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3-(2,4-Dichlorophenoxy)-1-(4-methoxybenzyl)-4-(4-nitrophenyl)azetidin-2-one

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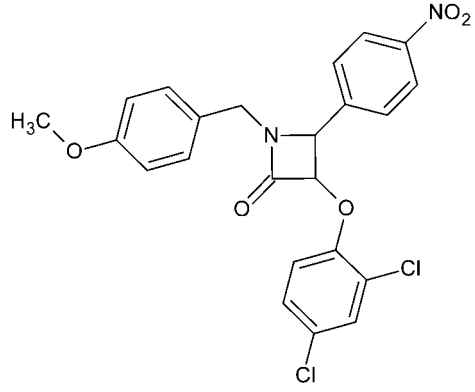
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.042; wR factor = 0.061; data-to-parameter ratio = 14.5.

The β-lactam ring of the title compound, $C_{23}H_{18}Cl_2N_2O_5$, is nearly planar [maximum deviation = 0.019 (2) Å for the N atom] and its mean plane makes dihedral angles of 56.86 (15), 68.83 (15) and 83.75 (15)° with the dichloro-, nitro- and methoxy-substituted benzene rings, respectively. In the crystal, molecules are linked by pairs of C—H···O hydrogen bonds, forming inversion dimers with $R_2^2(10)$ loops. The dimers are linked by further C—H···O hydrogen bonds, forming sheets lying parallel to (001). The molecular packing is further stabilized by C—H···π interactions.

Related literature

For general background to β-lactams, see: Schunk & Enders (2000); France *et al.* (2004); Pitts & Lectka (2014); Arya *et al.* (2014); Banik *et al.* (2003); Delpiccolo *et al.* (2003); Hodous & Fu (2002). For the crystal structures of some β-lactams, see: Akkurt *et al.* (2011); Butcher *et al.* (2011).



Experimental

Crystal data

$C_{23}H_{18}Cl_2N_2O_5$
M_r = 473.29
Monoclinic, $P2_1/n$
a = 5.0716 (5) Å
b = 20.9390 (12) Å
c = 20.1516 (18) Å
β = 96.457 (7)°
V = 2126.4 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.35 mm^{-1}
T = 296 K
0.59 × 0.28 × 0.06 mm

Data collection

Stoe IPDS 2 diffractometer
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.901, T_{max} = 0.972
15060 measured reflections
4179 independent reflections
2123 reflections with $I > 2\sigma(I)$
R_{int} = 0.062

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.061
S = 0.85
4179 reflections
289 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

Cg is the centroid of the C17–C22 benzene ring.

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O1^{i}	0.98	2.58	3.417 (3)	143
C6—H6···O5^{ii}	0.93	2.57	3.328 (3)	139
C12—H12···O3^{iii}	0.93	2.57	3.495 (4)	176
C16—H16*A*···*Cg*^{iv}	0.97	2.70	3.649 (3)	166

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

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS2013* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *WinGX* (Farrugia, 2012).

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Supporting information for this paper is available from the IUCr electronic archives (Reference: SU2747).

References

Akkurt, M., Türktekin, S., Jarrahpour, A., Badrabady, S. A. T. & Büyükgüngör, O. (2011). *Acta Cryst.* E**67**, o183.
Arya, N., Jagdale, A. Y., Patil, T. A., Yeramwar, S. S., Holikatti, S. S., Dwivedi, J., Shishoo, Ch. J. & Jain, K. S. (2014). *Eur. J. Med. Chem.* **74**, 619–656.
Banik, I., Becker, F. F. & Banik, B. K. (2003). *J. Med. Chem.* **46**, 12–15.
Butcher, R. J., Akkurt, M., Jarrahpour, A. & Badrabady, S. A. T. (2011). *Acta Cryst.* E**67**, o1101–o1102.
Delpiccolo, C. M. L., Fraga, M. A. & Mata, E. G. (2003). *J. Combin. Chem.* **5**, 208–210.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
France, S., Weatherwax, A., Taggi, A. & Lectka, T. (2004). *Acc. Chem. Res.* **37**, 592–600.

Hodous, B. L. & Fu, G. C. (2002). *J. Am. Chem. Soc.* **124**, 1578–1579.
Pitts, C. R. & Lectka, T. (2014). *Chem. Rev.* (Epub ahead of print). PMID: 24555548 [PubMed - as supplied by publisher]
Schunk, S. & Enders, D. (2000). *Org. Lett.* **2**, 907–910.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Stoe & Cie (2002). *X-AREA* and *X-RED32*. Stoe & Cie, Darmstadt, Germany.

supporting information

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3-(2,4-Dichlorophenoxy)-1-(4-methoxybenzyl)-4-(4-nitrophenyl)azetidin-2-one

Zeliha Atioğlu, Mehmet Akkurt, Aliasghar Jarrahpour, Roghayeh Heiran and Namık Özdemir

1. Comment

The *β*-lactam (2-azetidinone) ring is the most well known heterocycle to have been studied during the last century (Pitts & Lectka, 2014; France *et al.*, 2004; Arya *et al.*, 2014). The *β*-lactam framework is the structural element of a large class of broad-spectrum antibiotics such as penicillins, cephalosporins and monobactams (Delpiccolo *et al.*, 2003; Schunk & Enders, 2000; Banik *et al.*, 2003), that effectively combat bacterial infections (Schunk & Enders, 2000). However, the need for new antibiotics has been growing, as a result of the rapid emergence of bacterial strains′ resistance to traditional drugs (Hodous & Fu, 2002; Delpiccolo *et al.*, 2003). Therefore, in continuation of our research on the synthesis of *β*-lactams, we describe herein the synthesis and crystal structure of the title compound.

In the title molecule, Fig. 1, the *β*-lactam ring (N1/C1–C3) is nearly planar with a maximum deviation of -0.016 (1) Å for atom N1. The mean plane of this four-membered *β*-lactam ring is twisted from the planes of the dichloro-, nitro- and methoxy substituted benzene rings, making the dihedral angles of 56.86 (15), 68.83 (15) and 83.75 (15)°, respectively. The bond lengths and bond angles are within normal values and are comparable with those reported for similar compounds (Akkurt *et al.*, 2011; Butcher *et al.*, 2011).

In the crystal, molecules are linked by a pair of C—H···O hydrogen bonds forming inversion dimers with $R^2_2(10)$ loops (Table 1 and Fig. 2). The dimers are linked by further C-H···O hydrogen bonds forming sheets lying parallel to (001). The molecular packing is further stabilized by C—H···π interactions (Table 1).

2. Experimental

A mixture of *N*-(4-nitrobenzylidene) (4-methoxyphenyl) methanamine (0.27 g, 1.00 mmol), 2,4-dichlorophenoxyacetic acid (0.34 g, 1.50 mmol), tosyl chloride (0.28 g, 1.50 mmol) and triethylamine (0.25 g, 2.50 mmol) in dry CH_2Cl_2 was stirred at room temperature overnight. After completion of the reaction, monitored by TLC, the mixture was washed with HCl (1 N), saturated sodium bicarbonate solution, brine, dried over anhydrous Na_2SO_4 and the solvent was then evaporated under vacuum to afford the crude product. This was purified by recrystallization from EtOAc giving pale yellow prismatic crystals on slow evaporation of the solvent (yield 72%). M.p. 397 - 399 K. Spectroscopic data for the title compound are given in the archived CIF.

3. Refinement

All the H atoms were positioned geometrically and refined using a riding model: C—H = 0.93 - 0.98 Å with U_{iso}(H) = 1.2U_{eq}(C).

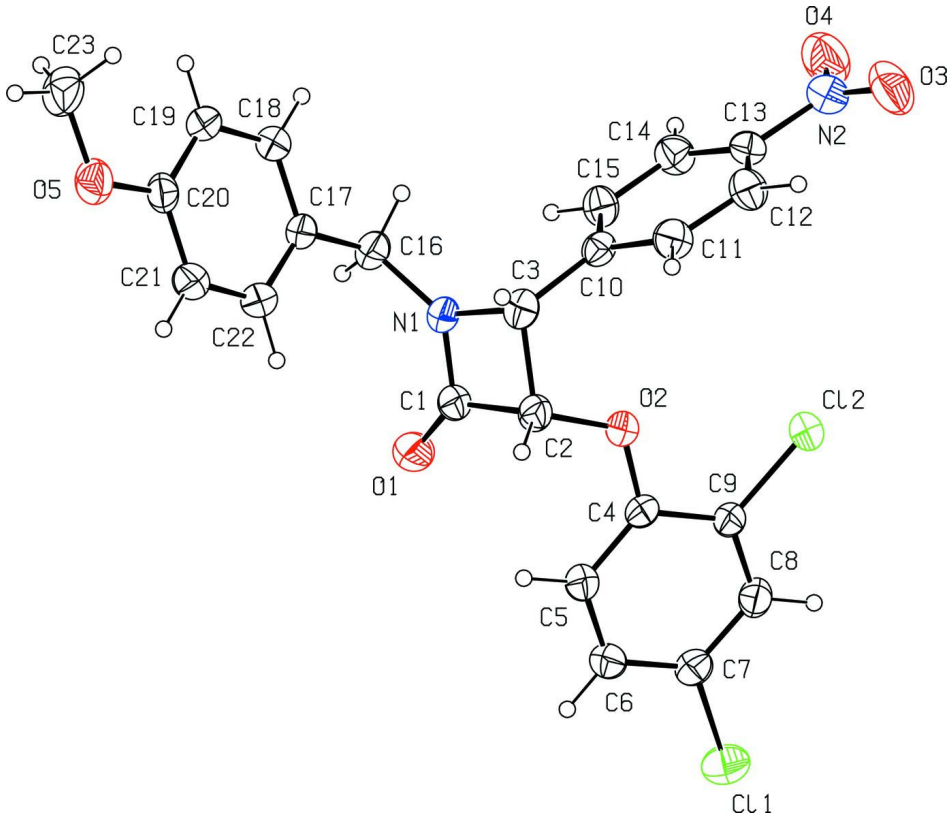

Figure 1

The molecular structure of the title molecule, with atom labelling. Displacement ellipsoids are drawn at the 30% probability level.

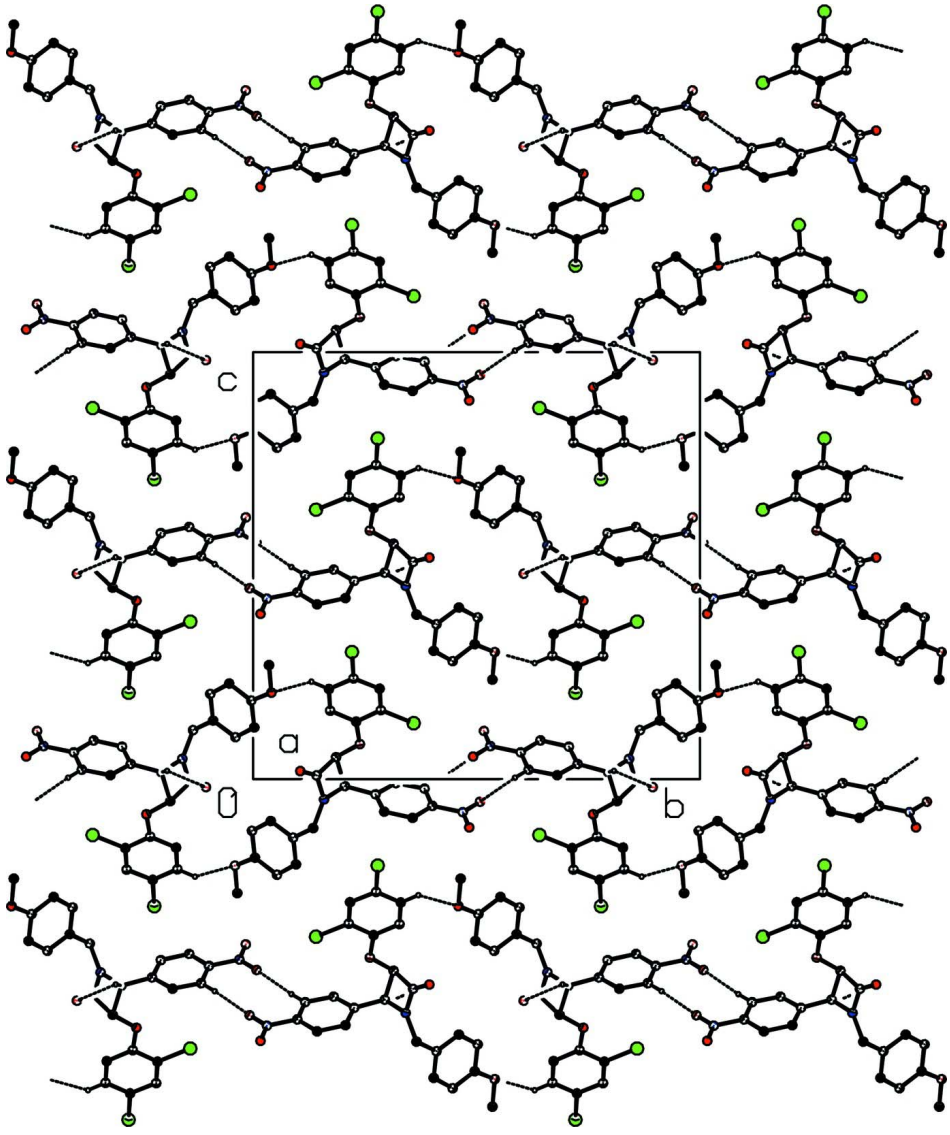

Figure 2

A view along the *a* axis of the crystal packing of the title compound. Hydrogen bonds are shown as dashed lines (see Table 1 for details).

3-(2,4-Dichlorophenoxy)-1-(4-methoxybenzyl)-4-(4-nitrophenyl)azetidin-2-one

Crystal data

$C_{23}H_{18}Cl_2N_2O_5$
M_r = 473.29
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 5.0716 (5) Å
b = 20.9390 (12) Å
c = 20.1516 (18) Å
β = 96.457 (7)°
V = 2126.4 (3) Å^3
Z = 4
$F(000)$ = 976
D_x = 1.478 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 10788 reflections
θ = 1.4–28.4°
μ = 0.35 mm^{-1}
T = 296 K
Prism, pale yellow
0.59 × 0.28 × 0.06 mm

Data collection

Stoe IPDS 2 diffractometer
Radiation source: sealed X-ray tube, 12 x 0.4 mm long-fine focus
Plane graphite monochromator
Detector resolution: 6.67 pixels mm^{-1}
ω scans
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.901, T_{max} = 0.972
15060 measured reflections
4179 independent reflections
2123 reflections with $I > 2\sigma(I)$
R_{int} = 0.062
θ_{max} = 26.0°, θ_{min} = 1.4°
$h = -6 \rightarrow 6$
$k = -25 \rightarrow 25$
$l = -24 \rightarrow 24$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.061
S = 0.85
4179 reflections
289 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0162P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e $Å^{-3}$
$\Delta\rho_{min}$ = −0.19 e $Å^{-3}$

Special details

Experimental. Spectroscopic data for the title compound:
IR (KBr, cm^{-1}): 1758 (CO β-lactam), 1352, 1520 (NO_2). ^{1}H-NMR ($CDCl_3$) δ (p.p.m.): 3.78 (OMe, s, 3H), 3.94 (CH_2, d, J =14.6 Hz, 1H), 4.78 (CH_2, d, J = 14.6 Hz, 1H), 4.85 (H-4, d, J = 4.8 Hz, 1H), 5.38 (H-3, d, J = 4.8 Hz, 1H), 6.80 (ArH, d, J = 8.7 Hz, 2H), 7.00 (ArH, d, J = 8.8 Hz, 1H), 7.03 (ArH, d, J = 8.7 Hz, 2H), 7.08 (ArH, d, J = 8.8 Hz, 1H), 7.19 (ArH, s, 1H), 7.45 (ArH, d, J = 8.8 Hz, 2H), 8.17 (ArH, d, J = 8.8 Hz, 2H). ^{13}C-NMR ($CDCl_3$) δ (p.p.m.): 44.4 (CH_2), 55.3 (OMe) 59.8 (C-4), 82.5 (C-3), 114.3, 116.2, 123.4, 123.8, 125.8, 127.5, 127.7, 129.4, 130.0, 130.1, 140.3, 148.1, 151.1, 159.5 (aromatic carbon), 164.36 (CO β-lactam). MS m/z = 472 [M^+].

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted R-factors wR and all goodnesses 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 observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating -R-factor-obs *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	0.91503 (14)	0.22124 (4)	0.29769 (3)	0.0722 (3)
Cl2	0.23669 (16)	0.35928 (3)	0.13099 (3)	0.0705 (3)
O1	0.2489 (4)	0.10566 (8)	0.01887 (8)	0.0716 (7)
O2	0.0396 (3)	0.23885 (7)	0.08223 (8)	0.0598 (6)
O3	−0.2578 (5)	0.50783 (10)	−0.05297 (12)	0.1058 (10)
O4	0.0490 (5)	0.48354 (10)	−0.11293 (12)	0.1037 (10)
O5	−0.9069 (4)	−0.04073 (8)	−0.20251 (9)	0.0712 (7)
N1	−0.0881 (4)	0.15859 (8)	−0.04739 (9)	0.0501 (7)
N2	−0.1189 (6)	0.46924 (12)	−0.07734 (13)	0.0730 (11)
C1	0.0631 (5)	0.14198 (12)	0.00884 (11)	0.0488 (9)
C2	−0.0932 (5)	0.18554 (11)	0.05155 (11)	0.0499 (9)

C3	−0.2519 (5)	0.20533 (11)	−0.01636 (11)	0.0493 (8)
C4	0.2330 (5)	0.23048 (11)	0.13478 (11)	0.0484 (9)
C5	0.3203 (5)	0.17250 (12)	0.16032 (11)	0.0560 (9)
C6	0.5282 (5)	0.16962 (12)	0.21112 (11)	0.0559 (10)
C7	0.6458 (5)	0.22470 (13)	0.23584 (11)	0.0518 (9)
C8	0.5564 (5)	0.28303 (12)	0.21190 (11)	0.0561 (9)
C9	0.3495 (5)	0.28593 (11)	0.16161 (10)	0.0493 (9)
C10	−0.2222 (4)	0.27389 (11)	−0.03557 (10)	0.0438 (8)
C11	−0.3762 (5)	0.31962 (12)	−0.00929 (12)	0.0576 (10)
C12	−0.3445 (6)	0.38355 (13)	−0.02211 (13)	0.0638 (11)
C13	−0.1588 (6)	0.40110 (12)	−0.06275 (12)	0.0536 (10)
C14	−0.0052 (5)	0.35691 (13)	−0.09064 (12)	0.0588 (10)
C15	−0.0372 (5)	0.29338 (12)	−0.07617 (11)	0.0547 (9)
C16	−0.0924 (5)	0.13434 (12)	−0.11484 (10)	0.0558 (9)
C17	−0.3177 (5)	0.08960 (11)	−0.13711 (11)	0.0465 (9)
C18	−0.4539 (5)	0.09594 (11)	−0.19956 (11)	0.0526 (9)
C19	−0.6509 (5)	0.05349 (12)	−0.22383 (11)	0.0540 (9)
C20	−0.7172 (5)	0.00404 (12)	−0.18411 (13)	0.0529 (9)
C21	−0.5827 (6)	−0.00258 (12)	−0.12111 (13)	0.0647 (10)
C22	−0.3856 (5)	0.03948 (12)	−0.09822 (12)	0.0614 (10)
C23	−1.0492 (6)	−0.03553 (13)	−0.26693 (14)	0.0812 (12)
H2	−0.20000	0.16210	0.08110	0.0600*
H3	−0.43840	0.19230	−0.01930	0.0590*
H5	0.23980	0.13510	0.14350	0.0670*
H6	0.58760	0.13030	0.22830	0.0670*
H8	0.63500	0.32040	0.22950	0.0670*
H11	−0.50410	0.30700	0.01770	0.0690*
H12	−0.44720	0.41410	−0.00350	0.0770*
H14	0.11830	0.36960	−0.11880	0.0710*
H15	0.06820	0.26310	−0.09420	0.0660*
H16A	0.07350	0.11230	−0.11850	0.0670*
H16B	−0.10090	0.17030	−0.14530	0.0670*
H18	−0.41260	0.12980	−0.22640	0.0630*
H19	−0.73760	0.05840	−0.26670	0.0650*
H21	−0.62600	−0.03590	−0.09380	0.0780*
H22	−0.29650	0.03400	−0.05570	0.0740*
H23A	−1.17560	−0.06970	−0.27350	0.1210*
H23B	−1.14060	0.00470	−0.27080	0.1210*
H23C	−0.92780	−0.03800	−0.30010	0.1210*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0632 (5)	0.0887 (5)	0.0622 (4)	−0.0012 (4)	−0.0041 (3)	0.0047 (4)
Cl2	0.1082 (6)	0.0417 (4)	0.0589 (4)	0.0053 (4)	−0.0028 (4)	−0.0010 (3)
O1	0.0839 (15)	0.0666 (12)	0.0620 (11)	0.0211 (12)	−0.0019 (10)	−0.0016 (10)
O2	0.0835 (13)	0.0417 (10)	0.0498 (10)	−0.0014 (9)	−0.0113 (9)	−0.0032 (8)
O3	0.136 (2)	0.0596 (14)	0.1232 (19)	0.0290 (15)	0.0202 (16)	0.0043 (13)

O4	0.138 (2)	0.0701 (15)	0.1078 (18)	−0.0071 (14)	0.0353 (16)	0.0130 (13)
O5	0.0738 (13)	0.0555 (11)	0.0812 (13)	−0.0087 (11)	−0.0044 (10)	−0.0089 (10)
N1	0.0607 (14)	0.0447 (12)	0.0437 (12)	0.0053 (11)	0.0008 (11)	−0.0081 (9)
N2	0.090 (2)	0.0609 (19)	0.0651 (17)	0.0106 (17)	−0.0044 (14)	0.0040 (14)
C1	0.0589 (19)	0.0406 (14)	0.0465 (15)	−0.0059 (14)	0.0046 (14)	0.0002 (13)
C2	0.0589 (17)	0.0437 (15)	0.0474 (14)	−0.0096 (13)	0.0075 (13)	−0.0017 (12)
C3	0.0402 (15)	0.0532 (15)	0.0548 (14)	−0.0045 (13)	0.0068 (12)	−0.0074 (13)
C4	0.0645 (17)	0.0472 (15)	0.0338 (12)	0.0010 (14)	0.0067 (12)	−0.0024 (12)
C5	0.0755 (19)	0.0424 (15)	0.0492 (15)	−0.0051 (15)	0.0025 (14)	−0.0012 (12)
C6	0.072 (2)	0.0489 (16)	0.0470 (15)	0.0060 (15)	0.0072 (14)	0.0036 (12)
C7	0.0547 (17)	0.0572 (17)	0.0439 (14)	−0.0010 (15)	0.0075 (12)	−0.0019 (13)
C8	0.0727 (18)	0.0465 (16)	0.0496 (15)	−0.0083 (15)	0.0087 (13)	−0.0078 (13)
C9	0.0691 (18)	0.0409 (15)	0.0381 (13)	−0.0018 (14)	0.0071 (13)	−0.0026 (11)
C10	0.0395 (14)	0.0495 (15)	0.0419 (13)	0.0010 (14)	0.0020 (11)	−0.0058 (12)
C11	0.0569 (18)	0.0614 (18)	0.0568 (15)	0.0101 (15)	0.0164 (13)	−0.0009 (14)
C12	0.068 (2)	0.0553 (18)	0.0683 (18)	0.0209 (15)	0.0080 (16)	−0.0036 (14)
C13	0.0605 (19)	0.0469 (17)	0.0505 (15)	0.0055 (15)	−0.0067 (14)	0.0030 (13)
C14	0.0599 (19)	0.0576 (17)	0.0602 (16)	0.0011 (16)	0.0123 (14)	0.0032 (14)
C15	0.0550 (17)	0.0492 (17)	0.0611 (15)	0.0059 (14)	0.0118 (14)	−0.0051 (13)
C16	0.0653 (18)	0.0551 (16)	0.0463 (14)	0.0029 (15)	0.0034 (12)	−0.0103 (13)
C17	0.0563 (17)	0.0410 (14)	0.0417 (14)	0.0041 (13)	0.0037 (12)	−0.0066 (12)
C18	0.0659 (18)	0.0450 (15)	0.0460 (15)	0.0013 (14)	0.0029 (13)	0.0012 (12)
C19	0.0637 (18)	0.0499 (16)	0.0455 (14)	0.0027 (14)	−0.0061 (13)	−0.0033 (13)
C20	0.0568 (17)	0.0396 (15)	0.0610 (17)	0.0043 (14)	0.0004 (14)	−0.0092 (13)
C21	0.087 (2)	0.0469 (15)	0.0570 (17)	−0.0076 (16)	−0.0059 (15)	0.0051 (13)
C22	0.082 (2)	0.0517 (16)	0.0470 (15)	0.0020 (16)	−0.0084 (14)	0.0000 (13)
C23	0.070 (2)	0.076 (2)	0.092 (2)	−0.0058 (17)	−0.0155 (17)	−0.0193 (17)

Geometric parameters (Å, º)

Cl1—C7	1.744 (2)	C14—C15	1.375 (4)
Cl2—C9	1.729 (2)	C16—C17	1.506 (3)
O1—C1	1.210 (3)	C17—C18	1.372 (3)
O2—C2	1.410 (3)	C17—C22	1.377 (3)
O2—C4	1.371 (3)	C18—C19	1.385 (3)
O3—N2	1.212 (4)	C19—C20	1.374 (4)
O4—N2	1.211 (4)	C20—C21	1.379 (4)
O5—C20	1.364 (3)	C21—C22	1.373 (4)
O5—C23	1.417 (3)	C2—H2	0.9800
N1—C1	1.341 (3)	C3—H3	0.9800
N1—C3	1.468 (3)	C5—H5	0.9300
N1—C16	1.449 (3)	C6—H6	0.9300
N2—C13	1.475 (4)	C8—H8	0.9300
C1—C2	1.534 (3)	C11—H11	0.9300
C2—C3	1.564 (3)	C12—H12	0.9300
C3—C10	1.499 (3)	C14—H14	0.9300
C4—C5	1.372 (3)	C15—H15	0.9300
C4—C9	1.385 (3)	C16—H16A	0.9700

C5—C6	1.386 (3)	C16—H16B	0.9700
C6—C7	1.367 (4)	C18—H18	0.9300
C7—C8	1.371 (4)	C19—H19	0.9300
C8—C9	1.376 (3)	C21—H21	0.9300
C10—C11	1.379 (3)	C22—H22	0.9300
C10—C15	1.374 (3)	C23—H23A	0.9600
C11—C12	1.376 (4)	C23—H23B	0.9600
C12—C13	1.366 (4)	C23—H23C	0.9600
C13—C14	1.370 (4)		

C2—O2—C4	120.15 (17)	O5—C20—C19	124.8 (2)
C20—O5—C23	117.9 (2)	O5—C20—C21	116.2 (2)
C1—N1—C3	96.33 (18)	C19—C20—C21	119.0 (2)
C1—N1—C16	130.5 (2)	C20—C21—C22	120.7 (2)
C3—N1—C16	133.13 (19)	C17—C22—C21	121.2 (2)
O3—N2—O4	123.7 (3)	O2—C2—H2	114.00
O3—N2—C13	117.7 (3)	C1—C2—H2	113.00
O4—N2—C13	118.6 (3)	C3—C2—H2	114.00
O1—C1—N1	131.7 (2)	N1—C3—H3	112.00
O1—C1—C2	136.2 (2)	C2—C3—H3	112.00
N1—C1—C2	92.14 (19)	C10—C3—H3	112.00
O2—C2—C1	117.7 (2)	C4—C5—H5	120.00
O2—C2—C3	110.33 (18)	C6—C5—H5	120.00
C1—C2—C3	85.08 (17)	C5—C6—H6	120.00
N1—C3—C2	86.32 (17)	C7—C6—H6	120.00
N1—C3—C10	116.77 (19)	C7—C8—H8	120.00
C2—C3—C10	115.03 (19)	C9—C8—H8	120.00
O2—C4—C5	125.1 (2)	C10—C11—H11	119.00
O2—C4—C9	115.5 (2)	C12—C11—H11	119.00
C5—C4—C9	119.3 (2)	C11—C12—H12	121.00
C4—C5—C6	120.1 (2)	C13—C12—H12	121.00
C5—C6—C7	119.8 (2)	C13—C14—H14	121.00
Cl1—C7—C6	120.0 (2)	C15—C14—H14	121.00
Cl1—C7—C8	119.3 (2)	C10—C15—H15	119.00
C6—C7—C8	120.7 (2)	C14—C15—H15	119.00
C7—C8—C9	119.5 (2)	N1—C16—H16A	108.00
Cl2—C9—C4	119.74 (18)	N1—C16—H16B	108.00
Cl2—C9—C8	119.78 (18)	C17—C16—H16A	108.00
C4—C9—C8	120.5 (2)	C17—C16—H16B	108.00
C3—C10—C11	119.2 (2)	H16A—C16—H16B	107.00
C3—C10—C15	122.2 (2)	C17—C18—H18	119.00
C11—C10—C15	118.5 (2)	C19—C18—H18	119.00
C10—C11—C12	121.4 (2)	C18—C19—H19	120.00
C11—C12—C13	118.5 (3)	C20—C19—H19	120.00
N2—C13—C12	119.9 (2)	C20—C21—H21	120.00
N2—C13—C14	118.3 (2)	C22—C21—H21	120.00
C12—C13—C14	121.8 (2)	C17—C22—H22	119.00
C13—C14—C15	118.7 (2)	C21—C22—H22	119.00

C10—C15—C14	121.2 (2)	O5—C23—H23A	109.00
N1—C16—C17	115.36 (19)	O5—C23—H23B	109.00
C16—C17—C18	120.0 (2)	O5—C23—H23C	109.00
C16—C17—C22	122.3 (2)	H23A—C23—H23B	110.00
C18—C17—C22	117.6 (2)	H23A—C23—H23C	110.00
C17—C18—C19	122.1 (2)	H23B—C23—H23C	109.00
C18—C19—C20	119.5 (2)		

C2—O2—C4—C5	2.3 (3)	O2—C4—C5—C6	176.2 (2)
C4—O2—C2—C1	−71.2 (3)	C5—C4—C9—C8	2.1 (4)
C4—O2—C2—C3	−166.47 (19)	O2—C4—C9—Cl2	3.1 (3)
C2—O2—C4—C9	−179.5 (2)	C9—C4—C5—C6	−1.9 (4)
C23—O5—C20—C21	−180.0 (2)	C4—C5—C6—C7	0.1 (4)
C23—O5—C20—C19	−1.0 (4)	C5—C6—C7—Cl1	−177.73 (19)
C16—N1—C1—C2	−173.9 (2)	C5—C6—C7—C8	1.4 (4)
C3—N1—C1—O1	−177.6 (3)	C6—C7—C8—C9	−1.2 (4)
C1—N1—C16—C17	103.0 (3)	Cl1—C7—C8—C9	177.94 (18)
C3—N1—C16—C17	−72.5 (3)	C7—C8—C9—Cl2	−179.75 (19)
C16—N1—C3—C10	−70.0 (3)	C7—C8—C9—C4	−0.5 (4)
C1—N1—C3—C2	−2.85 (19)	C3—C10—C15—C14	−177.0 (2)
C16—N1—C3—C2	173.8 (2)	C3—C10—C11—C12	175.9 (2)
C3—N1—C1—C2	2.90 (19)	C11—C10—C15—C14	−0.1 (3)
C1—N1—C3—C10	113.4 (2)	C15—C10—C11—C12	−1.1 (4)
C16—N1—C1—O1	5.7 (5)	C10—C11—C12—C13	1.1 (4)
O4—N2—C13—C14	−0.5 (4)	C11—C12—C13—N2	−179.8 (2)
O3—N2—C13—C12	−1.0 (4)	C11—C12—C13—C14	0.0 (4)
O4—N2—C13—C12	179.2 (3)	N2—C13—C14—C15	178.7 (2)
O3—N2—C13—C14	179.2 (3)	C12—C13—C14—C15	−1.1 (4)
N1—C1—C2—C3	−2.72 (18)	C13—C14—C15—C10	1.2 (4)
O1—C1—C2—O2	67.4 (4)	N1—C16—C17—C22	−47.4 (3)
O1—C1—C2—C3	177.8 (3)	N1—C16—C17—C18	136.2 (2)
N1—C1—C2—O2	−113.1 (2)	C16—C17—C22—C21	−176.6 (2)
C1—C2—C3—N1	2.49 (16)	C18—C17—C22—C21	−0.1 (4)
O2—C2—C3—C10	2.4 (3)	C16—C17—C18—C19	175.8 (2)
C1—C2—C3—C10	−115.4 (2)	C22—C17—C18—C19	−0.8 (4)
O2—C2—C3—N1	120.25 (19)	C17—C18—C19—C20	1.3 (4)
C2—C3—C10—C15	94.1 (3)	C18—C19—C20—C21	−0.9 (4)
N1—C3—C10—C15	−4.9 (3)	C18—C19—C20—O5	−179.9 (2)
C2—C3—C10—C11	−82.7 (3)	O5—C20—C21—C22	179.1 (2)
N1—C3—C10—C11	178.3 (2)	C19—C20—C21—C22	0.0 (4)
C5—C4—C9—Cl2	−178.70 (19)	C20—C21—C22—C17	0.5 (4)
O2—C4—C9—C8	−176.2 (2)		

Hydrogen-bond geometry (Å, º)

Cg is the centroid of the C17–C22 benzene ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O1^{i}	0.98	2.58	3.417 (3)	143

C6—H6···O5^{ii}	0.93	2.57	3.328 (3)	139
C12—H12···O3^{iii}	0.93	2.57	3.495 (4)	176
C16—H16*A*···*Cg*^{iv}	0.97	2.70	3.649 (3)	166

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