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5-(2,4-Dichlorophenyl)-3-(4-nitrophenyl)-1,2,4-oxadiazole

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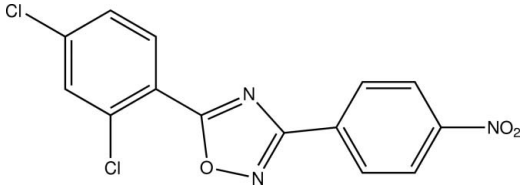
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.005 Å; R factor = 0.048; wR factor = 0.091; data-to-parameter ratio = 13.4.

In the title compound, $C_{14}H_7Cl_2N_3O_3$, the dichlorophenyl and nitrophenyl rings form dihedral angles of 5.4 (2) and 4.0 (2)°, respectively, with the oxadiazole ring. The nitro group is twisted out of the attached benzene ring by a dihedral angle of 10.4 (3)°. In the crystal, molecules are linked into a chain along the a axis by C—H···N hydrogen bonds.

Related literature

For the biological activity of heterocyclic compounds including oxadiazoles, see: Andersen *et al.* (1994); Showell *et al.* (1991); Watjen *et al.* (1989); Swain *et al.* (1991); Clitherow *et al.* (1996); Isloor *et al.* (2010); Chandrakantha *et al.* (2010). For related structures, see: Wang *et al.* (2006); Fun *et al.* (2010*a*,*b*).



Experimental

Crystal data

$C_{14}H_7Cl_2N_3O_3$
M_r = 336.13
Orthorhombic, $Pca2_1$
a = 13.5272 (5) Å
b = 6.5362 (2) Å
c = 15.6880 (5) Å
V = 1387.08 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.48 mm^{-1}
T = 296 K
0.37 × 0.11 × 0.04 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.843, T_{max} = 0.980
9845 measured reflections
2658 independent reflections
2029 reflections with $I > 2\sigma(I)$
R_{int} = 0.045

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.091
S = 1.06
2658 reflections
199 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}
Absolute structure: Flack (1983), 1243 Friedel pairs
Flack parameter: −0.01 (7)

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

D—H···A	D—H	H···A	D···A	D—H···A
C13—H13A···N1^{i}	0.93	2.54	3.338 (5)	144

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI5063).

References

Andersen, K. E., Jørgensen, A. S. & Braestrup, C. (1994). *Eur. J. Med. Chem.* **29**, 393–399.
Bruker (2005). *APEX2* , *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Chandrakantha, B., Shetty, P., Nambiyar, V., Isloor, N. & Isloor, A. M. (2010). *Eur. J. Med. Chem.* **45**, 1206–1210.
Clitherow, J. W., Beswick, P., Irving, W. J., Scopes, D. I. C., Barnes, J. C., Clapham, J., Brown, J. D., Evans, D. J. & Hayes, A. G. (1996). *Bioorg. Med. Chem. Lett.* **6**, 833–838.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Fun, H.-K., Rosli, M. M., Rai, S., Isloor, A. M. & Shetty, P. (2010*a*). *Acta Cryst.* E**66**, o772.
Fun, H.-K., Rosli, M. M., Rai, S., Isloor, A. M. & Shetty, P. (2010*b*). *Acta Cryst.* E**66**, o851.
Isloor, A. M., Kalluraya, B. & Pai, K. S. (2010). *Eur. J. Med. Chem.* **45**, 825–830.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Showell, G. A., Gibbons, T. L., Kneen, C. O., MacLeod, A. M., Merchant, K., Saunders, J., Freedman, S. B., Patel, S. & Baker, R. (1991). *J. Med. Chem.* **34**, 1086–1094.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

‡ Thomson Reuters ResearcherID: A-3561-2009.

Swain, C. J., Baker, R., Kneen, C., Moseley, J., Saunders, J., Seward, E. M., Stevenson, G., Beer, M., Stanton, J. & Watling, K. (1991). *J. Med. Chem.* **34**, 140–151.
Wang, H.-B., Liu, Z.-Q., Wang, H.-B. & Yan, X.-C. (2006). *Acta Cryst.* E**62**, o4715–o4716.
Watjen, F., Baker, R., Engelstoff, M., Herbert, R., MacLeod, A., Knight, A., Merchant, K., Moseley, J., Saunders, J., Swain, C. J., Wong, E. & Springer, J. P. (1989). *J. Med. Chem.* **32**, 2282–2291.

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Acta Cryst. (2010). E**66**, o1196–o1197 [https://doi.org/10.1107/S1600536810011153]

5-(2,4-Dichlorophenyl)-3-(4-nitrophenyl)-1,2,4-oxadiazole

Hoong-Kun Fun, Mohd Mustaqim Rosli, Sankappa Rai, Arun M Isloor and Prakash Shetty

S1. Comment

Heterocyclic compounds are becoming increasingly important in recent years due to their pharmacological activities (Isloor *et al.*, 2010). Nitrogen- and oxygen-containing five/six-membered heterocyclic compounds are of enormous significance in the field of medicinal chemistry (Chandrakantha *et al.*, 2010). Oxadiazoles play a very vital role in the preparation of various biologically active drugs with anti-inflammatory (Andersen *et al.*, 1994), anti-cancer (Showell *et al.*, 1991), anti-HIV (Watjen *et al.*, 1989), anti-diabetic and anti-microbial (Swain *et al.*, 1991) activities. The results of biological studies showed that oxadiazole derivatives also possess maximum anti-inflammatory, analgesic and minimum ulcerogenic and lipid per-oxidation (Clitherow *et al.*, 1996) properties.

Bond lengths and angles in the title molecule (Fig.1) are within the normal range and comparable to those observed in related structures (Wang *et al.*, 2006; Fun *et al.*, 2010*a*,b). The oxadiazole ring (C7/C8/N1/N2/O1) forms dihedral angles of 5.4 (2)° and 4.0 (2)°, respectively, with with the C1–C6 and C9–C14 benzene rings. The plane of the nitro group is twisted out of the C9–C14 benzene ring by a dihedral of 10.4 (3)°.

In the crystal structure (Fig. 2), the molecules are connected by intermolecular C13—H13A···N1 hydrogen bonds (Table 1) forming chains along the *a* axis.

S2. Experimental

The title compound was prepared by heating a solution of 2,4-dichlorobenzoyl chloride (1.15 g, 0.02 mol) and N′-hydroxy-4-nitrobenzamidine (1 g, 0.02 mol) in pyridine (30 ml). The reaction mixture was heated at 114°C for 1.5 h and concentrated under vacuum. Further purification was done by column chromatography. The solid obtained was recrystallized using dichloromethane (yield: 1.0 g (55%), m.p 458-461 K).

S3. Refinement

H atoms were positioned geometrically with C-H = 0.93 Å and were refined using a riding model with U_{iso}(H) = 1.2U_{eq}(C).

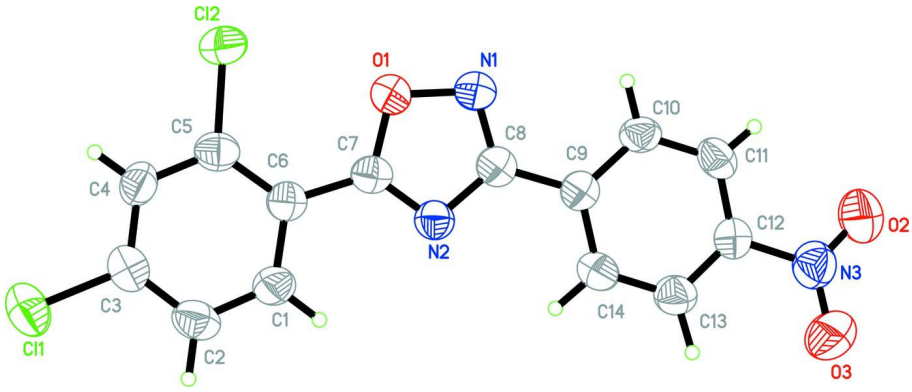

Figure 1

The molecular structure of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme.

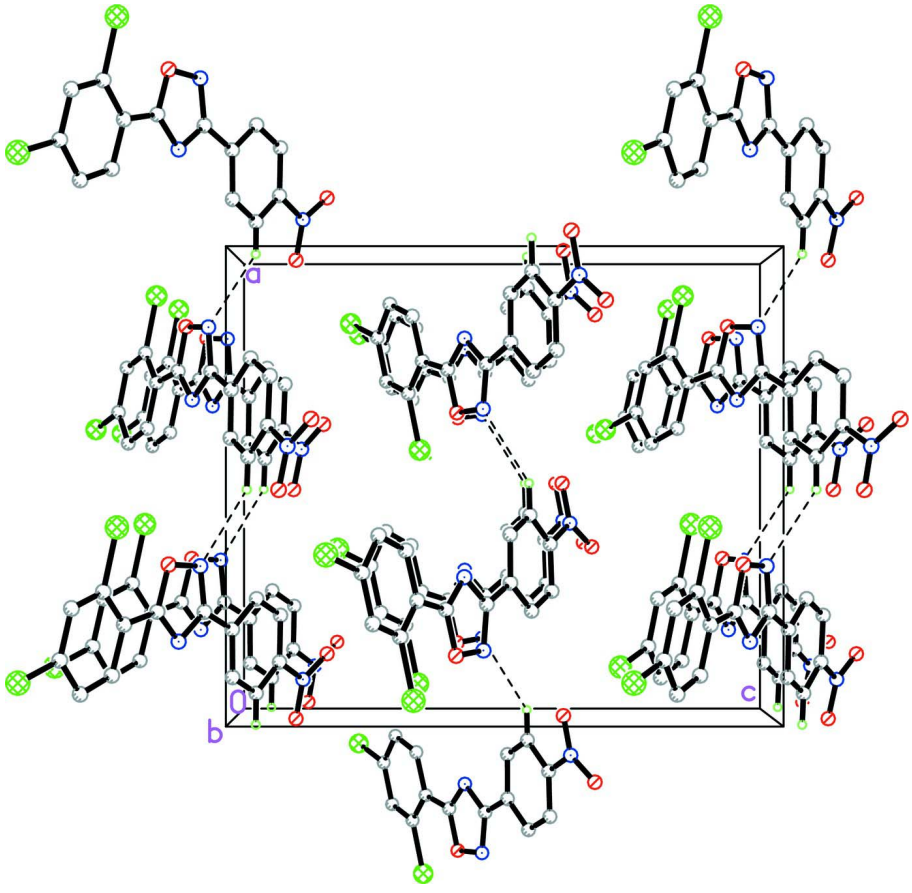

Figure 2

The crystal packing of the title compound, showing hydrogen-bonded (dashed lines) chains along the *a* axis. H atoms not involved in the interactions have been omitted.

5-(2,4-Dichlorophenyl)-3-(4-nitrophenyl)-1,2,4-oxadiazole

Crystal data

$C_{14}H_7Cl_2N_3O_3$
$M_r = 336.13$
Orthorhombic, $Pca2_1$
Hall symbol: P 2c -2ac
$a = 13.5272$ (5) Å
$b = 6.5362$ (2) Å
$c = 15.6880$ (5) Å
$V = 1387.08$ (8) Å^3
$Z = 4$
$F(000) = 680$
$D_x = 1.610$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2389 reflections
$\theta = 2.6$–$30.1°$
$\mu = 0.48$ mm^{-1}
$T = 296$ K
Plate, colourless
0.37 × 0.11 × 0.04 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min} = 0.843$, $T_{max} = 0.980$
9845 measured reflections
2658 independent reflections
2029 reflections with $I > 2\sigma(I)$
$R_{int} = 0.045$
$\theta_{max} = 26.0°$, $\theta_{min} = 2.6°$
$h = -13 \rightarrow 16$
$k = -7 \rightarrow 8$
$l = -17 \rightarrow 19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.091$
$S = 1.06$
2658 reflections
199 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0361P)^2 + 0.1623P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}
Absolute structure: Flack (1983), 1243 Friedel pairs
Absolute structure parameter: −0.01 (7)

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

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.37074 (7)	1.17134 (15)	0.20371 (7)	0.0672 (3)
Cl2	0.08039 (7)	0.73697 (16)	0.34239 (10)	0.0814 (4)
O1	0.15793 (17)	0.3687 (4)	0.41360 (18)	0.0643 (8)

O2	0.3673 (2)	−0.6615 (5)	0.6679 (2)	0.0975 (12)
O3	0.5067 (2)	−0.5915 (5)	0.6109 (2)	0.0959 (11)
N1	0.1636 (2)	0.1879 (5)	0.4615 (2)	0.0653 (10)
N2	0.3154 (2)	0.2966 (4)	0.4278 (2)	0.0454 (7)
N3	0.4200 (3)	−0.5541 (5)	0.6249 (2)	0.0646 (9)
C1	0.3731 (2)	0.6429 (5)	0.3289 (2)	0.0515 (10)
H1A	0.4198	0.5472	0.3463	0.062*
C2	0.4043 (3)	0.8143 (5)	0.2848 (3)	0.0562 (10)
H2A	0.4709	0.8346	0.2729	0.067*
C3	0.3342 (3)	0.9546 (5)	0.2589 (2)	0.0478 (9)
C4	0.2351 (3)	0.9269 (5)	0.2767 (2)	0.0493 (9)
H4A	0.1888	1.0233	0.2594	0.059*
C5	0.2060 (2)	0.7551 (5)	0.3204 (2)	0.0484 (9)
C6	0.2735 (2)	0.6087 (4)	0.3481 (2)	0.0417 (8)
C7	0.2522 (2)	0.4235 (5)	0.3964 (2)	0.0423 (8)
C8	0.2581 (3)	0.1536 (5)	0.4676 (2)	0.0434 (8)
C9	0.2984 (2)	−0.0278 (5)	0.5105 (2)	0.0411 (8)
C10	0.2372 (2)	−0.1701 (5)	0.5499 (2)	0.0463 (9)
H10A	0.1692	−0.1493	0.5501	0.056*
C11	0.2759 (3)	−0.3416 (5)	0.5887 (2)	0.0491 (10)
H11A	0.2350	−0.4357	0.6158	0.059*
C12	0.3767 (3)	−0.3693 (5)	0.5861 (2)	0.0457 (9)
C13	0.4388 (3)	−0.2323 (5)	0.5472 (2)	0.0547 (10)
H13A	0.5066	−0.2559	0.5458	0.066*
C14	0.3995 (2)	−0.0590 (5)	0.5103 (2)	0.0508 (9)
H14A	0.4411	0.0369	0.4851	0.061*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0778 (7)	0.0595 (6)	0.0642 (6)	−0.0179 (5)	0.0002 (6)	0.0100 (6)
Cl2	0.0403 (4)	0.0675 (6)	0.1363 (11)	−0.0026 (5)	−0.0083 (7)	0.0236 (7)
O1	0.0468 (14)	0.0596 (15)	0.086 (2)	0.0041 (12)	0.0112 (14)	0.0228 (15)
O2	0.089 (2)	0.0758 (19)	0.127 (3)	0.0000 (18)	0.006 (2)	0.045 (2)
O3	0.070 (2)	0.094 (2)	0.124 (3)	0.0193 (18)	−0.001 (2)	0.035 (2)
N1	0.0445 (17)	0.0621 (19)	0.089 (3)	0.0045 (15)	0.0144 (19)	0.0230 (19)
N2	0.0442 (16)	0.0428 (15)	0.0493 (18)	−0.0004 (14)	0.0019 (15)	−0.0026 (15)
N3	0.066 (2)	0.061 (2)	0.066 (2)	0.0007 (19)	−0.007 (2)	0.0065 (19)
C1	0.046 (2)	0.0437 (19)	0.065 (3)	0.0046 (14)	0.0124 (19)	−0.0034 (18)
C2	0.051 (2)	0.054 (2)	0.065 (3)	−0.0046 (18)	0.015 (2)	−0.009 (2)
C3	0.061 (2)	0.044 (2)	0.038 (2)	−0.0077 (17)	0.0005 (18)	−0.0042 (17)
C4	0.050 (2)	0.0450 (19)	0.053 (2)	0.0004 (16)	−0.0104 (18)	0.0052 (18)
C5	0.0418 (18)	0.0520 (19)	0.052 (2)	−0.0054 (16)	−0.0026 (17)	−0.0072 (19)
C6	0.0440 (18)	0.0357 (15)	0.046 (2)	0.0000 (13)	0.0014 (17)	−0.0082 (18)
C7	0.0402 (18)	0.0428 (17)	0.044 (2)	−0.0013 (16)	0.0029 (17)	−0.0085 (17)
C8	0.0472 (19)	0.0424 (17)	0.040 (2)	−0.0011 (17)	0.0037 (19)	−0.0072 (16)
C9	0.0462 (19)	0.0400 (17)	0.0370 (19)	0.0007 (15)	0.0012 (17)	−0.0081 (16)
C10	0.0367 (18)	0.052 (2)	0.050 (2)	0.0023 (16)	0.0099 (17)	−0.0048 (18)

C11	0.054 (2)	0.0464 (19)	0.047 (2)	−0.0071 (16)	0.0106 (19)	0.0000 (18)
C12	0.050 (2)	0.0432 (19)	0.043 (2)	−0.0008 (16)	−0.0008 (17)	0.0002 (17)
C13	0.045 (2)	0.059 (2)	0.060 (3)	−0.0018 (18)	−0.0032 (19)	0.006 (2)
C14	0.044 (2)	0.053 (2)	0.055 (2)	−0.0082 (16)	0.0048 (18)	0.0032 (19)

Geometric parameters (Å, º)

Cl1—C3	1.732 (3)	C4—C5	1.373 (5)
Cl2—C5	1.739 (3)	C4—H4A	0.93
O1—C7	1.352 (4)	C5—C6	1.392 (4)
O1—N1	1.403 (4)	C6—C7	1.458 (4)
O2—N3	1.207 (4)	C8—C9	1.468 (4)
O3—N3	1.218 (4)	C9—C14	1.384 (4)
N1—C8	1.302 (5)	C9—C10	1.390 (4)
N2—C7	1.289 (4)	C10—C11	1.378 (4)
N2—C8	1.366 (4)	C10—H10A	0.93
N3—C12	1.474 (5)	C11—C12	1.377 (5)
C1—C2	1.383 (5)	C11—H11A	0.93
C1—C6	1.398 (4)	C12—C13	1.371 (5)
C1—H1A	0.93	C13—C14	1.379 (5)
C2—C3	1.380 (5)	C13—H13A	0.93
C2—H2A	0.93	C14—H14A	0.93
C3—C4	1.381 (5)		

C7—O1—N1	106.2 (2)	N2—C7—O1	112.3 (3)
C8—N1—O1	103.8 (3)	N2—C7—C6	127.0 (3)
C7—N2—C8	103.8 (3)	O1—C7—C6	120.7 (3)
O2—N3—O3	123.6 (4)	N1—C8—N2	113.9 (3)
O2—N3—C12	118.2 (3)	N1—C8—C9	122.5 (3)
O3—N3—C12	118.2 (4)	N2—C8—C9	123.5 (3)
C2—C1—C6	122.1 (3)	C14—C9—C10	119.4 (3)
C2—C1—H1A	119.0	C14—C9—C8	119.0 (3)
C6—C1—H1A	119.0	C10—C9—C8	121.6 (3)
C3—C2—C1	118.4 (3)	C11—C10—C9	121.0 (3)
C3—C2—H2A	120.8	C11—C10—H10A	119.5
C1—C2—H2A	120.8	C9—C10—H10A	119.5
C2—C3—C4	121.3 (3)	C10—C11—C12	118.0 (3)
C2—C3—Cl1	119.6 (3)	C10—C11—H11A	121.0
C4—C3—Cl1	119.1 (3)	C12—C11—H11A	121.0
C5—C4—C3	119.1 (3)	C13—C12—C11	122.3 (3)
C5—C4—H4A	120.4	C13—C12—N3	118.4 (3)
C3—C4—H4A	120.4	C11—C12—N3	119.3 (3)
C4—C5—C6	122.0 (3)	C12—C13—C14	119.2 (3)
C4—C5—Cl2	115.8 (3)	C12—C13—H13A	120.4
C6—C5—Cl2	122.1 (3)	C14—C13—H13A	120.4
C5—C6—C1	117.0 (3)	C13—C14—C9	120.1 (3)
C5—C6—C7	127.1 (3)	C13—C14—H14A	120.0
C1—C6—C7	115.8 (3)	C9—C14—H14A	120.0

C7—O1—N1—C8	−0.2 (4)	O1—N1—C8—N2	0.1 (4)
C6—C1—C2—C3	−0.3 (5)	O1—N1—C8—C9	−177.5 (3)
C1—C2—C3—C4	0.4 (5)	C7—N2—C8—N1	0.2 (4)
C1—C2—C3—Cl1	179.9 (3)	C7—N2—C8—C9	177.7 (3)
C2—C3—C4—C5	−0.6 (5)	N1—C8—C9—C14	176.5 (4)
Cl1—C3—C4—C5	179.9 (3)	N2—C8—C9—C14	−0.8 (5)
C3—C4—C5—C6	0.7 (5)	N1—C8—C9—C10	−2.4 (5)
C3—C4—C5—Cl2	178.3 (3)	N2—C8—C9—C10	−179.7 (3)
C4—C5—C6—C1	−0.6 (5)	C14—C9—C10—C11	0.0 (5)
Cl2—C5—C6—C1	−178.0 (3)	C8—C9—C10—C11	178.9 (3)
C4—C5—C6—C7	178.5 (3)	C9—C10—C11—C12	−1.0 (5)
Cl2—C5—C6—C7	1.0 (5)	C10—C11—C12—C13	0.6 (5)
C2—C1—C6—C5	0.4 (5)	C10—C11—C12—N3	−178.3 (3)
C2—C1—C6—C7	−178.8 (3)	O2—N3—C12—C13	171.1 (4)
C8—N2—C7—O1	−0.3 (4)	O3—N3—C12—C13	−9.9 (6)
C8—N2—C7—C6	179.1 (3)	O2—N3—C12—C11	−9.9 (5)
N1—O1—C7—N2	0.4 (4)	O3—N3—C12—C11	169.0 (4)
N1—O1—C7—C6	−179.1 (3)	C11—C12—C13—C14	0.8 (6)
C5—C6—C7—N2	−174.0 (4)	N3—C12—C13—C14	179.7 (3)
C1—C6—C7—N2	5.0 (5)	C12—C13—C14—C9	−1.8 (6)
C5—C6—C7—O1	5.3 (5)	C10—C9—C14—C13	1.4 (6)
C1—C6—C7—O1	−175.6 (3)	C8—C9—C14—C13	−177.5 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C13—H13*A*···N1^{i}	0.93	2.54	3.338 (5)	144

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