Crystal structure and Hirshfeld surface analysis of 6-((E)-2-[4-[2-(4-chlorophenyl)-2-oxoethoxy]phenyl]ethenyl)-4,5-dihydropyridazin-3(2H)-one

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The pyridazine ring in the title compound, C20H17ClN2O3, adopts a screw-boat conformation. The whole molecule is flattened, the dihedral angles subtended by the least-squares plane of the central aromatic ring with those of the terminal benzene and pyridazine rings being 15.18 (19) and 11.23 (19)°, respectively. In the crystal, the molecules are linked by pairs of N—H···C=O bonds into centrosymmetric dimers and by C—H···C=C contacts into columns. The results of the Hirshfeld surface analysis show that the most prominent interactions are H···H, accounting for 36.5% of overall crystal packing, and H···O/O···H (18.6% contribution) contacts.

1. Chemical context

Pyridazinone derivatives are a class of nitrogenous heterocyclic compounds that have attracted considerable attention because of their prospective pharmacological and medicinal properties as anti-inflammatory (Boukharsa et al., 2018), antitumor (Bouchmaa et al., 2018, 2019), antifungal (Rozada et al., 2020), antidepressant (Boukharsa et al., 2016), antitubercular, anticonvulsant (Asif et al., 2020) and antiviral (El-Shanbaky et al., 2021) agents. In addition, pyridazinones demonstrate some interesting physicochemical properties (Daoui et al., 2020a; El Kalai et al., 2021a,b) and some studies have shown that these compounds are good corrosion inhibitors (Chelfi et al., 2020). Encouraged by the bioactivity of these compounds and in a continuation of our studies in the field of the synthesis, molecular structures and Hirshfeld surfaces analyses of new pyridazin-3(2H)-one derivatives (Daoui et al., 2020b, 2021), we report herein the crystal structure and the results of the Hirshfeld surface analysis of 6-((E)-2-[4-[2-(4-chlorophenyl)-2-oxoethoxy]phenyl]ethenyl)-4,5-dihydropyridazin-3(2H)-one.
2. Structural commentary

The molecular structure of the title compound is presented in Fig. 1. The bond lengths in the N1—C15 chain (Table 1) are consistent with an alternation of double and single bonds while those in the amide fragment indicate strong \( \pi \)-conjugation. The N1—N2 distance of 1.406 (4) Å agrees well with the values for related pyridazinones (Daoui, Çınar et al., 2019; Daoui, Baydere et al., 2019). The conformation of the dihydropyridazine ring is close to a screw-boat \([\Theta = 111.9 (6) ^\circ, \varphi = 34.6 (6) ^\circ]\). The whole molecule is flattened with the largest deviations from the least-squares plane of 0.356 (4) and 0.339 (5) Å being observed for atoms C18 and C19, respectively. The central benzene ring forms dihedral angles of 11.23 (19) and 15.18 (19)° with the planes of the terminal dihydropyridazine and benzene rings, respectively.

3. Supramolecular features

In the crystal, the molecules are linked into centrosymmetric dimers by pairs of N—H···O hydrogen bonds, giving rise to an \( R_2^2(8) \) graph-set motif (Fig. 2a, Table 2). No \( \pi \)-\( \pi \) interactions are present in this structure, but the molecules are connected by weak C—H···O contacts into stacks running along the \( a \) axis direction (Fig. 2b,c, Table 2). Other contacts of the C—H···O and C—H···Cl types further stabilize the crystal structure (Table 2).

4. Hirshfeld surface analysis

In order to visualize and study the intermolecular contacts, a Hirshfeld surface analysis of the title compound was undertaken using Crystal Explorer 17.5 (Turner et al., 2017). Fig. 3a shows the 3D surface mapped over \( d_{norm} \) over the range \(-0.484 \) (red) to 1.403 (blue) a.u. The pale-red spots on the surface represent short N—H···O and C—H···O interactions (Table 2). The surfaces mapped over \( d_s \) and \( d_i \) are presented in Fig. 3b and 3c.

The overall two-dimensional fingerprint plot and those delineated into H—H, H—C/C—H, H—O/O—H, H—Cl/Cl—H and C—C contacts are presented in Fig. 4. H—H

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**Table 1**

|          | \( d_{norm} \) (Å) | \( |d_s| \) (Å) | \( |d_i| \) (Å) |
|----------|-------------------|---------------|---------------|
| C20—O3   | 1.241 (4)         | 2.11          | 2.891 (4)     |
| N2—C20   | 1.333 (5)         | 2.44          | 3.327 (4)     |
| N1—N2    | 1.406 (4)         | 2.53          | 3.421 (4)     |
| N1—C17   | 1.292 (4)         | 2.94          | 3.737 (3)     |
| C7—O1    | 1.219 (4)         | 2.73          | 3.514 (3)     |

**Table 2**

|          | \( d_{norm} \) (Å) | \( |d_s| \) (Å) | \( |d_i| \) (Å) |
|----------|-------------------|---------------|---------------|
| N2—H2—O3 | 0.86              | 2.11          | 2.891 (4)     |
| C4—H4—O3 | 0.93              | 2.44          | 3.327 (4)     |
| C15—H15—Cl1 | 0.93         | 2.53          | 3.421 (4)     |
| C18—H18—Cl1 | 0.97         | 2.94          | 3.737 (3)     |
| C8—H8—Cl1 | 0.97              | 2.73          | 3.514 (3)     |

Symmetry codes: (i) \( x, y + 1, z + 1 \); (ii) \( x + 1, y, z + \frac{1}{2} \); (iii) \( x - 1, y + 1, z + \frac{1}{2} \); (iv) \( x, y - 1, z + \frac{1}{2} \); (v) \( x + 1, y, z + 1 \).
interactions are the most prominent, accounting for 36.5% of the overall crystal packing. H—C/H—C/O/O—H/C contacts, including intermolecular C—H···O and N—H···O hydrogen bonding, make a 18.6% contribution to the Hirshfeld surface. H—C/C···H and H—C/C···H interactions add a 15.4% contribution. The contributions from H—C/Cl···H and C···C contacts are 11.2% and 7.6%, respectively.

5. Database survey
A search of the Cambridge Structural Database (CSD, version 5.40, update March 2020; Groom et al., 2016) revealed two structures containing the same pyridazinone fragments as in the title structure but with different substituents, viz. 6-[(E)-6-(4-hydroxy-styryl)-4,5-dihydropyridazin-3(2H)-one (MUCLEE; Daoui, Çınar et al., 2019) and (E)-6-(4-hydroxy-3-methoxyphenyl)ethenyl-4,5-dihydropyridazin-3(2H)-one (LOSSOE; Daoui, Baydere et al., 2019). Both these structures exhibit bond lengths in the pyridazine ring and N—H···O hydrogen-bonding parameters that are very similar to those observed in the title structure.

6. Synthesis and crystallization
A mixture of (E)-6-(4-hydroxystyryl)-4,5-dihydropyridazin-3(2H)-one (0.5 g, 2.3 mmol), K2CO3 (0.79 g, 5.7 mmol) and 2-chloro-1-(4-chlorophenyl)ethan-1-one (0.47 g, 2.5 mmol) in acetone (50 ml) was refluxed overnight. After cooling, the solution was filtered and the solvent removed under reduced pressure. The residue was purified by recrystallization from ethanol to afford single crystals (yield 72%).

7. Refinement
Crystal data, data collection and structure refinement details are summarized in Table 3. Hydrogen atoms were positioned geometrically and treated as riding, with C—H = 0.96 Å for methylene [Uiso(H) = 1.5 Ueq(C)], C—H = 0.93 Å for aromatic [Uiso(H) = 1.2 Ueq(C)] and C—H = 0.98 Å for methine [Uiso

(H) = 1.2 Ueq(C)] H atoms.

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Author contributions are as follows. Conceptualization, SD, IM, EBÇ, AA, ND, NB and KK; synthesis, SD, KK, NB, AA, writing, IM and EBÇ, formal analysis ND and KK, validation IM, EBÇ and ND.

Table 3

| Crystal data | Chemical formula | C20H17ClN2O3 | Mr | 368.80 |
| Temperature (K) | 296 |
| a, b, c (Å) | 7.3514 (4), 11.5539 (7), 41.397 (3) |
| V (Å³) | 3516.2 (4) |
| Z | 8 |
| µ (mm⁻¹) | 0.24 |
| Crystal size (mm) | 0.45 × 0.20 × 0.05 |

Data collection
Diffractometer
Stoe IPDS 2
Absorption correction
Integration (X-RED32; Stoe & Cie, 2002)

| Tmin, Tmax | 0.925, 0.994 |
| No. of measured, independent and observed [I > 2σ(I)] reflections | 19519, 2913, 1682 |
| Rint | 0.113 |
| (sinθ/λ)max (Å⁻¹) | 0.584 |

Refinement

| R[F² > 2σ(F²)], wR(F²), S | 0.060, 0.128, 0.99 |
| No. of reflections | 2913 |
| No. of parameters | 235 |
| H-atom treatment | H-atom parameters constrained |
| Δρmax, Δρmin (e Å⁻³) | 0.34, –0.22 |

Computer programs: X-AREA and X-RED32 (Stoe & Cie, 2002), SHELXT2018/3 (Sheldrick, 2015a), SHELXL2018/3 (Sheldrick, 2015b), OLEX2 (Dolomanov et al., 2009), Mercury (Macrae et al., 2020), WinGX (Farrugia, 2012), PLATON (Spek, 2020) and publCIF (Westrip, 2010).

Figure 4
(a) The overall two-dimensional fingerprint plot, and those delineated into (b) H···H, (c) H···C/C···H, (d) H···O/O···H, (e) H···Cl/Cl···H and (f) C···C interactions.
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References

Asif, M. & Imran, M. (2020). *Anal. Chem. Lett.* **10**, 414–427.
Bouchmaa, N., Mrid, R. B., Boukharsa, Y., Bouargalne, Y., Nhiri, M., Idr, A., Taoufik, J., Ansar, M. & Zyad, A. (2019). *Drug Res. (Stuttg.)* **69**, 528–536.
Bouchmaa, N., Tilaoui, M., Boukharsa, Y., Jaâfari, A., Mouse, H. A., Ali Oukerrou, M., Taoufik, J., Ansar, M. & Zyad, A. (2018). *Pharm. Chem. J.* **51**, 893–901.
Bouchmaa, Y., Lakhlili, W., El harti, J., Meddah, B., Tiendrebeogo, R. Y., Taoufik, J., El Abbes Faouzi, M., Ibrahim, A. & Ansar, M. (2018). *J. Mol. Struct.* **1153**, 119–127.
Chelfi, T., Benchat, N., Bouklah, M., Daoui, S., Karrouchi, K., Allali, M., Taleb, M., Ech chihbi, E., Almalki, F. A. & Benhada, T. (2020). *J. Bio-Tribology* **6**, 1–14.
Daoui, S., Baydere, C., Akman, F., El Kalai, F., Mahi, L., Dege, N., Topcu, Y., Karrouchi, K. & Benchat, N. (2020a). *J. Mol. Struct.* **1225**, 129180.
Daoui, S., Baydere, C., Chelfi, T., El Kalai, F., Dege, N., Karrouchi, K. & Benchat, N. (2020b). *Acta Cryst.* **E76**, 432–437.
Daoui, S., Baydere, C., El Kalai, F., Saddik, R., Dege, N., Karrouchi, K. & Benchat, N. (2019). *Acta Cryst.* **E75**, 1734–1737.
Daoui, S., Cinar, E. B., Dege, N., Chelfi, T. El Kalai, F., Abudunia, A., Karrouchi, K. & Benchat, N. (2021). *Acta Cryst.* **E77**, 23–27.
Daoui, S., Çınar, E. B., El Kalai, F., Saddik, R., Dege, N., Karrouchi, K. & Benchat, N. (2019). *Acta Cryst.* **E75**, 1880–1883.
Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
El Kalai, F., Çınar, E. B., Lai, C. H., Daoui, S., Chelfi, T., Allali, M., Dege, N., Karrouchi, K. & Benchat, N. (2021a). *J. Mol. Struct.* **1228**, 129435.
El Kalai, F., Karrouchi, K., Baydere, C., Daoui, S., Allali, M., Dege, N., Benchat, N. & Brandán, S. A. (2021b). *J. Mol. Struct.* **1223**, 129213.
El-Shanbaky, H. M., El-Hameed, A. & Mohamed, M. S. (2021). *J. Adv. Pharm. Res.* **5**, 202–210.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* **B72**, 171–179.
Macrae, C. F., Sovago, I., Cottrell, S. J., Galek, P. T. A., McCabe, P., Piccock, E., Platings, M., Shields, G. P., Stevens, J. S., Towler, M. & Wood, P. A. (2020). *J. Appl. Cryst.* **53**, 226–235.
Rozada, A. M., Rodrigues-Vendramini, F. A., Gonçalves, D. S., Rosa, F. A., Basso, E. A., Seixas, F. A., Kioshima, É. S. & Gauze, G. F. (2020). *Bioorg. Med. Chem. Lett.* **30**, 127244.
Sheldrick, G. M. (2015a). *Acta Cryst.* **A71**, 3–8.
Sheldrick, G. M. (2015b). *Acta Cryst.* **C71**, 3–8.
Spek, A. L. (2020). *Acta Cryst.* **E76**, 1–11.
Stoe & Cie (2002). *X-AREA and X-RED32*. Stoe & Cie, Darmstadt, Germany.
Turner, M. J., McKinnon, J. J., Wolff, S. K., Grimwood, D. J., Spackman, P. R., Jayatilaka, D. & Spackman, M. A. (2017). *Crystal Explorer 17.5*. University of Western Australia.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.
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Computing details

Data collection: X-AREA (Stoe & Cie, 2002); cell refinement: X-AREA (Stoe & Cie, 2002); data reduction: X-RED32 (Stoe & Cie, 2002); program(s) used to solve structure: SHELXT2018/3 (Sheldrick, 2015a); program(s) used to refine structure: SHELXL2018/3 (Sheldrick, 2015b); molecular graphics: OLEX2 (Dolomanov et al., 2009) and Mercury (Macrae et al., 2020); software used to prepare material for publication: WinGX (Farrugia, 2012), SHELXL2018/3 (Sheldrick, 2015b), PLATON (Spek, 2020) and publCIF (Westrip, 2010).

6-((E)-2-{4-[2-(4-Chlorophenyl)-2-oxoethoxy]phenyl}ethenyl)-4,5-dihydropyridazin-3(2H)-one

Crystal data

C_{20}H_{17}ClN_{2}O_{3}  
Mr = 368.80
Orthorhombic, Pbca
a = 7.3514 (4) Å
b = 11.5539 (7) Å
c = 41.397 (3) Å
V = 3516.2 (4) Å³
Z = 8
F(000) = 1536

Data collection

STOE IPDS 2
diffractometer
Radiation source: sealed X-ray tube, 12 x 0.4 mm long-focus
Plane graphite monochromator
Detector resolution: 6.67 pixels mm⁻¹
rotation method scans
Absorption correction: integration
(X-RED32; Stoe & Cie, 2002)

Refinement

Refinement on F²
Least-squares matrix: full
R[F² > 2σ(F²)] = 0.060
wR(F²) = 0.128
S = 0.99
2913 reflections

supporting information

Acta Cryst. (2022). E78, 8-11  [https://doi.org/10.1107/S205698902101238X]
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained

\[ w = \frac{1}{\sigma^2(F^2_o) + (0.0518P)^2} \]
where \( P = (F^2_o + 2F^2_c)/3 \)

\((\Delta/\sigma)_{\text{max}} < 0.001\)
\(\Delta \rho_{\text{max}} = 0.34 \text{ e Å}^{-3}\)
\(\Delta \rho_{\text{min}} = -0.22 \text{ e Å}^{-3}\)

**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.

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

|      | x   | y   | z   | Uiso*/Ueq |
|------|-----|-----|-----|-----------|
| Cl   | 0.40565 (17) | 0.91913 (10) | 0.94188 (2) | 0.0775 (4) |
| O2   | 0.3849 (3) | 0.58839 (19) | 0.76710 (5) | 0.0548 (6) |
| O1   | 0.3725 (4) | 0.5145 (2) | 0.82642 (5) | 0.0652 (7) |
| O3   | 0.4691 (5) | 0.8576 (2) | 0.48754 (6) | 0.0901 (11) |
| N1   | 0.4362 (5) | 0.9053 (3) | 0.57229 (6) | 0.0583 (9) |
| N2   | 0.4339 (5) | 0.9186 (3) | 0.53853 (6) | 0.0627 (9) |
| H2   | 0.420683 | 0.987853 | 0.531280 | 0.075* |
| C9   | 0.3861 (5) | 0.6229 (3) | 0.73520 (7) | 0.0446 (8) |
| C12  | 0.3776 (5) | 0.6716 (3) | 0.66888 (7) | 0.0450 (8) |
| C7   | 0.3875 (5) | 0.6191 (3) | 0.82384 (7) | 0.0459 (8) |
| C14  | 0.4262 (5) | 0.7332 (3) | 0.72432 (7) | 0.0460 (9) |
| H14  | 0.455198 | 0.791510 | 0.738937 | 0.055* |
| C15  | 0.3713 (5) | 0.6918 (3) | 0.63384 (7) | 0.0485 (9) |
| H15  | 0.336983 | 0.628939 | 0.621208 | 0.058* |
| C8   | 0.4077 (5) | 0.6746 (3) | 0.79090 (7) | 0.0463 (8) |
| H8A  | 0.316866 | 0.734746 | 0.788149 | 0.056* |
| H8B  | 0.527120 | 0.709594 | 0.788902 | 0.056* |
| C10  | 0.3420 (5) | 0.5366 (3) | 0.71335 (8) | 0.0493 (9) |
| H10  | 0.315275 | 0.462357 | 0.720621 | 0.059* |
| C6   | 0.3939 (5) | 0.6959 (3) | 0.85261 (7) | 0.0442 (8) |
| C13  | 0.4229 (4) | 0.7563 (3) | 0.69135 (8) | 0.0480 (9) |
| H13  | 0.451610 | 0.830326 | 0.684145 | 0.058* |
| C11  | 0.3378 (5) | 0.5613 (3) | 0.68064 (8) | 0.0495 (9) |
| H11  | 0.307759 | 0.502891 | 0.666138 | 0.059* |
| C17  | 0.4022 (5) | 0.8025 (3) | 0.58300 (7) | 0.0480 (9) |
| C16  | 0.4088 (5) | 0.7891 (3) | 0.61803 (8) | 0.0513 (9) |
| H16  | 0.441662 | 0.853378 | 0.630205 | 0.062* |
| C5   | 0.3951 (5) | 0.6460 (3) | 0.88335 (7) | 0.0505 (9) |
| H5   | 0.395053 | 0.565786 | 0.885305 | 0.061* |
| C1   | 0.3956 (5) | 0.8158 (3) | 0.85007 (8) | 0.0507 (9) |
| H1   | 0.393722 | 0.850303 | 0.829780 | 0.061* |
| C4   | 0.3965 (5) | 0.7130 (3) | 0.91071 (8) | 0.0546 (10) |
| H4   | 0.394802 | 0.678837 | 0.931046 | 0.066* |
| C3   | 0.4005 (5) | 0.8324 (3) | 0.90763 (8) | 0.0542 (9) |
Atomic displacement parameters (Å²)

|   | U¹¹ | U¹² | U¹³ | U²² | U²³ | U³³ |
|---|-----|-----|-----|-----|-----|-----|
| Cl1 | 0.1050 (9) | 0.0789 (7) | 0.0487 (5) | −0.0053 (7) | −0.0003 (6) | −0.0147 (5) |
| O2 | 0.0842 (18) | 0.0491 (13) | 0.0311 (12) | −0.0041 (14) | −0.0035 (13) | 0.0037 (11) |
| O1 | 0.102 (2) | 0.0493 (16) | 0.0440 (14) | −0.0081 (15) | 0.0098 (15) | 0.0043 (12) |
| O3 | 0.168 (3) | 0.0733 (19) | 0.0293 (15) | −0.019 (2) | 0.0040 (16) | 0.0027 (13) |
| N1 | 0.089 (3) | 0.058 (2) | 0.0279 (14) | −0.0069 (18) | 0.0018 (15) | 0.0022 (14) |
| C14 | 0.054 (2) | 0.0548 (18) | 0.0319 (15) | −0.0002 (18) | 0.0013 (16) | 0.0067 (15) |
| C9 | 0.051 (2) | 0.052 (2) | 0.0315 (17) | −0.0038 (18) | 0.0023 (17) | 0.0030 (16) |
| C12 | 0.050 (2) | 0.051 (2) | 0.0340 (17) | −0.0002 (18) | 0.0026 (16) | −0.0011 (16) |
| C7 | 0.051 (2) | 0.050 (2) | 0.0368 (18) | −0.0005 (18) | 0.0030 (18) | 0.0056 (15) |
| C14 | 0.054 (2) | 0.048 (2) | 0.0361 (18) | −0.0049 (18) | −0.0008 (16) | 0.0006 (16) |
| C15 | 0.057 (2) | 0.056 (2) | 0.0321 (17) | −0.0005 (19) | 0.0012 (18) | −0.0004 (16) |
| C8 | 0.056 (2) | 0.048 (2) | 0.0347 (17) | −0.0009 (19) | 0.0018 (17) | 0.0015 (16) |
| C10 | 0.066 (3) | 0.0399 (19) | 0.0417 (19) | −0.0040 (17) | 0.0017 (17) | 0.0058 (16) |
| C6 | 0.049 (2) | 0.049 (2) | 0.0346 (17) | 0.0001 (18) | 0.0049 (17) | 0.0051 (15) |
| C13 | 0.054 (2) | 0.048 (2) | 0.0414 (19) | −0.0020 (19) | 0.0011 (17) | 0.0063 (16) |
| C11 | 0.064 (2) | 0.049 (2) | 0.0351 (18) | −0.0017 (18) | 0.0034 (16) | −0.0031 (17) |
| C17 | 0.057 (2) | 0.053 (2) | 0.0349 (17) | 0.0009 (19) | −0.0002 (18) | −0.0021 (16) |
| C16 | 0.060 (2) | 0.059 (2) | 0.0347 (18) | −0.004 (2) | 0.0018 (19) | −0.0018 (16) |
| C5 | 0.063 (2) | 0.047 (2) | 0.0409 (19) | −0.0010 (19) | −0.0006 (19) | 0.0083 (16) |
| C1 | 0.070 (3) | 0.047 (2) | 0.0348 (18) | 0.005 (2) | 0.0027 (19) | 0.0076 (16) |
| C4 | 0.069 (3) | 0.058 (2) | 0.0367 (19) | −0.003 (2) | −0.0005 (19) | 0.0076 (16) |
| C3 | 0.060 (2) | 0.063 (2) | 0.0389 (19) | 0.002 (2) | 0.0005 (19) | −0.0031 (18) |
| C2 | 0.071 (3) | 0.047 (2) | 0.050 (2) | 0.001 (2) | 0.002 (2) | 0.0007 (18) |
| C18 | 0.097 (3) | 0.057 (2) | 0.037 (2) | −0.008 (2) | 0.004 (2) | −0.0009 (18) |
| C20 | 0.102 (4) | 0.065 (3) | 0.034 (2) | −0.011 (2) | 0.002 (2) | 0.001 (2) |
| C19 | 0.126 (4) | 0.062 (3) | 0.043 (2) | −0.006 (3) | 0.011 (2) | −0.001 (2) |

Geometric parameters (Å, °)

|    | C2—O3 | 1.241 (4) | C10—C11 | 1.384 (5) |
|----|-------|-----------|---------|-----------|
| N2 | 1.333 (5) | C10—H10 | 0.9300   |
| N1 | 1.406 (4) | C6—C1 | 1.389 (5) |
| N1 | 1.292 (4) | C6—C5 | 1.397 (4) |
| C16 | 1.459 (4) | C13—H13 | 0.9300   |
| C15 | 1.329 (5) | C11—H11 | 0.9300   |
| Bond          | Distance (Å) | Bond          | Distance (Å) | Bond          | Distance (Å) |
|---------------|--------------|---------------|--------------|---------------|--------------|
| C12—C15       | 1.470 (4)    | C17—C18       | 1.492 (5)    | C14—C13       | 1.391 (4)    |
| C7—O1         | 1.219 (4)    | C16—H16       | 0.9300       | C12—C13       | 1.391 (4)    |
| C11—C3        | 1.737 (3)    | C5—C4         | 1.373 (5)    | C12—C13       | 1.391 (4)    |
| O2—C9         | 1.379 (4)    | C5—H5         | 0.9300       | C12—C13       | 1.391 (4)    |
| O2—C8         | 1.411 (4)    | C1—C2         | 1.385 (5)    | N2—H2         | 0.8600       |
| N2—H2         | 0.8600       | C1—H1         | 0.9300       | C9—C14        | 1.384 (4)    |
| C9—C14        | 1.384 (4)    | C4—C3         | 1.386 (5)    | C9—C10        | 1.385 (4)    |
| C9—C10        | 1.385 (4)    | C4—H4         | 0.9300       | C12—C13       | 1.391 (4)    |
| C12—C13       | 1.391 (4)    | C3—C2         | 1.383 (5)    | C12—C11       | 1.395 (5)    |
| C12—C11       | 1.395 (5)    | C2—H2A        | 0.9300       | C7—C6         | 1.486 (4)    |
| C7—C6         | 1.486 (4)    | C18—C19       | 1.487 (5)    | C7—C8         | 1.514 (4)    |
| C7—C8         | 1.514 (4)    | C18—H18A      | 0.9700       | C14—C13       | 1.391 (4)    |
| C14—C13       | 1.391 (4)    | C18—H18B      | 0.9700       | C14—H14       | 0.9300       |
| C14—H14       | 0.9300       | C20—C19       | 1.490 (5)    | C15—H15       | 0.9300       |
| C15—H15       | 0.9300       | C19—H19A      | 0.9700       | C8—H8A        | 0.9700       |
| C8—H8A        | 0.9700       | C19—H19B      | 0.9700       | C8—H8B        | 0.9700       |
| C9—O2—C8      | 117.6 (2)    | C12—C11—H11   | 119.2        | C9—O2—C8      | 117.6 (2)    |
| C17—N1—N2     | 116.0 (3)    | N1—C17—C16    | 115.6 (3)    | C17—N1—N2     | 116.0 (3)    |
| C20—N2—N1     | 126.5 (3)    | N1—C17—C18    | 121.7 (3)    | C20—N2—N1     | 126.5 (3)    |
| C20—N2—H2     | 116.7        | C16—C17—C18   | 122.7 (3)    | C20—N2—H2     | 116.7        |
| N1—N2—H2      | 116.7        | C15—C16—C17   | 124.9 (3)    | O2—C9—C14     | 125.4 (3)    |
| O2—C9—C14     | 125.4 (3)    | C15—C16—H16   | 117.5        | O2—C9—C14     | 125.4 (3)    |
| O2—C9—C10     | 114.6 (3)    | C17—C16—H16   | 117.5        | O2—C9—C10     | 114.6 (3)    |
| C14—C9—C10    | 120.0 (3)    | C4—C5—C6      | 121.2 (3)    | C14—C9—C10    | 120.0 (3)    |
| C13—C12—C11   | 117.4 (3)    | C4—C5—H5      | 119.4        | C13—C12—C11   | 117.4 (3)    |
| C13—C12—C15   | 123.8 (3)    | C6—C5—H5      | 119.4        | C13—C12—C15   | 123.8 (3)    |
| C11—C12—C15   | 118.9 (3)    | C2—C1—C6      | 120.5 (3)    | C11—C12—C15   | 118.9 (3)    |
| O1—C7—C6      | 121.7 (3)    | C2—C1—H1      | 119.7        | O1—C7—C6      | 121.7 (3)    |
| O1—C7—C8      | 120.5 (3)    | C6—C1—H1      | 119.7        | O1—C7—C8      | 120.5 (3)    |
| C6—C7—C8      | 117.8 (3)    | C5—C4—C3      | 119.1 (3)    | C6—C7—C8      | 117.8 (3)    |
| C9—C14—C13    | 119.5 (3)    | C5—C4—H4      | 120.5        | C9—C14—C13    | 119.5 (3)    |
| C9—C14—H14    | 120.3        | C3—C4—H4      | 120.5        | C9—C14—H14    | 120.3        |
| C13—C14—H14   | 120.3        | C2—C3—C4      | 121.0 (3)    | C13—C14—H14   | 120.3        |
| C16—C15—C12   | 127.9 (3)    | C2—C3—C11     | 119.1 (3)    | C16—C15—C12   | 127.9 (3)    |
| C16—C15—H15   | 116.1        | C4—C3—C11     | 120.0 (3)    | C16—C15—H15   | 116.1        |
| C12—C15—H15   | 116.1        | C3—C2—C1      | 119.4 (3)    | C12—C15—H15   | 116.1        |
| O2—C8—C7      | 108.5 (3)    | C3—C2—H2A     | 120.3        | O2—C8—C7      | 108.5 (3)    |
| O2—C8—H8A     | 110.0        | C1—C2—H2A     | 120.3        | O2—C8—H8A     | 110.0        |
| C7—C8—H8A     | 110.0        | C19—C18—C17   | 112.0 (3)    | C7—C8—H8A     | 110.0        |
| O2—C8—H8B     | 110.0        | C19—C18—H18A  | 109.2        | O2—C8—H8B     | 110.0        |
| C7—C8—H8B     | 110.0        | C17—C18—H18A  | 109.2        | C7—C8—H8B     | 110.0        |
| H8A—C8—H8B    | 108.4        | C19—C18—H18B  | 109.2        | H8A—C8—H8B    | 108.4        |
| C11—C10—C9    | 119.7 (3)    | C17—C18—H18B  | 109.2        | C11—C10—C9    | 119.7 (3)    |
| C11—C10—H10   | 120.1        | H18A—C18—H18B | 107.9        | C11—C10—H10   | 120.1        |
| C9—C10—H10    | 120.1        | O3—C20—N2     | 121.0 (4)    | C9—C10—H10    | 120.1        |
| C1—C6—C5      | 118.7 (3)    | O3—C20—C19    | 122.9 (4)    | C1—C6—C5      | 118.7 (3)    |
sup-5

C1—C6—C7 122.4 (3) N2—C20—C19 116.0 (3)
C5—C6—C7 118.9 (3) C18—C19—C20 111.4 (3)
C12—C13—C14 121.7 (3) C18—C19—H19A 109.3
C12—C13—H13 119.1 C20—C19—H19A 109.3
C14—C13—H13 119.1 C18—C19—H19B 109.3
C10—C11—C12 121.7 (3) C20—C19—H19B 109.3
C10—C11—H11 119.2 H19A—C19—H19B 108.0

C17—N1—N2—C20 −19.7 (6) N2—N1—C17—C16 178.7 (3)
C8—O2—C9—C14 7.0 (5) N2—N1—C17—C18 −2.0 (5)
C8—O2—C9—C10 −172.8 (3) O2—C9—C14—C13 179.9 (3)
O2—C9—C14—C13 179.9 (3) N1—C17—C16—C15 177.3 (4)
C12—C13—C14—C13 −0.4 (5) C18—C17—C16—C15 −1.9 (6)
C5—C6—C7—C8 121.7 (3) C13—C12—C11—C10 −0.9 (5)
C11—C12—C13—C14 −0.9 (5) C4—H4···O3ii 0.93 2.44 3.327 (4) 160
O1—C7—C8—O2 175.7 (3) C13—H13···O1iii 0.93 2.53 3.421 (4) 161
C14—C9—C10—C11 179.6 (3) C8—H8B···Cg3v 0.97 2.94 3.514 (3) 138
C15—C14—C13—C12 179.7 (3) N2—H2···O3i 0.86 2.11 2.891 (4) 151
C9—C10—C11—C12 0.9 (5) N1—N2—C20—O3 170.9 (4)
C9—C10—C11—C10 0.2 (6) C16—C17—C18—C19 −147.0 (4)
C13—C12—C11—C10 0.3 (5) C17—N1—N2—C20 −19.7 (6)
C15—C12—C11—C10 179.8 (3) C17—N1—N2—C20 178.7 (3)

Hydrogen-bond geometry (Å, °)
Cg3 is the centroid of the C9–C14 ring.

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|------|-------|-------|---------|
| N2—H2···O3i | 0.86 | 2.11 | 2.891 (4) | 151 |
| C4—H4···O3ii | 0.93 | 2.44 | 3.327 (4) | 160 |
| C13—H13···O1iii | 0.93 | 2.53 | 3.421 (4) | 161 |
| C18—H18···C11iv | 0.97 | 2.94 | 3.737 (3) | 140 |
| C8—H8B···Cg3v | 0.97 | 2.73 | 3.514 (3) | 138 |

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