KK; synthesis, SD and KK; writing (review and editing of the manuscript) SD, SK, FAA and KK; formal analysis, SD, KK and NB; crystal-structure determination, KK, SK and ND; validation, KK, ES and NB; project administration, KK, SD, ES and SK; molecular docking, FAA.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{22}H_{24}N_2O_6$
M_r	412.43
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	296
a, b, c (Å)	8.1864 (4), 9.2061 (5), 27.7903 (18)
V (Å^3)	2094.4 (2)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.10
Crystal size (mm)	0.68 × 0.44 × 0.22
Data collection	
Diffractometer	Stoe IPDS 2
Absorption correction	Integration (*X-RED32*; Stoe & Cie, 2002)
T_{min}, T_{max}	0.945, 0.979
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	11156, 4091, 2453
R_{int}	0.037
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.617
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.073, 0.246, 1.01
No. of reflections	4091
No. of parameters	254
No. of restraints	2
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.50, −0.67
Absolute structure	Refined as an inversion twin, but the absolute structure was indeterminate
Absolute structure parameter	−1 (4)

Computer programs: *X-AREA* and *X-RED* (Stoe & Cie, 2002), *SHELXT2017/1* (Sheldrick, 2015*a*), *SHELXL2017/1* (Sheldrick, 2015*b*), *PLATON* (Spek, 2020) and *WinGX* (Farrugia, 2012).

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References

Baolin, L., Jian, G., Xiquan, Z., Yitian, L. & Huaiming, H. (2007). *Z. Naturforsch. B: Chem. Sci.* **62**, 244–248.

Biovia (2017). *Discovery Studio Visualizer*. Vol. 936. Biovia, San Diego, CA, USA.

El Arrouji, S., Karrouchi, K., Berisha, A., Alaoui, K. I., Warad, I., Rais, Z., Radi, S., Taleb, M., Ansar, M. & Zarrouk, A. (2020). *Colloids Surf. A Physicochem. Eng. Asp.* **604**, 125325.

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

Fun, H.-K., Quah, C. K., Aich, K., Das, S. & Goswami, S. (2013). *Acta Cryst.* E**69**, o502.

Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* B**72**, 171–179.

Kansiz, S., Tatlidil, D., Dege, N., Aktas, F. A., Al-Asbahy, S. O. M. & Alaman Agar, A. (2021). *Acta Cryst.* E**77**, 658–662.

Karrouchi, K., Chemlal, L., Taoufik, J., Cherrah, Y., Radi, S., El Abbes Faouzi, M. & Ansar, M. (2016). *Annales Pharmaceutiques Françaises*, **74**, 431–438.

Karrouchi, K., Fettach, S., Tamer, Ö., Avci, D., Başoğlu, A., Atalay, Y., Ayaz, Z., Radi, S., Ghabbour, H. A., Mabkhot, Y. N., Faouzi, M. E. A. & Ansar, M. (2022). *J. Mol. Struct.* **1248**, 131506.

Liu, X. (2014). *Acta Cryst.* E**70**, o51.

Maaref, H., Sheikhhosseini, E., Foroughi, M. M., Akhgar, M. R. & Jahani, S. (2020). *Appl. Organomet. Chem.* **34**, e5557.

Manawar, R. B., Mamtora, M. J., Shah, M. K., Jotani, M. M. & Tiekink, E. R. T. (2019). *Acta Cryst.* E**76**, 53–61.

Murtaza, G., Mumtaz, A., Khan, F. A., Ahmad, S., Azhar, S., Khan, S. A., Najam-Ul-Haq, M., Atif, M., Khan, S. A., Maalik, A., Azhar, S. & Murtaza, G. (2014). *Acta Pol. Pharm.* **71**, 531–535.

Peng, Y., McCorvy, J. D., Harpsøe, K., Lansu, K., Yuan, S., Popov, P., Qu, L., Pu, M., Che, T., Nikolajsen, L. F., Huang, X.-P., Wu, Y., Shen, L., Bjørn-Yoshimoto, W. E., Ding, K., Wacker, D., Han, G. W., Cheng, J., Katritch, V., Jensen, A. A., Hanson, M. A., Zhao, S., Gloriam, D. E., Roth, B. L., Stevens, R. C. & Liu, Z.-J. (2018). *Cell*, **172**, 719–730.

Rana, K., Pandurangan, A., Singh, N. & Tiwari, A. K. (2012). *Int. J. Curr. Pharm. Res.* **4**, 5–11.

Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.

Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.

Spek, A. L. (2020). *Acta Cryst.* E**76**, 1–11.

Stoe & Cie (2002). *X-AREA* and *X-RED32*. Stoe & Cie GmbH, Darmstadt, Germany.

Thamotharan, S., Parthasarathi, V., Sunagar, V., Badami, B. & Schenk, K. J. (2003). *Acta Cryst.* E**59**, o1272–o1274.

Trott, O. & Olson, A. J. (2010). *J. Comput. Chem.* **31**, 455–461.

Wallner, F. K., Spjut, S., Boström, D. & Elofsson, M. (2007). *Org. Biomol. Chem.* **5**, 2464–2471.

Xu, G., Wang, J., Si, G., Wang, M., Wu, B. & Zhou, S. (2015). *Dyes Pigments*, **123**, 267–273.

Yang, Z. H., Wang, L. X., Zhou, Z. H., Zhou, Q. L. & Tang, C. C. (2001). *Tetrahedron Asymmetry*, **12**, 1579–1582.

supporting information

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Crystal structure and molecular docking study of diethyl 2,2′-({[(1*E*,1′*E*)-(hydrazine-1,2-diylidene)bis(methanylylidene)]bis(4,1-phenylene)}bis(oxy))diacetate

Said Daoui, Sevgi Kansiz, Feyzi Alkim Aktas, Necmi Dege, Eiad Saif, Noureddine Benchat and Khalid Karrouchi

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: *SHELXT2017/1* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2017/1* (Sheldrick, 2015b); molecular graphics: *PLATON* (Spek, 2020); software used to prepare material for publication: *WinGX* (Farrugia, 2012).

Diethyl 2,2′-({[(1*E*,1′*E*)-(hydrazine-1,2-diylidene)bis(methanylylidene)]bis(4,1-phenylene)}bis(oxy))diacetate

Crystal data

$C_{22}H_{24}N_2O_6$
M_r = 412.43
Orthorhombic, $P2_12_12_1$
a = 8.1864 (4) Å
b = 9.2061 (5) Å
c = 27.7903 (18) Å
V = 2094.4 (2) Å^3
Z = 4
$F(000)$ = 872
D_x = 1.308 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9754 reflections
θ = 2.2–27.8°
μ = 0.10 mm^{-1}
T = 296 K
Prism, colorless
0.68 × 0.44 × 0.22 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} = 0.945, T_{max} = 0.979
11156 measured reflections
4091 independent reflections
2453 reflections with $I > 2\sigma(I)$
R_{int} = 0.037
θ_{max} = 26.0°, θ_{min} = 2.3°
h = −8→10
k = −11→11
l = −27→34

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.073
$wR(F^2)$ = 0.246
S = 1.00
4091 reflections
254 parameters
2 restraints
Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1576P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.50$ e Å^{-3}
$\Delta\rho_{min} = -0.67$ e Å^{-3}
Absolute structure: Refined as an inversion twin
Absolute structure parameter: −1 (4)

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O6	0.4659 (6)	−0.4221 (5)	−0.88303 (18)	0.0861 (14)
O3	−0.9934 (5)	−0.3444 (5)	−0.61477 (18)	0.0815 (13)
N2	−0.1980 (6)	−0.3968 (4)	−0.76468 (19)	0.0658 (12)
O2	−1.2412 (6)	−0.2842 (6)	−0.5611 (2)	0.0968 (15)
N1	−0.3161 (6)	−0.3494 (5)	−0.73078 (19)	0.0671 (13)
O4	0.7008 (8)	−0.4943 (6)	−0.9435 (2)	0.122 (2)
O5	0.8443 (9)	−0.2982 (8)	−0.9259 (3)	0.145 (2)
O1	−1.3702 (7)	−0.4910 (7)	−0.5761 (2)	0.123 (2)
C19	0.0707 (7)	−0.3464 (5)	−0.7945 (2)	0.0587 (13)
C8	−0.5825 (7)	−0.4041 (5)	−0.7005 (2)	0.0590 (13)
C3	−1.2583 (9)	−0.4068 (9)	−0.5824 (3)	0.090 (2)
C5	−0.8617 (7)	−0.3742 (6)	−0.6434 (2)	0.0659 (15)
C16	0.3406 (8)	−0.3906 (6)	−0.8529 (2)	0.0656 (15)
C11	−0.4446 (7)	−0.4275 (5)	−0.7311 (2)	0.0618 (14)
H11	−0.448969	−0.504718	−0.752671	0.074*
C9	−0.7122 (7)	−0.5040 (6)	−0.7029 (2)	0.0689 (15)
H9	−0.705537	−0.581369	−0.724270	0.083*
C18	0.2009 (7)	−0.2516 (5)	−0.7937 (2)	0.0655 (15)
H18	0.198036	−0.172160	−0.773028	0.079*
C17	0.3358 (7)	−0.2717 (6)	−0.8230 (2)	0.0677 (15)
H17	0.421932	−0.205814	−0.822424	0.081*
C10	−0.8487 (7)	−0.4902 (6)	−0.6743 (2)	0.0677 (15)
H10	−0.931717	−0.558927	−0.675966	0.081*
C22	−0.0682 (7)	−0.3205 (5)	−0.7625 (2)	0.0617 (14)
H22	−0.061751	−0.246143	−0.739922	0.074*
C20	0.0776 (9)	−0.4663 (5)	−0.8255 (2)	0.0710 (16)
H20	−0.009621	−0.530867	−0.827039	0.085*
C6	−0.7324 (8)	−0.2740 (6)	−0.6404 (2)	0.0764 (17)
H6	−0.739197	−0.196246	−0.619141	0.092*
C21	0.2131 (8)	−0.4884 (6)	−0.8537 (2)	0.0756 (17)
H21	0.218970	−0.569887	−0.873361	0.091*
C4	−1.1264 (9)	−0.4449 (8)	−0.6175 (3)	0.0857 (19)
H4A	−1.086821	−0.542135	−0.610821	0.103*
H4B	−1.171001	−0.444297	−0.649865	0.103*

C7	−0.5976 (8)	−0.2901 (6)	−0.6684 (2)	0.0711 (16)
H7	−0.513326	−0.222775	−0.665958	0.085*
C14	0.7176 (9)	−0.3829 (8)	−0.9207 (3)	0.0848 (19)
C15	0.6016 (8)	−0.3258 (7)	−0.8849 (3)	0.0785 (17)
H15A	0.653522	−0.320003	−0.853552	0.094*
H15B	0.565809	−0.229246	−0.894006	0.094*
C12	0.9676 (12)	−0.3616 (12)	−0.9585 (5)	0.145 (2)
H12A	1.024040	−0.440591	−0.942466	0.175*
H12B	0.914541	−0.400186	−0.986975	0.175*
C13	1.0808 (13)	−0.2527 (12)	−0.9718 (4)	0.145 (2)
H13A	1.161245	−0.293458	−0.993055	0.218*
H13B	1.024480	−0.175271	−0.987935	0.218*
H13C	1.133662	−0.215559	−0.943555	0.218*
C2	−1.3727 (13)	−0.2480 (11)	−0.5256 (4)	0.139 (3)
H2A	−1.381933	−0.323663	−0.501410	0.167*
H2B	−1.477161	−0.237448	−0.541630	0.167*
C1	−1.3215 (13)	−0.1070 (10)	−0.5029 (4)	0.139 (3)
H1A	−1.401592	−0.077904	−0.479592	0.209*
H1B	−1.217725	−0.119369	−0.487328	0.209*
H1C	−1.312388	−0.033665	−0.527309	0.209*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O6	0.080 (3)	0.089 (3)	0.089 (3)	0.000 (2)	0.019 (3)	−0.013 (2)
O3	0.069 (3)	0.093 (3)	0.082 (3)	−0.014 (2)	0.010 (2)	−0.004 (2)
N2	0.066 (3)	0.055 (2)	0.076 (3)	0.001 (2)	−0.002 (3)	−0.001 (2)
O2	0.077 (3)	0.097 (3)	0.117 (4)	0.014 (3)	0.032 (3)	0.019 (3)
N1	0.063 (3)	0.063 (2)	0.076 (3)	0.001 (2)	0.005 (3)	0.000 (2)
O4	0.132 (5)	0.109 (4)	0.124 (4)	0.004 (4)	0.050 (4)	−0.017 (3)
O5	0.118 (4)	0.160 (4)	0.159 (5)	−0.008 (3)	0.057 (4)	−0.021 (4)
O1	0.091 (4)	0.144 (4)	0.133 (5)	−0.035 (4)	0.027 (4)	0.004 (4)
C19	0.060 (3)	0.051 (2)	0.065 (3)	0.004 (2)	−0.002 (3)	0.002 (2)
C8	0.059 (3)	0.054 (2)	0.064 (3)	0.003 (2)	−0.005 (3)	0.004 (2)
C3	0.071 (5)	0.096 (5)	0.102 (5)	−0.003 (4)	−0.002 (4)	0.034 (4)
C5	0.056 (3)	0.071 (3)	0.071 (4)	0.003 (3)	0.004 (3)	0.008 (3)
C16	0.068 (3)	0.063 (3)	0.066 (4)	0.002 (3)	0.002 (3)	−0.001 (3)
C11	0.066 (4)	0.052 (2)	0.067 (3)	0.005 (2)	−0.010 (3)	0.004 (2)
C9	0.064 (4)	0.064 (3)	0.079 (4)	−0.005 (3)	−0.006 (3)	−0.008 (3)
C18	0.062 (3)	0.056 (3)	0.078 (4)	0.006 (2)	0.001 (3)	−0.005 (3)
C17	0.064 (3)	0.063 (3)	0.077 (4)	−0.002 (3)	−0.004 (3)	−0.003 (3)
C10	0.061 (3)	0.064 (3)	0.078 (4)	−0.016 (3)	−0.001 (3)	−0.007 (3)
C22	0.062 (3)	0.047 (2)	0.076 (4)	−0.002 (2)	−0.002 (3)	−0.003 (2)
C20	0.075 (4)	0.056 (3)	0.082 (4)	−0.006 (3)	−0.001 (4)	−0.001 (3)
C6	0.075 (4)	0.069 (3)	0.085 (4)	−0.003 (3)	0.002 (4)	−0.018 (3)
C21	0.082 (4)	0.062 (3)	0.083 (4)	−0.006 (3)	0.009 (4)	−0.015 (3)
C4	0.079 (5)	0.099 (4)	0.079 (4)	−0.018 (4)	0.004 (4)	0.007 (4)
C7	0.064 (4)	0.060 (3)	0.089 (4)	−0.007 (3)	−0.007 (4)	−0.004 (3)

C14	0.082 (5)	0.089 (4)	0.083 (5)	0.011 (4)	0.017 (4)	−0.004 (4)
C15	0.075 (4)	0.082 (3)	0.078 (4)	−0.001 (3)	0.008 (4)	0.006 (3)
C12	0.118 (4)	0.160 (4)	0.159 (5)	−0.008 (3)	0.057 (4)	−0.021 (4)
C13	0.118 (4)	0.160 (4)	0.159 (5)	−0.008 (3)	0.057 (4)	−0.021 (4)
C2	0.121 (5)	0.146 (5)	0.151 (6)	0.013 (5)	0.061 (5)	0.007 (5)
C1	0.121 (5)	0.146 (5)	0.151 (6)	0.013 (5)	0.061 (5)	0.007 (5)

Geometric parameters (Å, º)

O6—C16	1.355 (7)	C18—H18	0.9300
O6—C15	1.422 (8)	C17—H17	0.9300
O3—C5	1.367 (7)	C10—H10	0.9300
O3—C4	1.432 (8)	C22—H22	0.9300
N2—C22	1.275 (7)	C20—C21	1.372 (9)
N2—N1	1.419 (7)	C20—H20	0.9300
O2—C3	1.282 (9)	C6—C7	1.359 (9)
O2—C2	1.497 (10)	C6—H6	0.9300
N1—C11	1.274 (7)	C21—H21	0.9300
O4—C14	1.212 (8)	C4—H4A	0.9700
O5—C14	1.306 (9)	C4—H4B	0.9700
O5—C12	1.477 (10)	C7—H7	0.9300
O1—C3	1.213 (8)	C14—C15	1.474 (9)
C19—C18	1.378 (7)	C15—H15A	0.9700
C19—C20	1.403 (8)	C15—H15B	0.9700
C19—C22	1.463 (8)	C12—C13	1.415 (10)
C8—C7	1.382 (8)	C12—H12A	0.9700
C8—C9	1.406 (8)	C12—H12B	0.9700
C8—C11	1.431 (8)	C13—H13A	0.9600
C3—C4	1.498 (10)	C13—H13B	0.9600
C5—C10	1.375 (8)	C13—H13C	0.9600
C5—C6	1.407 (8)	C2—C1	1.502 (10)
C16—C17	1.375 (8)	C2—H2A	0.9700
C16—C21	1.379 (8)	C2—H2B	0.9700
C11—H11	0.9300	C1—H1A	0.9600
C9—C10	1.376 (8)	C1—H1B	0.9600
C9—H9	0.9300	C1—H1C	0.9600
C18—C17	1.385 (8)		

C16—O6—C15	118.7 (5)	C20—C21—C16	120.4 (5)
C5—O3—C4	116.0 (5)	C20—C21—H21	119.8
C22—N2—N1	111.5 (5)	C16—C21—H21	119.8
C3—O2—C2	114.9 (6)	O3—C4—C3	111.2 (6)
C11—N1—N2	112.6 (4)	O3—C4—H4A	109.4
C14—O5—C12	112.0 (7)	C3—C4—H4A	109.4
C18—C19—C20	118.5 (5)	O3—C4—H4B	109.4
C18—C19—C22	119.3 (5)	C3—C4—H4B	109.4
C20—C19—C22	122.2 (5)	H4A—C4—H4B	108.0
C7—C8—C9	117.4 (6)	C6—C7—C8	121.6 (6)

C7—C8—C11	124.6 (5)	C6—C7—H7	119.2
C9—C8—C11	118.0 (5)	C8—C7—H7	119.2
O1—C3—O2	125.4 (8)	O4—C14—O5	122.6 (7)
O1—C3—C4	119.2 (8)	O4—C14—C15	125.5 (7)
O2—C3—C4	115.4 (6)	O5—C14—C15	111.9 (6)
O3—C5—C10	125.5 (5)	O6—C15—C14	107.8 (6)
O3—C5—C6	115.3 (5)	O6—C15—H15A	110.1
C10—C5—C6	119.2 (6)	C14—C15—H15A	110.1
O6—C16—C17	124.4 (6)	O6—C15—H15B	110.1
O6—C16—C21	115.1 (5)	C14—C15—H15B	110.1
C17—C16—C21	120.5 (6)	H15A—C15—H15B	108.5
N1—C11—C8	124.2 (5)	C13—C12—O5	109.2 (8)
N1—C11—H11	117.9	C13—C12—H12A	109.8
C8—C11—H11	117.9	O5—C12—H12A	109.8
C10—C9—C8	121.7 (5)	C13—C12—H12B	109.8
C10—C9—H9	119.1	O5—C12—H12B	109.8
C8—C9—H9	119.1	H12A—C12—H12B	108.3
C19—C18—C17	121.6 (5)	C12—C13—H13A	109.5
C19—C18—H18	119.2	C12—C13—H13B	109.5
C17—C18—H18	119.2	H13A—C13—H13B	109.5
C16—C17—C18	119.0 (6)	C12—C13—H13C	109.5
C16—C17—H17	120.5	H13A—C13—H13C	109.5
C18—C17—H17	120.5	H13B—C13—H13C	109.5
C5—C10—C9	119.7 (5)	O2—C2—C1	105.5 (8)
C5—C10—H10	120.2	O2—C2—H2A	110.6
C9—C10—H10	120.2	C1—C2—H2A	110.6
N2—C22—C19	122.0 (5)	O2—C2—H2B	110.6
N2—C22—H22	119.0	C1—C2—H2B	110.6
C19—C22—H22	119.0	H2A—C2—H2B	108.8
C21—C20—C19	120.0 (6)	C2—C1—H1A	109.5
C21—C20—H20	120.0	C2—C1—H1B	109.5
C19—C20—H20	120.0	H1A—C1—H1B	109.5
C7—C6—C5	120.4 (5)	C2—C1—H1C	109.5
C7—C6—H6	119.8	H1A—C1—H1C	109.5
C5—C6—H6	119.8	H1B—C1—H1C	109.5

C22—N2—N1—C11	−177.6 (5)	C20—C19—C22—N2	−7.5 (8)
C2—O2—C3—O1	0.7 (11)	C18—C19—C20—C21	1.1 (8)
C2—O2—C3—C4	−179.3 (7)	C22—C19—C20—C21	−177.9 (6)
C4—O3—C5—C10	0.8 (9)	O3—C5—C6—C7	−177.8 (6)
C4—O3—C5—C6	179.7 (5)	C10—C5—C6—C7	1.2 (9)
C15—O6—C16—C17	−1.0 (9)	C19—C20—C21—C16	−2.3 (9)
C15—O6—C16—C21	179.3 (5)	O6—C16—C21—C20	−178.5 (6)
N2—N1—C11—C8	179.8 (5)	C17—C16—C21—C20	1.8 (9)
C7—C8—C11—N1	3.4 (9)	C5—O3—C4—C3	176.2 (5)
C9—C8—C11—N1	−176.4 (5)	O1—C3—C4—O3	−171.2 (6)
C7—C8—C9—C10	−0.4 (8)	O2—C3—C4—O3	8.8 (9)
C11—C8—C9—C10	179.4 (5)	C5—C6—C7—C8	0.0 (10)

C20—C19—C18—C17	0.6 (8)	C9—C8—C7—C6	−0.4 (9)
C22—C19—C18—C17	179.6 (5)	C11—C8—C7—C6	179.8 (5)
O6—C16—C17—C18	−179.8 (5)	C12—O5—C14—O4	−5.3 (13)
C21—C16—C17—C18	−0.1 (9)	C12—O5—C14—C15	174.9 (8)
C19—C18—C17—C16	−1.1 (9)	C16—O6—C15—C14	−178.8 (5)
O3—C5—C10—C9	176.9 (6)	O4—C14—C15—O6	−1.8 (11)
C6—C5—C10—C9	−2.0 (9)	O5—C14—C15—O6	178.0 (6)
C8—C9—C10—C5	1.6 (9)	C14—O5—C12—C13	165.9 (10)
N1—N2—C22—C19	−178.9 (5)	C3—O2—C2—C1	176.1 (8)
C18—C19—C22—N2	173.6 (5)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···O4^{i}	0.93	2.57	3.483 (9)	169
C4—H4*B*···N1^{ii}	0.97	2.69	3.618 (10)	161

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