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1-{(1*Z*)-1-[6-(4-Chlorophenoxy)hexyloxy]-1-(2,4-difluorophenyl)prop-1-en-2-yl}-1*H*-1,2,4-triazol-4-ium nitrate

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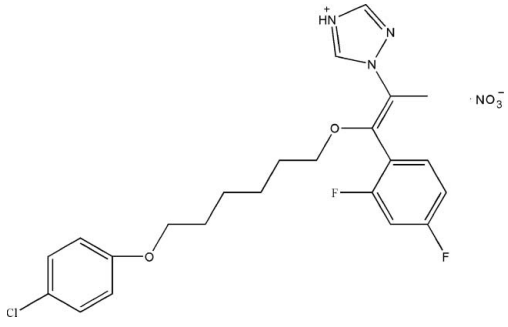
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.006 Å; R factor = 0.064; wR factor = 0.176; data-to-parameter ratio = 14.6.

In the title compound, $C_{23}H_{25}ClF_2N_3O_2^+\cdot NO_3^-$, the triazole ring makes dihedral angles of 60.9 (4) and 25.0 (3)° with the 6-chlorophenyl and 2,4-difluorophenyl rings, respectively. The molecule adopts a Z configuration about the C=C double bond. In the crystal, the cations and anions are linked by N—H···O hydrogen bonds and weak C—H···O interactions.

Related literature

For the use of triazole derivatives as antifungal agents, see: Jeu *et al.* (2003); Fromtling & Castaner (1996). For the synthesis, see: Zirngibl & Thiele (1985).



Experimental

Crystal data

$C_{23}H_{25}ClF_2N_3O_2^+\cdot NO_3^-$ M_r = 510.92
Monoclinic, $C2/c$
a = 35.538 (7) Å
b = 8.5550 (17) Å
c = 17.072 (3) Å
β = 105.21 (3)°
V = 5008.6 (17) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.21 mm^{-1}
T = 293 K
0.30 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.940, T_{max} = 0.980
9194 measured reflections
4628 independent reflections
1954 reflections with $I > 2\sigma(I)$
R_{int} = 0.063
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.176
S = 1.00
4628 reflections
317 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3A···O4^{i}	0.86	1.8	2.661 (5)	175
C22—H22A···O4^{ii}	0.93	2.38	3.077 (6)	131

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

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NC2240).

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supporting information

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1-{(1*Z*)-1-[6-(4-Chlorophenoxy)hexyloxy]-1-(2,4-difluorophenyl)prop-1-en-2-yl}-1*H*-1,2,4-triazol-4-ium nitrate

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S1. Comment

Triazole derivatives such as Voriconazole ((*2R,3S*)-2-(2,4-difluorophenyl) -3-(5-fluoropyrimidin-4-yl)-1-(1*H*-1,2,4-triazol-1-yl) butan-2-ol) and Posaconazole (4-(4-(4-(4-(((*3R,5R*)-5-(2,4-difluorophenyl)-5-(1,2,4- triazol-1-ylmethyl)-oxolan-3-yl)methoxy)phenyl)piperazin-1-yl)phenyl)- 2-((*2S,3S*)-2-hydroxypentan-3-yl)-1,2,4-triazol-3-one) are safe and effective antifungal agents. (Jeu *et al.*, 2003; Fromtling & Castaner, 1996) As part of our studies on the synthesis of new triazole derivatives, the crystal structure of the title compound was determined.

In the molecular structure of the title compound the double bond is Z configurated. In the crystal structure the anions and cations are connected via N—H···O hydrogen bonding and weak and C—H···O interactions (Table 1 and Fig. 2).

S2. Experimental

Details on the synthesis can be found in the literature reported by Zirngibl & Thiele (1985). 3 g (0.01 mol) 1-(2,4-difluorophenyl)-2-(1,2,4-triazol)-1-y1)propan-1-one, 10 g of a 50% aqueous sodium hydroxide, 15 ml toluene and 1.5 ml of a 40% aqueous solution of tetrabutyl ammonium hydroxide are mixed and heated to 323.15 K under vigorous stirring. 3.0 g (0.01 mol) 1-bromo-6- (4-chlorophenoxy)-hexane, dissolved in 10 ml toluene, is instilled into the stirred and warmed solution in the course of 10 h. The mixture is subsequently stirred for another 20 h at 323.15 K. The reaction mixture is mixed with as much water and chloroform so that the aqueous phase becomes lighter than the organic phase. Thereafter, the organic and aqueous phases are separated. The organic phase is dried with sodium sulfate. The solvents are distilled under reduced pressure. The remaining residue is a dark oil that is diluted with 10 ml 2-propanol and then adjusted to a PH-value of 2 by means of 30% aqueous nitric acid. The thus derived nitric acid solution is then cooled in the refrigerator. The impure precipitated product herein is subsequently crystallized from a 1:1 mixture of ethyl acetate and ethanol. The purified product may be analytically identified as an approximately pure *Z*-isomer of propylene nitrate. Crystals of title compound suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution.

S3. Refinement

H atoms were positioned geometrically with C—H = 0.93 and 0.97 Å for aromatic and methylene H atoms, respectively, and with N—H = 0.86 Å for triazole H atom, and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2 (or 1.5 for methyl groups) times U_{eq}(C).

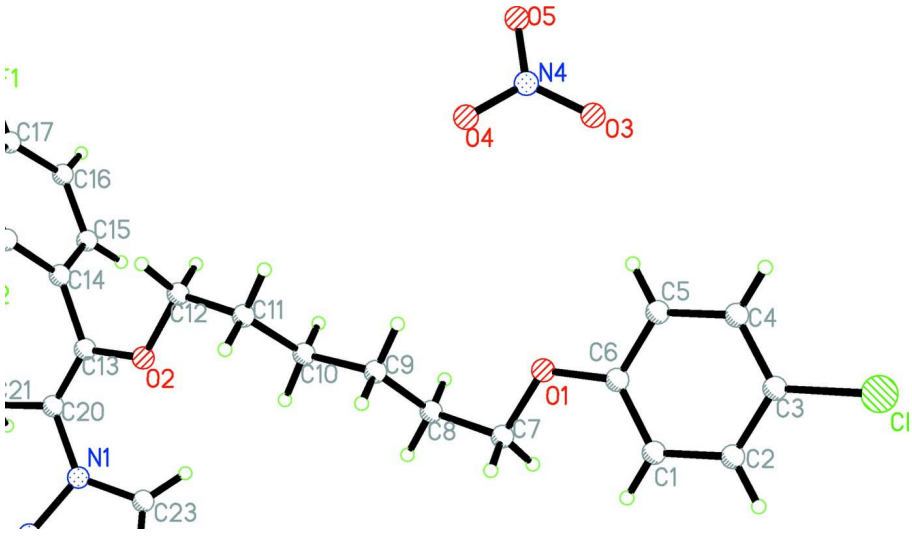

Figure 1

The molecular structure of the title molecule, with the atom numbering scheme. Displacement ellipsoids are drawn at 30% probability levels.

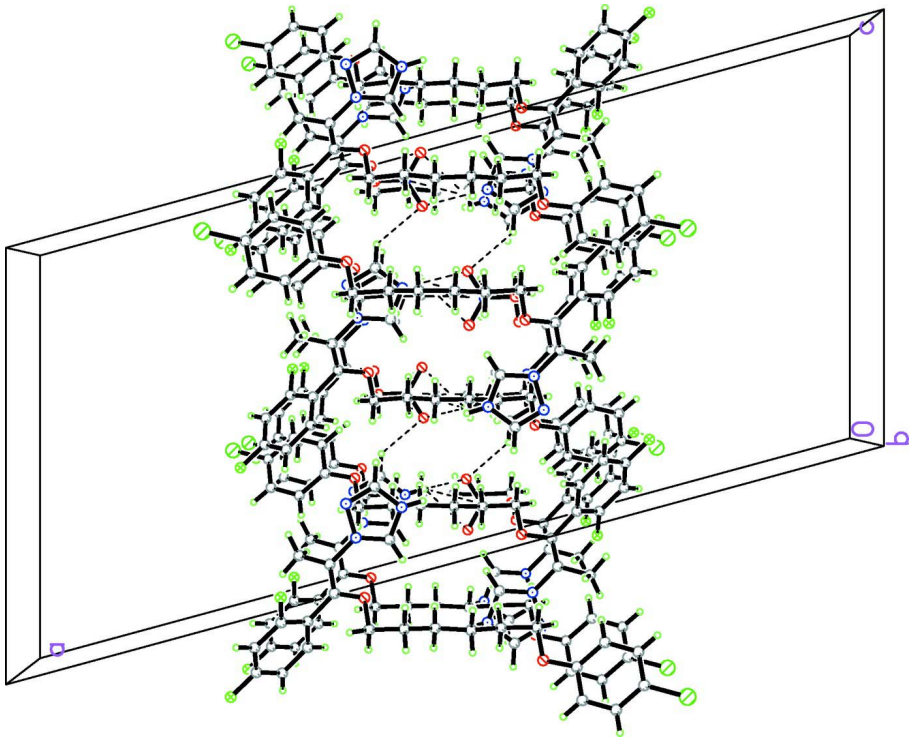

Figure 2

The packing diagram of the title compound. Hydron bonds are shown as dashed lines.

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Crystal data

$C_{23}H_{25}ClF_2N_3O_2^+\cdot NO_3^-$
M_r = 510.92
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
a = 35.538 (7) Å
b = 8.5550 (17) Å
c = 17.072 (3) Å
β = 105.21 (3)°
V = 5008.6 (17) Å^3
Z = 8
F(000) = 2128
D_x = 1.355 Mg m^{-3}
Melting point: 383.15 K
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 25 reflections
θ = 9–13°
μ = 0.21 mm^{-1}
T = 293 K
Block, yellow
0.30 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω/2*θ* scans
Absorption correction: *ψ* scan (North *et al.*, 1968)
T_{min} = 0.940, T_{max} = 0.980
9194 measured reflections
4628 independent reflections
1954 reflections with $I > 2\sigma(I)$
R_{int} = 0.063
θ_{max} = 25.5°, θ_{min} = 1.2°
h = −42→42
k = −10→0
l = −20→20
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.176
S = 1.00
4628 reflections
317 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
$w = 1/[\sigma^2(F_o^2) + (0.068P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0017 (3)

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 $F^2 > \sigma(F^2)$ 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}
Cl	0.24234 (4)	0.19455 (19)	0.11286 (9)	0.1265 (6)

N1	0.60062 (8)	0.8994 (3)	0.62210 (18)	0.0581 (8)
O1	0.39276 (8)	0.4929 (4)	0.25522 (17)	0.0823 (9)
F1	0.73827 (8)	0.7269 (3)	0.34649 (16)	0.1146 (10)
C1	0.32631 (12)	0.4430 (5)	0.2609 (2)	0.0796 (13)
H1A	0.3293	0.5017	0.3080	0.096*
O2	0.58960 (7)	0.7156 (3)	0.48863 (16)	0.0806 (9)
F2	0.68002 (7)	0.5624 (3)	0.54599 (15)	0.1046 (9)
N2	0.61212 (9)	0.9274 (4)	0.70427 (19)	0.0790 (10)
C2	0.29134 (12)	0.3703 (6)	0.2254 (3)	0.0834 (13)
H2B	0.2707	0.3798	0.2493	0.100*
N3	0.54892 (9)	0.8995 (4)	0.6624 (2)	0.0728 (10)
H3A	0.5249	0.8934	0.6638	0.087*
C3	0.28647 (12)	0.2854 (6)	0.1564 (3)	0.0805 (13)
C4	0.31692 (13)	0.2685 (5)	0.1204 (3)	0.0774 (12)
H4A	0.3137	0.2095	0.0734	0.093*
C5	0.35210 (11)	0.3405 (5)	0.1553 (2)	0.0693 (11)
H5A	0.3727	0.3306	0.1314	0.083*
C6	0.35694 (11)	0.4270 (5)	0.2254 (2)	0.0666 (11)
C7	0.39858 (12)	0.5892 (6)	0.3251 (3)	0.0944 (14)
H7A	0.3968	0.5268	0.3714	0.113*
H7B	0.3786	0.6692	0.3162	0.113*
C8	0.43785 (13)	0.6638 (6)	0.3416 (3)	0.1040 (16)
H8A	0.4391	0.7249	0.2945	0.125*
H8B	0.4406	0.7357	0.3867	0.125*
C9	0.47111 (12)	0.5551 (6)	0.3607 (3)	0.0940 (15)
H9A	0.4698	0.4879	0.3143	0.113*
H9B	0.4695	0.4895	0.4060	0.113*
C10	0.51068 (13)	0.6424 (6)	0.3825 (3)	0.1051 (16)
H10A	0.5125	0.7054	0.3364	0.126*
H10B	0.5114	0.7126	0.4275	0.126*
C11	0.54449 (13)	0.5380 (6)	0.4046 (3)	0.1036 (16)
H11A	0.5423	0.4622	0.3613	0.124*
H11B	0.5435	0.4810	0.4531	0.124*
C12	0.58357 (12)	0.6168 (6)	0.4198 (3)	0.1026 (16)
H12A	0.6040	0.5384	0.4288	0.123*
H12B	0.5847	0.6779	0.3726	0.123*
C13	0.62371 (10)	0.7959 (5)	0.5136 (2)	0.0553 (9)
C14	0.65339 (10)	0.7764 (5)	0.4670 (2)	0.0562 (10)
C15	0.65451 (12)	0.8748 (5)	0.4041 (2)	0.0796 (12)
H15A	0.6361	0.9540	0.3894	0.095*
C16	0.68307 (14)	0.8571 (6)	0.3618 (3)	0.0880 (14)
H16A	0.6835	0.9218	0.3182	0.106*
C17	0.71014 (12)	0.7424 (6)	0.3862 (3)	0.0754 (13)
C18	0.71030 (11)	0.6422 (5)	0.4471 (3)	0.0742 (12)
H18A	0.7289	0.5638	0.4622	0.089*
C19	0.68107 (11)	0.6632 (5)	0.4859 (2)	0.0638 (11)
C20	0.62999 (10)	0.8855 (5)	0.5780 (2)	0.0584 (10)
C21	0.66594 (11)	0.9797 (6)	0.6133 (3)	0.0979 (16)

H21A	0.6842	0.9647	0.5813	0.147*
H21B	0.6775	0.9464	0.6680	0.147*
H21C	0.6592	1.0883	0.6131	0.147*
C22	0.57978 (13)	0.9257 (5)	0.7252 (3)	0.0823 (13)
H22A	0.5781	0.9410	0.7781	0.099*
C23	0.56259 (11)	0.8848 (5)	0.5979 (2)	0.0700 (11)
H23A	0.5479	0.8672	0.5450	0.084*
N4	0.45438 (12)	0.1212 (5)	0.0878 (3)	0.0839 (11)
O3	0.41881 (10)	0.1263 (5)	0.0716 (2)	0.1251 (13)
O4	0.47371 (9)	0.1062 (5)	0.1602 (2)	0.1148 (13)
O5	0.47201 (9)	0.1337 (4)	0.03508 (19)	0.1087 (11)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl	0.0756 (9)	0.1377 (13)	0.1570 (13)	−0.0353 (8)	0.0145 (8)	−0.0120 (10)
N1	0.0538 (19)	0.066 (2)	0.058 (2)	0.0018 (16)	0.0214 (15)	−0.0005 (17)
O1	0.0680 (19)	0.100 (2)	0.0800 (19)	−0.0165 (17)	0.0211 (14)	−0.0192 (18)
F1	0.1021 (19)	0.133 (2)	0.142 (2)	−0.0203 (17)	0.0900 (18)	−0.0211 (19)
C1	0.071 (3)	0.106 (4)	0.068 (3)	−0.006 (3)	0.028 (2)	−0.001 (3)
O2	0.0637 (18)	0.101 (2)	0.088 (2)	−0.0172 (16)	0.0399 (15)	−0.0352 (18)
F2	0.0991 (19)	0.124 (2)	0.1046 (19)	0.0447 (16)	0.0508 (15)	0.0433 (17)
N2	0.070 (2)	0.114 (3)	0.060 (2)	0.001 (2)	0.0299 (18)	−0.004 (2)
C2	0.063 (3)	0.106 (4)	0.089 (3)	−0.011 (3)	0.034 (2)	0.014 (3)
N3	0.059 (2)	0.080 (3)	0.091 (3)	−0.0028 (19)	0.040 (2)	−0.006 (2)
C3	0.064 (3)	0.086 (3)	0.091 (3)	−0.014 (2)	0.018 (3)	0.007 (3)
C4	0.079 (3)	0.073 (3)	0.077 (3)	−0.010 (3)	0.013 (2)	−0.003 (2)
C5	0.067 (3)	0.072 (3)	0.074 (3)	−0.005 (2)	0.027 (2)	0.000 (2)
C6	0.054 (2)	0.074 (3)	0.070 (3)	−0.009 (2)	0.013 (2)	0.004 (2)
C7	0.077 (3)	0.105 (4)	0.096 (3)	−0.015 (3)	0.015 (3)	−0.019 (3)
C8	0.084 (4)	0.101 (4)	0.113 (4)	−0.013 (3)	0.001 (3)	−0.024 (3)
C9	0.069 (3)	0.106 (4)	0.099 (3)	−0.020 (3)	0.008 (2)	−0.005 (3)
C10	0.080 (3)	0.113 (4)	0.115 (4)	−0.015 (3)	0.014 (3)	−0.019 (3)
C11	0.077 (3)	0.119 (4)	0.117 (4)	−0.014 (3)	0.029 (3)	−0.023 (3)
C12	0.063 (3)	0.139 (4)	0.109 (4)	−0.017 (3)	0.029 (3)	−0.050 (4)
C13	0.046 (2)	0.063 (3)	0.059 (2)	0.001 (2)	0.0191 (18)	0.002 (2)
C14	0.052 (2)	0.065 (3)	0.057 (2)	0.001 (2)	0.0227 (18)	0.002 (2)
C15	0.079 (3)	0.084 (3)	0.085 (3)	0.012 (2)	0.038 (2)	0.012 (3)
C16	0.103 (4)	0.093 (4)	0.083 (3)	−0.011 (3)	0.052 (3)	0.010 (3)
C17	0.066 (3)	0.084 (4)	0.091 (3)	−0.019 (3)	0.046 (3)	−0.020 (3)
C18	0.053 (2)	0.085 (3)	0.091 (3)	−0.001 (2)	0.031 (2)	−0.011 (3)
C19	0.061 (2)	0.075 (3)	0.059 (2)	0.001 (2)	0.023 (2)	0.006 (2)
C20	0.049 (2)	0.073 (3)	0.058 (2)	0.004 (2)	0.0211 (18)	0.001 (2)
C21	0.067 (3)	0.132 (4)	0.105 (3)	−0.030 (3)	0.040 (2)	−0.040 (3)
C22	0.083 (3)	0.109 (4)	0.064 (3)	−0.008 (3)	0.036 (3)	−0.011 (3)
C23	0.051 (2)	0.094 (3)	0.070 (3)	−0.001 (2)	0.025 (2)	−0.011 (3)
N4	0.071 (3)	0.099 (3)	0.091 (3)	−0.004 (2)	0.037 (2)	−0.015 (3)
O3	0.064 (2)	0.180 (4)	0.134 (3)	−0.024 (2)	0.0314 (19)	−0.032 (3)

O4	0.076 (2)	0.192 (4)	0.089 (2)	0.014 (2)	0.0453 (19)	0.026 (3)
O5	0.092 (2)	0.156 (3)	0.094 (2)	0.004 (2)	0.0514 (19)	0.001 (2)

Geometric parameters (Å, º)

Cl—C3	1.733 (4)	C9—H9A	0.9700
N1—C23	1.311 (4)	C9—H9B	0.9700
N1—N2	1.375 (4)	C10—C11	1.465 (6)
N1—C20	1.443 (4)	C10—H10A	0.9700
O1—C6	1.363 (4)	C10—H10B	0.9700
O1—C7	1.420 (5)	C11—C12	1.503 (5)
F1—C17	1.353 (4)	C11—H11A	0.9700
C1—C2	1.379 (5)	C11—H11B	0.9700
C1—C6	1.385 (5)	C12—H12A	0.9700
C1—H1A	0.9300	C12—H12B	0.9700
O2—C13	1.361 (4)	C13—C20	1.310 (5)
O2—C12	1.418 (5)	C13—C14	1.488 (5)
F2—C19	1.349 (4)	C14—C19	1.358 (5)
N2—C22	1.290 (4)	C14—C15	1.374 (5)
C2—C3	1.357 (5)	C15—C16	1.400 (5)
C2—H2B	0.9300	C15—H15A	0.9300
N3—C23	1.321 (4)	C16—C17	1.361 (6)
N3—C22	1.335 (5)	C16—H16A	0.9300
N3—H3A	0.8600	C17—C18	1.347 (5)
C3—C4	1.385 (6)	C18—C19	1.381 (5)
C4—C5	1.381 (5)	C18—H18A	0.9300
C4—H4A	0.9300	C20—C21	1.497 (5)
C5—C6	1.378 (5)	C21—H21A	0.9600
C5—H5A	0.9300	C21—H21B	0.9600
C7—C8	1.492 (5)	C21—H21C	0.9600
C7—H7A	0.9700	C22—H22A	0.9300
C7—H7B	0.9700	C23—H23A	0.9300
C8—C9	1.472 (6)	N4—O3	1.222 (4)
C8—H8A	0.9700	N4—O4	1.253 (4)
C8—H8B	0.9700	N4—O5	1.229 (4)
C9—C10	1.548 (6)		

C23—N1—N2	110.1 (3)	C10—C11—C12	115.4 (5)
C23—N1—C20	130.9 (3)	C10—C11—H11A	108.4
N2—N1—C20	119.0 (3)	C12—C11—H11A	108.4
C6—O1—C7	118.1 (3)	C10—C11—H11B	108.4
C2—C1—C6	119.0 (4)	C12—C11—H11B	108.4
C2—C1—H1A	120.5	H11A—C11—H11B	107.5
C6—C1—H1A	120.5	O2—C12—C11	110.0 (4)
C13—O2—C12	119.3 (3)	O2—C12—H12A	109.7
C22—N2—N1	103.4 (3)	C11—C12—H12A	109.7
C3—C2—C1	121.2 (4)	O2—C12—H12B	109.7
C3—C2—H2B	119.4	C11—C12—H12B	109.7

C1—C2—H2B	119.4	H12A—C12—H12B	108.2
C23—N3—C22	106.3 (3)	C20—C13—O2	120.0 (3)
C23—N3—H3A	126.9	C20—C13—C14	121.7 (3)
C22—N3—H3A	126.9	O2—C13—C14	118.3 (3)
C2—C3—C4	120.3 (4)	C19—C14—C15	117.0 (3)
C2—C3—Cl	120.5 (4)	C19—C14—C13	121.7 (3)
C4—C3—Cl	119.2 (4)	C15—C14—C13	121.3 (4)
C3—C4—C5	119.1 (4)	C14—C15—C16	120.7 (4)
C3—C4—H4A	120.4	C14—C15—H15A	119.6
C5—C4—H4A	120.4	C16—C15—H15A	119.6
C6—C5—C4	120.5 (4)	C17—C16—C15	118.0 (4)
C6—C5—H5A	119.8	C17—C16—H16A	121.0
C4—C5—H5A	119.8	C15—C16—H16A	121.0
O1—C6—C5	116.0 (3)	C18—C17—F1	118.1 (5)
O1—C6—C1	124.0 (4)	C18—C17—C16	123.7 (4)
C5—C6—C1	120.0 (4)	F1—C17—C16	118.2 (5)
O1—C7—C8	109.3 (4)	C17—C18—C19	115.8 (4)
O1—C7—H7A	109.8	C17—C18—H18A	122.1
C8—C7—H7A	109.8	C19—C18—H18A	122.1
O1—C7—H7B	109.8	F2—C19—C14	118.3 (3)
C8—C7—H7B	109.8	F2—C19—C18	117.1 (4)
H7A—C7—H7B	108.3	C14—C19—C18	124.6 (4)
C9—C8—C7	115.4 (4)	C13—C20—N1	119.9 (3)
C9—C8—H8A	108.4	C13—C20—C21	126.3 (3)
C7—C8—H8A	108.4	N1—C20—C21	113.8 (3)
C9—C8—H8B	108.4	C20—C21—H21A	109.5
C7—C8—H8B	108.4	C20—C21—H21B	109.5
H8A—C8—H8B	107.5	H21A—C21—H21B	109.5
C8—C9—C10	112.0 (4)	C20—C21—H21C	109.5
C8—C9—H9A	109.2	H21A—C21—H21C	109.5
C10—C9—H9A	109.2	H21B—C21—H21C	109.5
C8—C9—H9B	109.2	N2—C22—N3	112.6 (4)
C10—C9—H9B	109.2	N2—C22—H22A	123.7
H9A—C9—H9B	107.9	N3—C22—H22A	123.7
C11—C10—C9	113.6 (4)	N1—C23—N3	107.6 (3)
C11—C10—H10A	108.8	N1—C23—H23A	126.2
C9—C10—H10A	108.8	N3—C23—H23A	126.2
C11—C10—H10B	108.8	O3—N4—O4	119.6 (4)
C9—C10—H10B	108.8	O3—N4—O5	121.8 (4)
H10A—C10—H10B	107.7	O4—N4—O5	118.6 (4)

C23—N1—N2—C22	0.9 (5)	C19—C14—C15—C16	0.2 (6)
C20—N1—N2—C22	−177.0 (3)	C13—C14—C15—C16	178.7 (4)
C6—C1—C2—C3	−0.6 (7)	C14—C15—C16—C17	−1.6 (7)
C1—C2—C3—C4	0.7 (7)	C15—C16—C17—C18	2.0 (7)
C1—C2—C3—Cl	179.8 (3)	C15—C16—C17—F1	−178.7 (4)
C2—C3—C4—C5	−0.6 (7)	F1—C17—C18—C19	179.8 (3)
Cl—C3—C4—C5	−179.8 (3)	C16—C17—C18—C19	−0.9 (6)

C3—C4—C5—C6	0.5 (6)	C15—C14—C19—F2	−178.2 (3)
C7—O1—C6—C5	177.0 (4)	C13—C14—C19—F2	3.2 (5)
C7—O1—C6—C1	−2.8 (6)	C15—C14—C19—C18	1.1 (6)
C4—C5—C6—O1	179.8 (3)	C13—C14—C19—C18	−177.5 (4)
C4—C5—C6—C1	−0.4 (6)	C17—C18—C19—F2	178.6 (3)
C2—C1—C6—O1	−179.7 (4)	C17—C18—C19—C14	−0.7 (6)
C2—C1—C6—C5	0.4 (6)	O2—C13—C20—N1	−0.3 (5)
C6—O1—C7—C8	−173.4 (4)	C14—C13—C20—N1	−179.1 (3)
O1—C7—C8—C9	−62.7 (6)	O2—C13—C20—C21	179.5 (4)
C7—C8—C9—C10	−176.3 (4)	C14—C13—C20—C21	0.7 (6)
C8—C9—C10—C11	178.0 (4)	C23—N1—C20—C13	−27.6 (6)
C9—C10—C11—C12	175.4 (4)	N2—N1—C20—C13	149.8 (3)
C13—O2—C12—C11	178.8 (4)	C23—N1—C20—C21	152.6 (4)
C10—C11—C12—O2	64.8 (6)	N2—N1—C20—C21	−30.0 (5)
C12—O2—C13—C20	−178.8 (4)	N1—N2—C22—N3	−0.3 (5)
C12—O2—C13—C14	0.1 (5)	C23—N3—C22—N2	−0.4 (5)
C20—C13—C14—C19	89.2 (5)	N2—N1—C23—N3	−1.2 (5)
O2—C13—C14—C19	−89.7 (4)	C20—N1—C23—N3	176.4 (3)
C20—C13—C14—C15	−89.3 (5)	C22—N3—C23—N1	1.0 (5)
O2—C13—C14—C15	91.8 (5)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3*A*···O4^{i}	0.86	1.8	2.661 (5)	175
C22—H22*A*···O4^{ii}	0.93	2.38	3.077 (6)	131

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