Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-(1*H*-1,2,3-Benzotriazol-1-yl)-1-(2-fluorobenzoyl)ethyl 2-chlorobenzoate

Wu-Lan Zeng,* Yan Pang and Hua-Xiang Zhang

Department of Chemistry and Chemical Engineering, Weifang University, Weifang 261061, People's Republic of China
Correspondence e-mail: wulanzeng@163.com

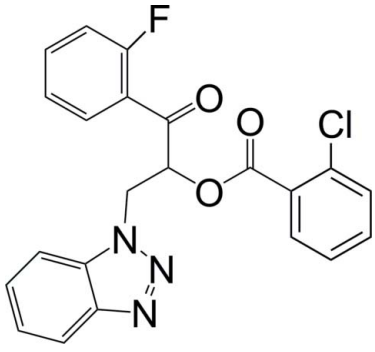
Received 30 May 2007; accepted 1 June 2007

Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.006 Å; R factor = 0.061; wR factor = 0.208; data-to-parameter ratio = 14.7.

In the crystal structure of the title compound, $C_{22}H_{15}ClFN_3O_3$, the crystal packing is controlled by van der Waals forces. Weak intramolecular C—H···O and C—H···F hydrogen bonds link the molecules into chains extended along the c axis.The packing is further stabilized by van der Waals forces.

Related literature

For background, see: Chen & Wu (2005). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{22}H_{15}ClFN_3O_3$
M_r = 423.82
Monoclinic, $P2_1/c$
a = 10.858 (3) Å
b = 8.279 (2) Å
c = 22.236 (6) Å
β = 100.599 (5)°
V = 1964.9 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 294 (2) K
0.20 × 0.16 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.955, T_{max} = 0.977
10896 measured reflections
3993 independent reflections
1838 reflections with $I > 2\sigma(I)$
R_{int} = 0.050

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.208
S = 1.01
3993 reflections
271 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2441).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bruker (1997). *SMART*, *SAINT*, *SADABS* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Chen, Z.-Y. & Wu, M.-J. (2005). *Org. Lett.* **7**, 475–477.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

  doi:10.1107/S1600536807026980

supplementary materials

Acta Cryst. (2007). E**63**, o3256 [**doi:10.1107/S1600536807026980**]

2-(1*H*-1,2,3-Benzotriazol-1-yl)-1-(2-fluorobenzoyl)ethyl 2-chlorobenzoate

W.-L. Zeng, Y. Pang and H.-X. Zhang

Comment

1*H*-Benzotriazole and its derivatives exhibit a broad spectrum of pharmacological activities such as antifungal, antitumor and antineoplastic activities (Chen & Wu, 2005). We report here the synthesis and structure of the title compound, (I) (Fig. 1), as part of our ongoing studies on new benzotriazole compounds with higher bioactivity.

All the bond lengths and angles in (I) are within their normal ranges (Allen *et al.*, 1987). The benzotriazole ring system is essentially planar, with a dihedral angle of 0.9 (1)° between the triazole ring (atoms N1—N3/C1/C6) and the C1—C6 benzene ring. The dihedral angles between the mean planes of the benzotriazole system and the C10—C15 and C17—C22 aromatic rings are 5.21 (1)° and 61.81 (1)°, respectively. The dihedral angle between rings C10—C15 and C17—C22 is 57.7 (2)°. Molecule (I) is chiral. In the arbitrarily chosen asymmetric molecule, C8 has *R* configuration, but crystal symmetry generates a racemic mixture.

Experimental

Bromine (3.2 g, 0.02 mol) was added dropwise to a solution of 3-(1*H*-benzo[*d*][1,2,3]triazol-1-yl)-1-(2-fluorophenyl)propan-1-one (5.38 g, 0.02 mol) and sodium acetate (1.6 g, 0.02 mol) in acetic acid (50 ml). The reaction proceeded for 7 h. Water (50 ml) and chloroform (20 ml) were then added. The organic layer was washed successively with saturated sodium bicarbonate solution and brine, dried over anhydrous magnesium sulfate and the chloroform solution filtered. It was cooled with ice-water, and then an acetone solution (10 ml) of 2-chlorobenzoic acid (3.1 g, 0.02 mol) and triethylamine (2.8 ml) was added. The mixture was stirred with ice–water for 6 h. The solution was then filtered and concentrated. Single crystals of (I) were obtained by slow evaporation of an acetone-ethylacetate (1:1 *v*/*v*) solution at room temperature over a period of one week.

Refinement

The H atoms were geometrically placed (C—H = 0.93–0.97 Å), and refined as riding with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

Figures

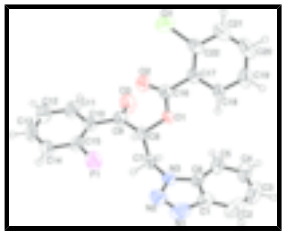


Fig. 1. The molecular structure of (I), drawn with 30% probability ellipsoids (arbitrary spheres for the H atoms).

2-(1<it>*H*</it>-1,2,3-Benzotriazol-1-yl)-1-(2-fluorobenzoyl)ethyl 2-chlorobenzoate

Crystal data

$C_{22}H_{15}ClFN_3O_3$	F_{000} = 872
M_r = 423.82	D_x = 1.433 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 1651 reflections
a = 10.858 (3) Å	θ = 2.4–21.0º
b = 8.279 (2) Å	μ = 0.23 mm^{-1}
c = 22.236 (6) Å	T = 294 (2) K
β = 100.599 (5)º	Block, colourless
V = 1964.9 (9) Å^3	0.20 × 0.16 × 0.10 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	3993 independent reflections
Radiation source: fine-focus sealed tube	1838 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.050
T = 294(2) K	θ_{max} = 26.4º
ω scans	θ_{min} = 1.9º
Absorption correction: multi-scan (SADABS; Bruker, 1997)	h = −13→10
T_{min} = 0.955, T_{max} = 0.977	k = −10→10
10896 measured reflections	l = −27→27

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.061	H-atom parameters constrained
$wR(F^2)$ = 0.208	$w = 1/[\sigma^2(F_o^2) + (0.1046P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.01	$(\Delta/\sigma)_{max}$ = 0.002
3993 reflections	$\Delta\rho_{max}$ = 0.46 e Å^{-3}
271 parameters	$\Delta\rho_{min}$ = −0.31 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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	0.16809 (12)	0.54811 (16)	1.00954 (5)	0.0998 (5)
F1	0.2942 (2)	0.7314 (3)	0.71581 (10)	0.1010 (9)
O1	0.40423 (19)	0.8137 (2)	0.90054 (9)	0.0485 (6)
O2	0.2520 (3)	0.6305 (3)	0.89726 (11)	0.0778 (8)
O3	0.1967 (3)	0.9868 (4)	0.86057 (13)	0.0912 (10)
N1	0.7216 (4)	0.7326 (5)	0.78904 (19)	0.0916 (11)
N2	0.6068 (4)	0.7844 (4)	0.77393 (14)	0.0773 (10)
N3	0.5786 (3)	0.8780 (3)	0.81969 (12)	0.0537 (7)
C1	0.7691 (4)	0.7934 (5)	0.8454 (2)	0.0678 (11)
C2	0.8887 (4)	0.7754 (6)	0.8829 (3)	0.0968 (16)
H2	0.9516	0.7151	0.8702	0.116*
C3	0.9073 (5)	0.8501 (6)	0.9380 (3)	0.0999 (16)
H3	0.9849	0.8408	0.9636	0.120*
C4	0.8128 (5)	0.9413 (5)	0.9577 (2)	0.0884 (14)
H4	0.8295	0.9884	0.9963	0.106*
C5	0.6996 (4)	0.9625 (4)	0.92264 (18)	0.0664 (11)
H5	0.6383	1.0249	0.9356	0.080*
C6	0.6785 (3)	0.8861 (4)	0.86548 (16)	0.0506 (9)
C7	0.4558 (3)	0.9480 (4)	0.81416 (15)	0.0576 (9)
H7A	0.4250	0.9761	0.7717	0.069*
H7B	0.4614	1.0466	0.8380	0.069*
C8	0.3624 (3)	0.8336 (4)	0.83592 (13)	0.0513 (9)
H8	0.3636	0.7289	0.8155	0.062*
C9	0.2313 (3)	0.9028 (4)	0.82231 (17)	0.0606 (10)
C10	0.1471 (3)	0.8742 (4)	0.76184 (15)	0.0549 (9)
C11	0.0281 (4)	0.9362 (5)	0.7543 (2)	0.0757 (12)
H11	0.0032	0.9919	0.7863	0.091*
C12	−0.0549 (4)	0.9173 (6)	0.7001 (2)	0.0919 (14)
H12	−0.1354	0.9597	0.6961	0.110*
C13	−0.0210 (4)	0.8378 (6)	0.6526 (2)	0.0818 (13)
H13	−0.0782	0.8279	0.6162	0.098*
C14	0.0958 (4)	0.7718 (5)	0.65701 (18)	0.0774 (12)
H14	0.1196	0.7160	0.6247	0.093*

C15	0.1774 (3)	0.7928 (5)	0.71290 (18)	0.0651 (10)
C16	0.3388 (4)	0.7037 (4)	0.92604 (15)	0.0539 (9)
C17	0.3902 (3)	0.6798 (4)	0.99247 (14)	0.0495 (9)
C18	0.5139 (3)	0.7262 (4)	1.01529 (15)	0.0577 (9)
H18	0.5608	0.7757	0.9895	0.069*
C19	0.5670 (4)	0.6991 (5)	1.07574 (18)	0.0751 (12)
H19	0.6492	0.7303	1.0907	0.090*
C20	0.4973 (5)	0.6255 (5)	1.11360 (18)	0.0806 (14)
H20	0.5331	0.6068	1.1543	0.097*
C21	0.3755 (5)	0.5791 (5)	1.09238 (18)	0.0735 (12)
H21	0.3291	0.5304	1.1186	0.088*
C22	0.3228 (4)	0.6054 (4)	1.03192 (16)	0.0590 (10)

Atomic displacement parameters (Å^2)

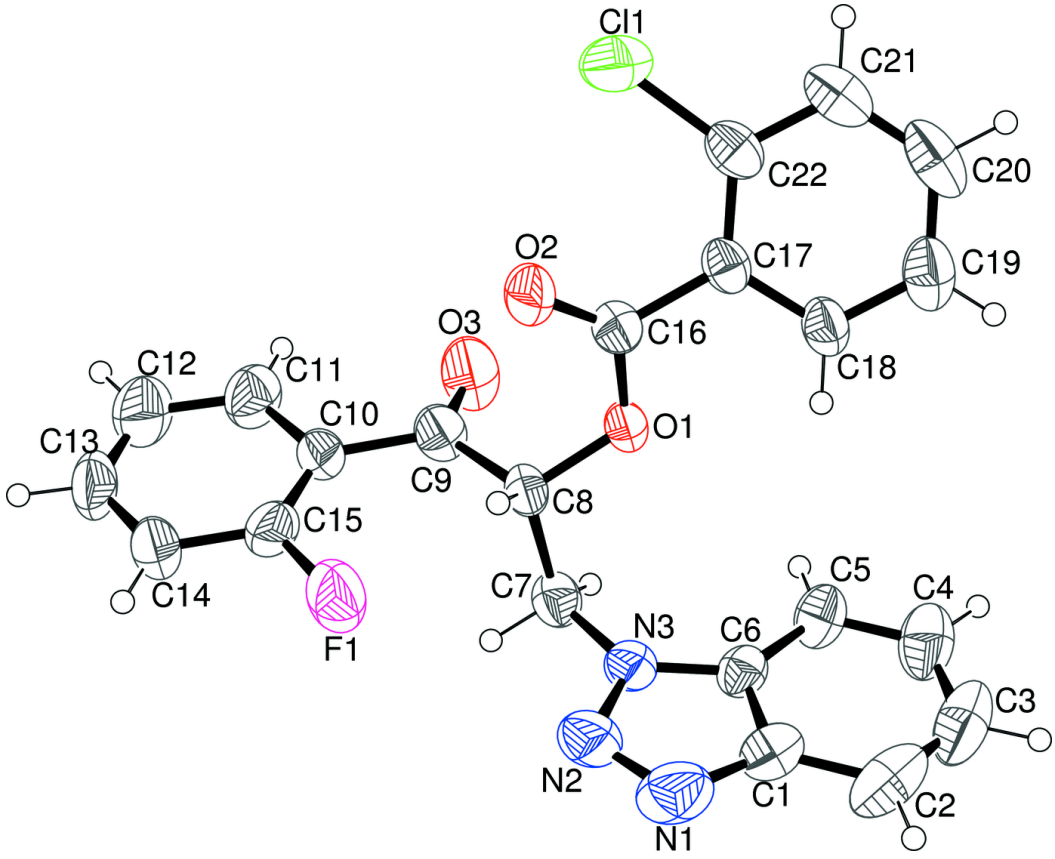
	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0918 (9)	0.1274 (11)	0.0858 (8)	−0.0368 (8)	0.0311 (7)	0.0029 (7)
F1	0.0808 (17)	0.155 (2)	0.0642 (14)	0.0226 (17)	0.0061 (12)	−0.0280 (14)
O1	0.0490 (13)	0.0555 (14)	0.0380 (12)	−0.0029 (11)	0.0005 (10)	0.0016 (10)
O2	0.0747 (19)	0.098 (2)	0.0545 (15)	−0.0333 (17)	−0.0044 (14)	−0.0018 (14)
O3	0.082 (2)	0.113 (2)	0.0746 (19)	0.0369 (18)	0.0036 (16)	−0.0210 (17)
N1	0.079 (3)	0.103 (3)	0.102 (3)	0.003 (2)	0.039 (2)	−0.023 (2)
N2	0.081 (3)	0.095 (3)	0.059 (2)	−0.007 (2)	0.0243 (19)	−0.0179 (18)
N3	0.0548 (19)	0.0581 (18)	0.0495 (17)	−0.0003 (15)	0.0132 (15)	−0.0046 (14)
C1	0.057 (3)	0.064 (2)	0.085 (3)	−0.004 (2)	0.020 (2)	0.000 (2)
C2	0.054 (3)	0.075 (3)	0.164 (5)	0.000 (2)	0.025 (3)	0.012 (3)
C3	0.060 (3)	0.086 (3)	0.139 (5)	−0.010 (3)	−0.019 (3)	0.016 (3)
C4	0.076 (3)	0.084 (3)	0.093 (3)	−0.015 (3)	−0.014 (3)	−0.009 (3)
C5	0.056 (2)	0.062 (2)	0.076 (3)	−0.0047 (19)	−0.001 (2)	−0.010 (2)
C6	0.046 (2)	0.0460 (19)	0.059 (2)	−0.0070 (17)	0.0072 (18)	0.0008 (17)
C7	0.060 (2)	0.059 (2)	0.051 (2)	0.0031 (19)	0.0035 (18)	0.0064 (17)
C8	0.049 (2)	0.063 (2)	0.0385 (18)	0.0026 (17)	0.0007 (15)	0.0003 (16)
C9	0.062 (2)	0.065 (2)	0.054 (2)	0.011 (2)	0.0083 (19)	−0.0019 (18)
C10	0.055 (2)	0.062 (2)	0.046 (2)	−0.0048 (19)	0.0041 (17)	0.0071 (17)
C11	0.060 (3)	0.081 (3)	0.083 (3)	0.012 (2)	0.003 (2)	0.008 (2)
C12	0.067 (3)	0.107 (4)	0.094 (4)	0.018 (3)	−0.005 (3)	0.019 (3)
C13	0.068 (3)	0.100 (3)	0.064 (3)	−0.014 (3)	−0.022 (2)	0.018 (2)
C14	0.075 (3)	0.099 (3)	0.051 (2)	−0.016 (3)	−0.004 (2)	0.004 (2)
C15	0.043 (2)	0.081 (3)	0.070 (3)	0.006 (2)	0.006 (2)	0.014 (2)
C16	0.054 (2)	0.061 (2)	0.047 (2)	0.0004 (19)	0.0075 (18)	−0.0006 (17)
C17	0.059 (2)	0.049 (2)	0.0394 (18)	0.0058 (17)	0.0052 (17)	−0.0060 (15)
C18	0.060 (2)	0.068 (2)	0.041 (2)	0.0046 (19)	0.0000 (18)	−0.0062 (16)
C19	0.073 (3)	0.085 (3)	0.061 (3)	0.021 (2)	−0.006 (2)	−0.017 (2)
C20	0.126 (4)	0.070 (3)	0.042 (2)	0.035 (3)	0.004 (3)	−0.001 (2)
C21	0.109 (4)	0.064 (3)	0.051 (2)	0.009 (3)	0.023 (2)	0.0048 (19)
C22	0.078 (3)	0.053 (2)	0.047 (2)	0.008 (2)	0.0142 (19)	−0.0013 (16)

Geometric parameters (Å, °)

Cl1—C22	1.728 (4)	C8—C9	1.513 (5)
F1—C15	1.357 (4)	C8—H8	0.9800
O1—C16	1.344 (4)	C9—C10	1.499 (5)
O1—C8	1.435 (4)	C10—C15	1.371 (5)
O2—C16	1.201 (4)	C10—C11	1.371 (5)
O3—C9	1.211 (4)	C11—C12	1.374 (6)
N1—N2	1.303 (5)	C11—H11	0.9300
N1—C1	1.361 (5)	C12—C13	1.353 (6)
N2—N3	1.358 (4)	C12—H12	0.9300
N3—C6	1.346 (4)	C13—C14	1.368 (6)
N3—C7	1.438 (4)	C13—H13	0.9300
C1—C6	1.385 (5)	C14—C15	1.398 (5)
C1—C2	1.416 (6)	C14—H14	0.9300
C2—C3	1.354 (7)	C16—C17	1.493 (4)
C2—H2	0.9300	C17—C22	1.386 (5)
C3—C4	1.408 (6)	C17—C18	1.400 (5)
C3—H3	0.9300	C18—C19	1.380 (5)
C4—C5	1.340 (6)	C18—H18	0.9300
C4—H4	0.9300	C19—C20	1.374 (6)
C5—C6	1.401 (5)	C19—H19	0.9300
C5—H5	0.9300	C20—C21	1.374 (6)
C7—C8	1.529 (5)	C20—H20	0.9300
C7—H7A	0.9700	C21—C22	1.378 (5)
C7—H7B	0.9700	C21—H21	0.9300
C16—O1—C8	113.9 (2)	C15—C10—C11	116.4 (3)
N2—N1—C1	107.4 (3)	C15—C10—C9	126.4 (3)
N1—N2—N3	109.4 (3)	C11—C10—C9	117.2 (4)
C6—N3—N2	109.5 (3)	C10—C11—C12	121.1 (4)
C6—N3—C7	130.6 (3)	C10—C11—H11	119.5
N2—N3—C7	119.9 (3)	C12—C11—H11	119.5
N1—C1—C6	109.3 (4)	C13—C12—C11	120.7 (4)
N1—C1—C2	130.9 (4)	C13—C12—H12	119.6
C6—C1—C2	119.9 (4)	C11—C12—H12	119.6
C3—C2—C1	116.9 (5)	C12—C13—C14	121.4 (4)
C3—C2—H2	121.6	C12—C13—H13	119.3
C1—C2—H2	121.6	C14—C13—H13	119.3
C2—C3—C4	122.1 (4)	C13—C14—C15	116.2 (4)
C2—C3—H3	118.9	C13—C14—H14	121.9
C4—C3—H3	118.9	C15—C14—H14	121.9
C5—C4—C3	122.1 (4)	F1—C15—C10	120.4 (3)
C5—C4—H4	118.9	F1—C15—C14	115.4 (4)
C3—C4—H4	118.9	C10—C15—C14	124.2 (4)
C4—C5—C6	116.7 (4)	O2—C16—O1	122.8 (3)
C4—C5—H5	121.7	O2—C16—C17	125.1 (3)
C6—C5—H5	121.7	O1—C16—C17	112.1 (3)
N3—C6—C1	104.5 (3)	C22—C17—C18	118.4 (3)

N3—C6—C5	133.2 (3)	C22—C17—C16	122.7 (3)
C1—C6—C5	122.3 (4)	C18—C17—C16	118.8 (3)
N3—C7—C8	112.4 (3)	C19—C18—C17	120.6 (4)
N3—C7—H7A	109.1	C19—C18—H18	119.7
C8—C7—H7A	109.1	C17—C18—H18	119.7
N3—C7—H7B	109.1	C20—C19—C18	119.4 (4)
C8—C7—H7B	109.1	C20—C19—H19	120.3
H7A—C7—H7B	107.8	C18—C19—H19	120.3
O1—C8—C9	110.9 (3)	C19—C20—C21	121.1 (4)
O1—C8—C7	106.4 (3)	C19—C20—H20	119.4
C9—C8—C7	110.9 (3)	C21—C20—H20	119.4
O1—C8—H8	109.5	C20—C21—C22	119.5 (4)
C9—C8—H8	109.5	C20—C21—H21	120.3
C7—C8—H8	109.5	C22—C21—H21	120.3
O3—C9—C10	120.3 (3)	C21—C22—C17	120.9 (4)
O3—C9—C8	118.6 (3)	C21—C22—Cl1	116.4 (3)
C10—C9—C8	121.1 (3)	C17—C22—Cl1	122.6 (3)
C1—N1—N2—N3	−0.1 (5)	C8—C9—C10—C15	−2.8 (6)
N1—N2—N3—C6	−0.1 (4)	O3—C9—C10—C11	−5.0 (6)
N1—N2—N3—C7	−179.1 (3)	C8—C9—C10—C11	177.6 (3)
N2—N1—C1—C6	0.2 (5)	C15—C10—C11—C12	−0.4 (6)
N2—N1—C1—C2	−179.9 (4)	C9—C10—C11—C12	179.3 (4)
N1—C1—C2—C3	−179.0 (4)	C10—C11—C12—C13	−0.4 (7)
C6—C1—C2—C3	0.9 (6)	C11—C12—C13—C14	1.0 (7)
C1—C2—C3—C4	0.1 (7)	C12—C13—C14—C15	−0.6 (6)
C2—C3—C4—C5	−1.4 (7)	C11—C10—C15—F1	178.2 (3)
C3—C4—C5—C6	1.4 (6)	C9—C10—C15—F1	−1.4 (6)
N2—N3—C6—C1	0.2 (4)	C11—C10—C15—C14	0.7 (6)
C7—N3—C6—C1	179.1 (3)	C9—C10—C15—C14	−178.9 (4)
N2—N3—C6—C5	−178.9 (4)	C13—C14—C15—F1	−177.8 (3)
C7—N3—C6—C5	0.0 (6)	C13—C14—C15—C10	−0.2 (6)
N1—C1—C6—N3	−0.2 (4)	C8—O1—C16—O2	−1.0 (5)
C2—C1—C6—N3	179.8 (3)	C8—O1—C16—C17	176.6 (2)
N1—C1—C6—C5	179.0 (3)	O2—C16—C17—C22	−19.1 (5)
C2—C1—C6—C5	−0.9 (6)	O1—C16—C17—C22	163.4 (3)
C4—C5—C6—N3	178.7 (4)	O2—C16—C17—C18	157.3 (3)
C4—C5—C6—C1	−0.3 (6)	O1—C16—C17—C18	−20.3 (4)
C6—N3—C7—C8	−92.6 (4)	C22—C17—C18—C19	−0.3 (5)
N2—N3—C7—C8	86.2 (4)	C16—C17—C18—C19	−176.9 (3)
C16—O1—C8—C9	65.4 (3)	C17—C18—C19—C20	0.1 (5)
C16—O1—C8—C7	−173.9 (3)	C18—C19—C20—C21	−0.3 (6)
N3—C7—C8—O1	66.2 (3)	C19—C20—C21—C22	0.7 (6)
N3—C7—C8—C9	−173.1 (3)	C20—C21—C22—C17	−0.9 (5)
O1—C8—C9—O3	27.6 (5)	C20—C21—C22—Cl1	−178.4 (3)
C7—C8—C9—O3	−90.3 (4)	C18—C17—C22—C21	0.7 (5)
O1—C8—C9—C10	−154.9 (3)	C16—C17—C22—C21	177.1 (3)
C7—C8—C9—C10	87.1 (4)	C18—C17—C22—Cl1	178.1 (3)
O3—C9—C10—C15	174.6 (4)	C16—C17—C22—Cl1	−5.5 (5)

Fig. 1


Cl1
C21
C22
C20
O2
C17
C19
O3
C16
C12
C11
C18
C10
O1
C13
C9
C8
C15
C5
C14
C7
C4
F1
N3
C6
C3
N2
C1
C2
N1