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# 2-(4-Chloro-2-nitrophenyl)-5-[4-(propyloxy)phenyl]-1,3,4-oxadiazole

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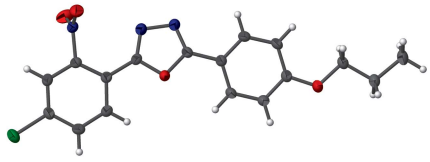
**Keywords:** crystal structure; 1,3,4-oxadiazole; nitro compound; hydrogen bonding; C—H···$\pi$ interactions.

**CCDC reference**: 1515447

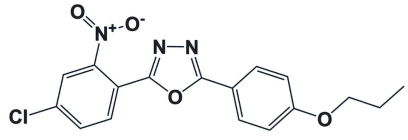
**Structural data**: full structural data are available from iucrdata.iucr.org

The title compound, $C_{17}H_{14}ClN_3O_4$, was prepared by the Huisgen reaction of 4-chloro-2-nitrobenzoyl chloride and 5-(4-propyloxyphenyl)tetrazole. The diphenyl-1,3,4-oxadiazole unit is nearly planar. The oxadiazole ring is inclined to the 4-chloro-2-nitrophenyl ring by 7.77 (8)°, and by 7.93 (8)° to the 4-propyloxyphenyl ring. The benzene rings are inclined to one another by 1.32 (7)°. The nitro group is twisted out of the plane of the benzene ring to which it is attached by 73.59 (16)°. The propoxy chain mean plane is inclined to the benzene ring to which it is attached by 4.46 (13)°. In the crystal, C—H···O and C—H···N hydrogen bonds connect the molecules, forming ribbons propagating along the *b*-axis direction. The ribbons are linked by C—H···$\pi$ interactions, forming slabs parallel to the *ab* plane.

**3D view**



**Chemical scheme**




## Structure description

Donor–acceptor substituted $\pi$-systems are solvatochromic (Detert *et al.* 2002; Rettig 1986), making these materials interesting for sensing and non-linear optical applications (Schmitt *et al.* 2008; Schönhaber *et al.* 2010; Franco *et al.* 2010). *o*-Nitrobiaryl compounds are starting materials for the Cadogan cyclization (Cadogan 1962; Letessier *et al.* 2013).

Apart from the nitro group, te title compound, Fig. 1, is nearly planar. The oxadiazole ring (O8/N10/N11/C7/C9) is inclined to the benzene ring (C1–C6) by 7.77 (8)° and by 7.93 (8)° to benzene ring (C12–C17). The benzene rings are inclined to one another by 1.32 (7)°. The nitro group is twisted out of the plane of the benzene ring (C1–C6), to which it is attached, by 73.59 (16)%. The propoxy chain mean plane (O18/C19–C21) is inclined to the benzene ring (C12–C17), to which it is attached, by 4.46 (13)°. The torsion angles along the biaryl axes are −7.5 (2)° (C6—C1—C7—N11) and 7.7 (2)° (N10—C9—C12—C17). The bonds connecting the central 1,3,4-oxadiazole ring with the donor- and acceptor-substituted benzene rings, 1.4555 (17) and 1.4527 (17) Å, respectively, are very

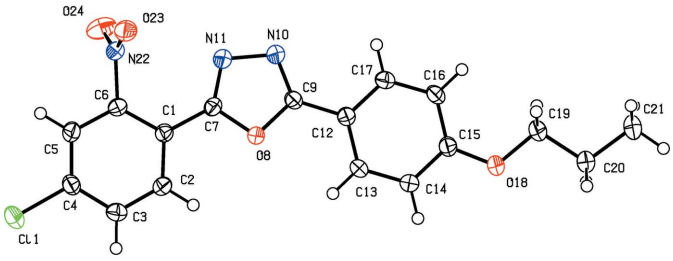



**Figure 1**
A view of the molecular structure of the title compound, with the atom labelling and displacement ellipsoids drawn at the 50% probability level.

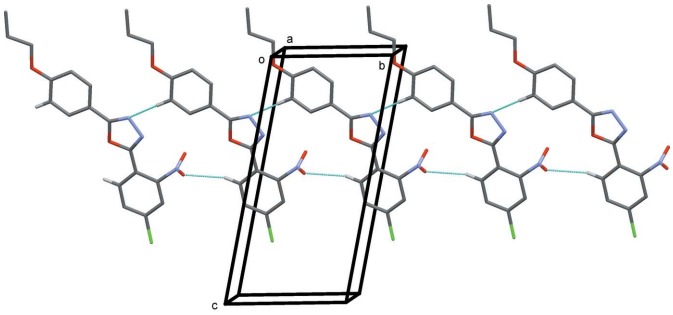



**Figure 2**
A partial view along the *a* axis of the crystal packing of the title compound. The hydrogen bonds are shown as dashed lines (see Table 1), and H atoms not involved in these interactions have been excluded.

similar. The C4—Cl1 bond length of 1.7270 (14) Å is nearly identical to the bond lengths [1.727, 1.728 Å] found in 1-chloro-3,4-dinitrobenzene (Wilkins & Small, 1985).

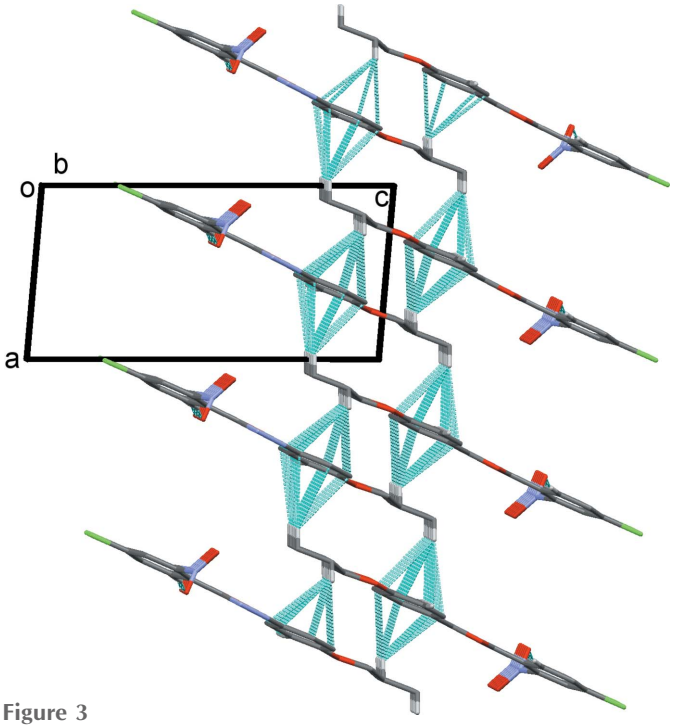



**Figure 3**
A partial view along the *b* axis of the crystal packing of the title compound. The C—H···$\pi$ interactions are illustrated as dashed lines (see Table 1), and H atoms not involved in the intermolecular interactions have been excluded.

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

*Cg* is the centroid of ring C12–C17.

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C2—H2···O24$^{i}$ | 0.95 | 2.57 | 3.4821 (18) | 161 |
| C14—H14···N10$^{i}$ | 0.95 | 2.41 | 3.3351 (19) | 163 |
| C20—H20*B*···*Cg*$^{ii}$ | 0.99 | 2.86 | 3.7524 (17) | 151 |
| C21—H21*B*···*Cg*$^{iii}$ | 0.99 | 2.84 | 3.6473 (17) | 140 |

Symmetry codes: (i) $x, y-1, z$; (ii) $-x+1, -y, -z$; (iii) $-x, -y, -z$.

**Table 2**
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | $C_{17}H_{14}ClN_3O_4$ |
| $M_r$ | 359.76 |
| Crystal system, space group | Triclinic, $P\overline{1}$ |
| Temperature (K) | 193 |
| *a*, *b*, *c* (Å) | 7.5691 (5), 7.7907 (5), 14.904 (1) |
| $\alpha, \beta, \gamma$ (°) | 101.911 (5), 91.311 (5), 107.224 (5) |
| *V* (Å$^3$) | 818.06 (10) |
| *Z* | 2 |
| Radiation type | Mo *K*$\alpha$ |
| $\mu$ (mm$^{-1}$) | 0.26 |
| Crystal size (mm) | 0.44 × 0.28 × 0.08 |
| | |
| Data collection | |
| Diffractometer | STOE *IPDS* 2T |
| Absorption correction | – |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 7659, 3942, 3221 |
| $R_{int}$ | 0.018 |
| $(\sin\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.662 |
| | |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S* | 0.036, 0.098, 1.04 |
| No. of reflections | 3942 |
| No. of parameters | 227 |
| H-atom treatment | H-atom parameters constrained |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.35, −0.38 |

Computer programs: *X-AREA* and *X-RED32* (Stoe & Cie, 1996), *SHELXT2014* (Sheldrick, 2015*a*), *PLATON* (Spek, 2009), *Mercury* (Macrae *et al.*, 2008) and *SHELXL2014* (Sheldrick, 2015*b*).

In the crystal, molecules are connected *via* C—H···O and C—H···N hydrogen bonds, involving the aromatic rings to the the nitro and oxadiazole groups, respectively, forming ribbons that propagate along the *b*-axis direction (Table 1 and Fig. 2). The ribbons are linked by C—H···$\pi$ interactions, forming slabs parallel to the *ab* plane (Table 1 and Fig. 3).

## Synthesis and crystallization

4-Chloro-2-nitrobenzoic acid (640 mg, 3.18 mmol) was refluxed for 12 h in thionyl chloride (6 ml, 82.7 mmol), excess thionyl chloride was distilled off *in vacuo* and the crude benzoyl chloride was dissolved in toluene (15 ml), then 5-(4-propyloxyphenyl)tetrazole (466 mg, 2.28 mmol) and 2,4,6-collidine (0.6 ml, 4.53 mmol) were added. After refluxing for 72 h, the mixture was cooled and 2*N* $HCl_{(aq)}$ (25 ml) was added. The aqueous phase was extracted with chloroform (3 × 20 ml). The combined organic phase was dried with $MgSO_4$ followed by evaporation and column chromatography (silica

gel, toluene: ethyl acetate = 40:1) to afforded 723 mg of the title compound (yield 88%). $^1$H NMR ($CDCl_3$) $\delta$ 8.07 (*d*, *J* = 8.4 Hz, 1H), 7.98–7.95 (*m*, 3H), 7.75 (*dd*, *J* = 8.4, 2.1 Hz, 1H), 7.02 – 6.99 (*m*, 2H), 4.00 (*t*, *J* = 6.5 Hz, 2H), 1.89–1.80 (*m*, 2H), 1.06 (*t*, *J* = 7.4 Hz, 3H). $^{13}$C NMR ($CDCl_3$) $\delta$ 166.1, 162.6, 160.0, 138.6, 133.2, 132.5, 129.1, 125.0, 117.1, 115.4, 115.3, 8.16, 69.9, 22.6, 10.6. The title compound was crystallized from a dichloromethane solution giving colourless plate-like crystals (m.p. 419–411 K).

## Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

## References


Cadogan, J. I. G. (1962). *Q. Rev. Chem. Soc.* **16**, 208–239.

Detert, H., Sugiono, E. & Kruse, G. (2002). *J. Phys. Org. Chem.* **15**, 638–641.

Franco, A., Brusatin, G., Guglielmi, M., Stracci, G., De Matteis, F., Casalboni, M., Detert, H., Grimm, B. & Schrader, S. (2010). *J. Non-Cryst. Solids*, **356**, 1689–1695.

Letessier, J., Geffe, M., Schollmeyer, D. & Detert, H. (2013). *Synthesis*, **45**, 3173–3178.

Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.

Rettig, W. (1986). *Angew. Chem. Int. Ed. Engl.* **25**, 971–988.

Schmitt, V., Glang, S., Preis, J. & Detert, H. (2008). *Sen Lett*, **6**, 524–530.

Schönhaber, J., Frank, W. & Müller, T. J. J. (2010). *Org. Lett.* **12**, 4122–4125.

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. (2009). *Acta Cryst.* D**65**, 148–155.

Stoe & Cie (1996). *X-AREA* and *X-RED32*. Stoe & Cie, Darmstadt, Germany.

Wilkins, A. & Small, R. W. H. (1985). *Acta Cryst.* C**41**, 1509–1512.

# full crystallographic data

*IUCrData* (2016). **1**, x161782 [https://doi.org/10.1107/S241431461601782X]

## 2-(4-Chloro-2-nitrophenyl)-5-[4-(propyloxy)phenyl]-1,3,4-oxadiazole

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### 2-(4-Chloro-2-nitrophenyl)-5-[4-(propyloxy)phenyl]-1,3,4-oxadiazole

*Crystal data*

$C_{17}H_{14}ClN_3O_4$
$M_r$ = 359.76
Triclinic, $P\overline{1}$
$a$ = 7.5691 (5) Å
$b$ = 7.7907 (5) Å
$c$ = 14.904 (1) Å
$\alpha$ = 101.911 (5)°
$\beta$ = 91.311 (5)°
$\gamma$ = 107.224 (5)°
$V$ = 818.06 (10) Å$^3$
$Z$ = 2
$F(000)$ = 372
$D_x$ = 1.461 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 8906 reflections
$\theta$ = 2.8–28.2°
$\mu$ = 0.26 mm$^{-1}$
$T$ = 193 K
Plate, colourless
0.44 × 0.28 × 0.08 mm

*Data collection*

STOE IPDS 2T diffractometer
Detector resolution: 6.67 pixels mm$^{-1}$
rotation method scans
7659 measured reflections
3942 independent reflections
3221 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.018
$\theta_{max}$ = 28.1°, $\theta_{min}$ = 2.8°
$h$ = −8→10
$k$ = −10→10
$l$ = −19→19

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.098
$S$ = 1.04
3942 reflections
227 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0509P)^2 + 0.2041P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.35 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.38 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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Cl1 | 0.99714 (6) | 1.19481 (5) | 0.78094 (3) | 0.04350 (12) |
| C1 | 0.74265 (17) | 0.98285 (17) | 0.48656 (9) | 0.0236 (2) |
| C2 | 0.76872 (19) | 0.86300 (17) | 0.53984 (9) | 0.0266 (3) |
| H2 | 0.7314 | 0.7343 | 0.5137 | 0.032* |
| C3 | 0.84763 (19) | 0.92731 (19) | 0.62969 (9) | 0.0290 (3) |
| H3 | 0.8658 | 0.8438 | 0.6646 | 0.035* |
| C4 | 0.90006 (19) | 1.11479 (19) | 0.66840 (9) | 0.0284 (3) |
| C5 | 0.87652 (19) | 1.23889 (18) | 0.61822 (9) | 0.0293 (3) |
| H5 | 0.9124 | 1.3673 | 0.6449 | 0.035* |
| C6 | 0.79957 (18) | 1.17076 (17) | 0.52846 (9) | 0.0251 (3) |
| C7 | 0.65423 (18) | 0.91012 (17) | 0.39304 (9) | 0.0244 (3) |
| O8 | 0.61819 (13) | 0.72641 (12) | 0.35657 (6) | 0.0254 (2) |
| C9 | 0.53202 (18) | 0.70529 (17) | 0.27188 (9) | 0.0247 (3) |
| N10 | 0.51751 (18) | 0.85882 (16) | 0.25766 (8) | 0.0330 (3) |
| N11 | 0.59744 (18) | 0.99363 (16) | 0.33768 (8) | 0.0328 (3) |
| C12 | 0.46798 (18) | 0.52310 (17) | 0.21119 (9) | 0.0249 (3) |
| C13 | 0.47390 (18) | 0.36665 (18) | 0.24229 (9) | 0.0261 (3) |
| H13 | 0.5218 | 0.3791 | 0.3036 | 0.031* |
| C14 | 0.41008 (19) | 0.19464 (18) | 0.18386 (9) | 0.0272 (3) |
| H14 | 0.4129 | 0.0887 | 0.2053 | 0.033* |
| C15 | 0.34137 (19) | 0.17554 (18) | 0.09339 (9) | 0.0264 (3) |
| C16 | 0.33658 (19) | 0.33078 (18) | 0.06153 (9) | 0.0281 (3) |
| H16 | 0.2917 | 0.3185 | −0.0003 | 0.034* |
| C17 | 0.39800 (19) | 0.50300 (18) | 0.12099 (9) | 0.0284 (3) |
| H17 | 0.3923 | 0.6086 | 0.1000 | 0.034* |
| O18 | 0.28345 (15) | 0.00031 (13) | 0.04241 (7) | 0.0329 (2) |
| C19 | 0.2206 (2) | −0.03094 (19) | −0.05283 (9) | 0.0302 (3) |
| H19A | 0.3227 | 0.0304 | −0.0868 | 0.036* |
| H19B | 0.1163 | 0.0193 | −0.0590 | 0.036* |
| C20 | 0.1582 (2) | −0.23635 (19) | −0.09084 (10) | 0.0306 (3) |
| H20A | 0.0605 | −0.2966 | −0.0544 | 0.037* |
| H20B | 0.2643 | −0.2845 | −0.0855 | 0.037* |
| C21 | 0.0825 (2) | −0.2826 (2) | −0.19156 (10) | 0.0399 (4) |
| H21A | 0.1759 | −0.2153 | −0.2267 | 0.060* |
| H21B | −0.0304 | −0.2466 | −0.1961 | 0.060* |
| H21C | 0.0534 | −0.4154 | −0.2166 | 0.060* |
| N22 | 0.78583 (16) | 1.30994 (15) | 0.47701 (8) | 0.0285 (2) |
| O23 | 0.89671 (16) | 1.34446 (14) | 0.42036 (7) | 0.0382 (3) |
| O24 | 0.66822 (17) | 1.38593 (16) | 0.49721 (9) | 0.0472 (3) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cl1 | 0.0577 (3) | 0.0393 (2) | 0.02601 (18) | 0.00562 (17) | −0.01008 (15) | 0.00578 (14) |
| C1 | 0.0236 (6) | 0.0223 (6) | 0.0239 (6) | 0.0054 (5) | 0.0023 (5) | 0.0054 (5) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C2 | 0.0300 (6) | 0.0216 (6) | 0.0277 (6) | 0.0069 (5) | 0.0025 (5) | 0.0059 (5) |
| C3 | 0.0327 (7) | 0.0289 (6) | 0.0271 (6) | 0.0090 (5) | 0.0029 (5) | 0.0110 (5) |
| C4 | 0.0314 (7) | 0.0295 (7) | 0.0212 (6) | 0.0056 (5) | −0.0001 (5) | 0.0044 (5) |
| C5 | 0.0342 (7) | 0.0227 (6) | 0.0264 (6) | 0.0036 (5) | 0.0006 (5) | 0.0035 (5) |
| C6 | 0.0267 (6) | 0.0220 (6) | 0.0259 (6) | 0.0052 (5) | 0.0026 (5) | 0.0075 (5) |
| C7 | 0.0256 (6) | 0.0196 (5) | 0.0264 (6) | 0.0052 (5) | 0.0022 (5) | 0.0044 (5) |
| O8 | 0.0323 (5) | 0.0207 (4) | 0.0219 (4) | 0.0071 (3) | −0.0015 (4) | 0.0037 (3) |
| C9 | 0.0257 (6) | 0.0259 (6) | 0.0216 (6) | 0.0065 (5) | −0.0009 (5) | 0.0059 (5) |
| N10 | 0.0424 (7) | 0.0242 (5) | 0.0299 (6) | 0.0090 (5) | −0.0093 (5) | 0.0036 (4) |
| N11 | 0.0410 (7) | 0.0243 (5) | 0.0306 (6) | 0.0086 (5) | −0.0076 (5) | 0.0037 (5) |
| C12 | 0.0261 (6) | 0.0241 (6) | 0.0231 (6) | 0.0060 (5) | 0.0007 (5) | 0.0051 (5) |
| C13 | 0.0303 (6) | 0.0272 (6) | 0.0206 (6) | 0.0086 (5) | −0.0024 (5) | 0.0055 (5) |
| C14 | 0.0327 (7) | 0.0251 (6) | 0.0247 (6) | 0.0096 (5) | −0.0004 (5) | 0.0069 (5) |
| C15 | 0.0281 (6) | 0.0264 (6) | 0.0228 (6) | 0.0073 (5) | 0.0009 (5) | 0.0034 (5) |
| C16 | 0.0324 (7) | 0.0309 (7) | 0.0196 (6) | 0.0073 (5) | −0.0020 (5) | 0.0068 (5) |
| C17 | 0.0331 (7) | 0.0266 (6) | 0.0256 (6) | 0.0070 (5) | −0.0003 (5) | 0.0097 (5) |
| O18 | 0.0459 (6) | 0.0260 (5) | 0.0229 (5) | 0.0082 (4) | −0.0053 (4) | 0.0018 (4) |
| C19 | 0.0355 (7) | 0.0306 (7) | 0.0218 (6) | 0.0083 (5) | −0.0010 (5) | 0.0028 (5) |
| C20 | 0.0320 (7) | 0.0288 (6) | 0.0268 (6) | 0.0065 (5) | −0.0024 (5) | 0.0018 (5) |
| C21 | 0.0479 (9) | 0.0358 (8) | 0.0290 (7) | 0.0100 (7) | −0.0071 (6) | −0.0029 (6) |
| N22 | 0.0340 (6) | 0.0206 (5) | 0.0279 (6) | 0.0041 (4) | −0.0025 (5) | 0.0056 (4) |
| O23 | 0.0511 (6) | 0.0310 (5) | 0.0313 (5) | 0.0067 (5) | 0.0074 (5) | 0.0125 (4) |
| O24 | 0.0483 (7) | 0.0397 (6) | 0.0648 (8) | 0.0234 (5) | 0.0108 (6) | 0.0214 (6) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Cl1—C4 | 1.7270 (14) | C13—H13 | 0.9500 |
| C1—C2 | 1.3961 (18) | C14—C15 | 1.3964 (18) |
| C1—C6 | 1.3984 (17) | C14—H14 | 0.9500 |
| C1—C7 | 1.4555 (17) | C15—O18 | 1.3548 (16) |
| C2—C3 | 1.3822 (19) | C15—C16 | 1.3970 (19) |
| C2—H2 | 0.9500 | C16—C17 | 1.3867 (18) |
| C3—C4 | 1.3864 (19) | C16—H16 | 0.9500 |
| C3—H3 | 0.9500 | C17—H17 | 0.9500 |
| C4—C5 | 1.3857 (19) | O18—C19 | 1.4346 (16) |
| C5—C6 | 1.3779 (18) | C19—C20 | 1.5069 (18) |
| C5—H5 | 0.9500 | C19—H19A | 0.9900 |
| C6—N22 | 1.4768 (16) | C19—H19B | 0.9900 |
| C7—N11 | 1.2883 (17) | C20—C21 | 1.5253 (19) |
| C7—O8 | 1.3635 (14) | C20—H20A | 0.9900 |
| O8—C9 | 1.3633 (15) | C20—H20B | 0.9900 |
| C9—N10 | 1.2920 (17) | C21—H21A | 0.9800 |
| C9—C12 | 1.4527 (17) | C21—H21B | 0.9800 |
| N10—N11 | 1.4019 (16) | C21—H21C | 0.9800 |
| C12—C17 | 1.3941 (18) | N22—O23 | 1.2173 (16) |
| C12—C13 | 1.4026 (17) | N22—O24 | 1.2183 (17) |
| C13—C14 | 1.3796 (18) | | |

| | | | |
|---|---|---|---|
| C2—C1—C6 | 116.88 (12) | C13—C14—H14 | 119.8 |
| C2—C1—C7 | 120.09 (11) | C15—C14—H14 | 119.8 |
| C6—C1—C7 | 123.00 (11) | O18—C15—C14 | 115.03 (12) |
| C3—C2—C1 | 121.50 (12) | O18—C15—C16 | 124.85 (12) |
| C3—C2—H2 | 119.2 | C14—C15—C16 | 120.13 (12) |
| C1—C2—H2 | 119.2 | C17—C16—C15 | 119.33 (12) |
| C2—C3—C4 | 119.40 (12) | C17—C16—H16 | 120.3 |
| C2—C3—H3 | 120.3 | C15—C16—H16 | 120.3 |
| C4—C3—H3 | 120.3 | C16—C17—C12 | 120.80 (12) |
| C5—C4—C3 | 121.12 (12) | C16—C17—H17 | 119.6 |
| C5—C4—Cl1 | 119.52 (10) | C12—C17—H17 | 119.6 |
| C3—C4—Cl1 | 119.36 (11) | C15—O18—C19 | 118.75 (11) |
| C6—C5—C4 | 118.10 (12) | O18—C19—C20 | 107.23 (11) |
| C6—C5—H5 | 120.9 | O18—C19—H19A | 110.3 |
| C4—C5—H5 | 120.9 | C20—C19—H19A | 110.3 |
| C5—C6—C1 | 122.98 (12) | O18—C19—H19B | 110.3 |
| C5—C6—N22 | 115.73 (11) | C20—C19—H19B | 110.3 |
| C1—C6—N22 | 121.25 (11) | H19A—C19—H19B | 108.5 |
| N11—C7—O8 | 112.64 (11) | C19—C20—C21 | 110.74 (12) |
| N11—C7—C1 | 129.40 (12) | C19—C20—H20A | 109.5 |
| O8—C7—C1 | 117.93 (11) | C21—C20—H20A | 109.5 |
| C9—O8—C7 | 102.51 (10) | C19—C20—H20B | 109.5 |
| N10—C9—O8 | 112.22 (11) | C21—C20—H20B | 109.5 |
| N10—C9—C12 | 128.58 (12) | H20A—C20—H20B | 108.1 |
| O8—C9—C12 | 119.19 (11) | C20—C21—H21A | 109.5 |
| C9—N10—N11 | 106.49 (11) | C20—C21—H21B | 109.5 |
| C7—N11—N10 | 106.13 (11) | H21A—C21—H21B | 109.5 |
| C17—C12—C13 | 119.45 (12) | C20—C21—H21C | 109.5 |
| C17—C12—C9 | 119.63 (12) | H21A—C21—H21C | 109.5 |
| C13—C12—C9 | 120.92 (11) | H21B—C21—H21C | 109.5 |
| C14—C13—C12 | 119.97 (12) | O23—N22—O24 | 125.20 (12) |
| C14—C13—H13 | 120.0 | O23—N22—C6 | 117.67 (11) |
| C12—C13—H13 | 120.0 | O24—N22—C6 | 117.08 (12) |
| C13—C14—C15 | 120.31 (12) | | |

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| C6—C1—C2—C3 | −0.19 (19) | C1—C7—N11—N10 | −178.06 (13) |
| C7—C1—C2—C3 | −178.23 (13) | C9—N10—N11—C7 | 0.37 (16) |
| C1—C2—C3—C4 | 0.8 (2) | N10—C9—C12—C17 | 7.7 (2) |
| C2—C3—C4—C5 | −0.7 (2) | O8—C9—C12—C17 | −173.09 (12) |
| C2—C3—C4—Cl1 | 179.54 (11) | N10—C9—C12—C13 | −171.67 (14) |
| C3—C4—C5—C6 | −0.1 (2) | O8—C9—C12—C13 | 7.51 (19) |
| Cl1—C4—C5—C6 | 179.66 (11) | C17—C12—C13—C14 | −0.3 (2) |
| C4—C5—C6—C1 | 0.8 (2) | C9—C12—C13—C14 | 179.14 (12) |
| C4—C5—C6—N22 | −177.27 (12) | C12—C13—C14—C15 | 0.7 (2) |
| C2—C1—C6—C5 | −0.6 (2) | C13—C14—C15—O18 | 179.73 (12) |
| C7—C1—C6—C5 | 177.33 (13) | C13—C14—C15—C16 | −0.1 (2) |
| C2—C1—C6—N22 | 177.30 (12) | O18—C15—C16—C17 | 179.22 (13) |
| C7—C1—C6—N22 | −4.7 (2) | C14—C15—C16—C17 | −1.0 (2) |

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| C2—C1—C7—N11 | 170.45 (14) | C15—C16—C17—C12 | 1.4 (2) |
| C6—C1—C7—N11 | −7.5 (2) | C13—C12—C17—C16 | −0.8 (2) |
| C2—C1—C7—O8 | −7.31 (18) | C9—C12—C17—C16 | 179.77 (13) |
| C6—C1—C7—O8 | 174.78 (11) | C14—C15—O18—C19 | −176.50 (12) |
| N11—C7—O8—C9 | −0.04 (15) | C16—C15—O18—C19 | 3.3 (2) |
| C1—C7—O8—C9 | 178.09 (11) | C15—O18—C19—C20 | −177.75 (12) |
| C7—O8—C9—N10 | 0.30 (15) | O18—C19—C20—C21 | 178.03 (12) |
| C7—O8—C9—C12 | −179.01 (11) | C5—C6—N22—O23 | 104.46 (14) |
| O8—C9—N10—N11 | −0.42 (16) | C1—C6—N22—O23 | −73.63 (16) |
| C12—C9—N10—N11 | 178.80 (13) | C5—C6—N22—O24 | −72.95 (16) |
| O8—C7—N11—N10 | −0.20 (16) | C1—C6—N22—O24 | 108.97 (15) |

*Hydrogen-bond geometry (Å, º)*

Cg is the centroid of ring C12–C17.

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C2—H2···O24$^{i}$ | 0.95 | 2.57 | 3.4821 (18) | 161 |
| C14—H14···N10$^{i}$ | 0.95 | 2.41 | 3.3351 (19) | 163 |
| C20—H20*B*···*Cg*$^{ii}$ | 0.99 | 2.86 | 3.7524 (17) | 151 |
| C21—H21*B*···*Cg*$^{iii}$ | 0.99 | 2.84 | 3.6473 (17) | 140 |

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