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1-{(*Z*)-1-[3-(4-Bromophenoxy)propoxy]-1-(2,4-difluorophenyl)prop-1-en-2-yl}-1*H*-1,2,4-triazol-4-ium nitrate

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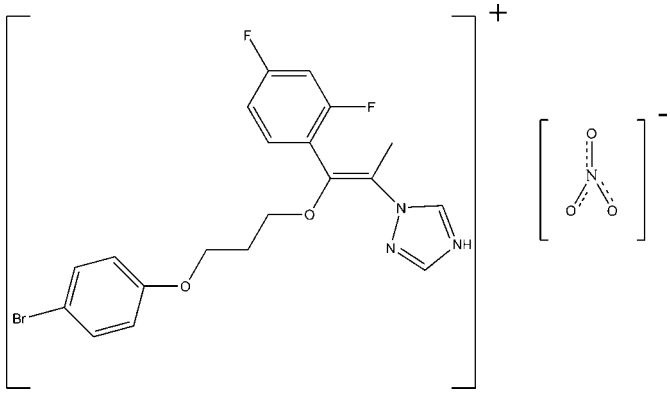
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.008 Å; R factor = 0.063; wR factor = 0.152; data-to-parameter ratio = 13.9.

In the title molecular salt, $C_{20}H_{19}BrF_2N_3O_2^+\cdot NO_3^-$, the N atom at position 4 of the heterocyclic ring is protonated. The triazole ring makes dihedral angles of 96.6 (4) and 54.4 (3)° with the 4-bromophenyl and 2,4-difluorophenyl rings, respectively, and the molecule adopts a *Z* conformation about the C=C double bond. In the crystal, cations and anions are linked by N—H···O and C—H···O hydrogen bonds.

Related literature

For background to the uses of triazole derivatives, see: Jeu *et al.* (2003); Fromtling & Castaner (1996). For further synthetic details, see: Ludwig & Kurt (1985).



Experimental

Crystal data

$C_{20}H_{19}BrF_2N_3O_2^+\cdot NO_3^-$ M_r = 513.30
Triclinic, $P\overline{1}$
a = 8.3030 (17) Å
b = 8.4260 (17) Å
c = 16.170 (3) Å
α = 91.10 (3)°
β = 95.80 (3)°
γ = 102.30 (3)°
V = 1098.7 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.93 mm^{-1}
T = 293 K
0.20 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.699, T_{max} = 0.831
4329 measured reflections
4029 independent reflections
2250 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.152
S = 1.01
4029 reflections
289 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.49 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3A···O4	0.86	1.95	2.790 (6)	167
C9—H9A···O3^{i}	0.93	2.55	3.271 (7)	135
C10—H10A···O3^{ii}	0.93	2.49	3.263 (7)	140
C10—H10A···O5^{ii}	0.93	2.42	3.340 (7)	168
C19—H19A···O3	0.93	2.54	3.276 (7)	137

Symmetry codes: (i) $-x+2, -y+1, -z+1$; (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: HB6699).

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supplementary materials

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1-{(*Z*)-1-[3-(4-Bromophenoxy)propoxy]-1-(2,4-difluorophenyl)prop-1-en-2-yl}-1*H*-1,2,4-triazol-4-ium nitrate

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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 has a Z conformation. In the crystal structure the anions and cations are connected via N—H···O and C—H···O hydrogen bonding (Table 1 and Fig. 2).

Experimental

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. 2.93 g (0.01 mol) 1-bromo-3-(4-bromophenoxy)-propane, 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. Colourless plates of the title compound were obtained by slow evaporation of an ethanol solution. Details on the synthesis can be found in the literature reported by Ludwig & Kurt (1985).

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).

Computing details

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); 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).

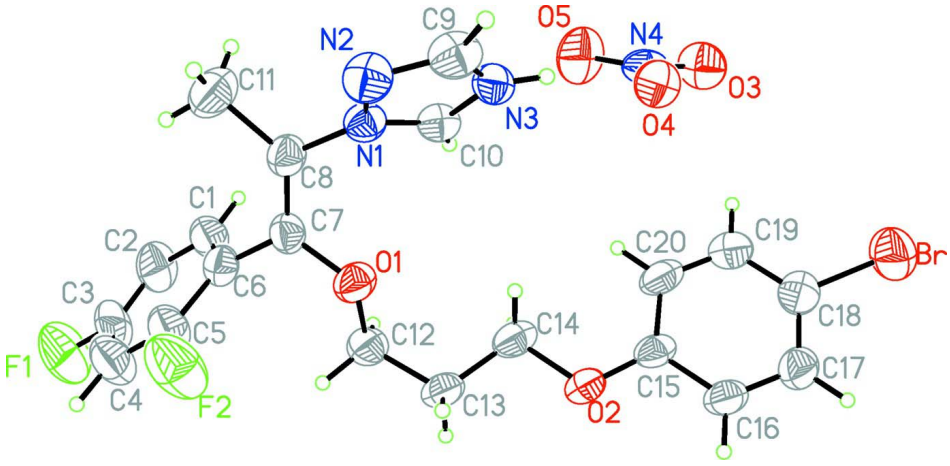

Figure 1

The molecular structure of the title molecule, with displacement ellipsoids drawn at 30% probability levels.

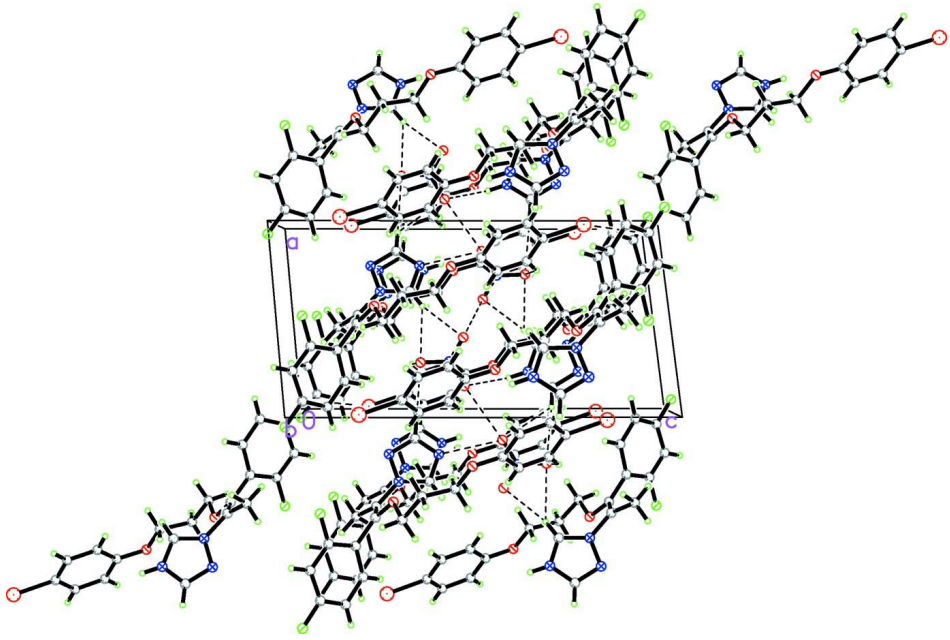

Figure 2

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

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Crystal data

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Hall symbol: -P 1
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$b = 8.4260$ (17) Å
$c = 16.170$ (3) Å
$\alpha = 91.10$ (3)°
$\beta = 95.80$ (3)°
$\gamma = 102.30$ (3)°
$V = 1098.7$ (4) Å^3
$Z = 2$
$F(000) = 520$
$D_x = 1.552$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections

θ = 9–13°
μ = 1.93 mm^{-1}
T = 293 K
Plate, colorless
0.20 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.699, T_{max} = 0.831
4329 measured reflections
4029 independent reflections
2250 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 25.4°, θ_{min} = 1.3°
h = 0→10
k = −10→9
l = −19→19
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.152
S = 1.01
4029 reflections
289 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
$w = 1/[\sigma^2(F_o^2) + (0.075P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.49 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 e Å^{-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}
Br	0.98343 (10)	0.93868 (9)	0.81164 (4)	0.0842 (3)
O1	0.5622 (4)	0.7633 (4)	0.2460 (2)	0.0504 (9)
N1	0.6426 (5)	0.4746 (5)	0.2587 (2)	0.0415 (10)
F1	−0.0592 (5)	0.7759 (6)	−0.0124 (3)	0.1075 (14)
C1	0.1701 (7)	0.5970 (7)	0.1459 (3)	0.0567 (15)
H1A	0.1495	0.5219	0.1871	0.068*
O2	0.7624 (4)	1.0648 (4)	0.4584 (2)	0.0546 (10)
F2	0.5119 (5)	0.8501 (5)	0.0607 (2)	0.0953 (13)
N2	0.7812 (5)	0.4226 (5)	0.2392 (3)	0.0516 (11)
C2	0.0372 (7)	0.6312 (8)	0.0959 (4)	0.0686 (17)
H2B	−0.0714	0.5805	0.1027	0.082*
N3	0.7821 (5)	0.4579 (5)	0.3707 (3)	0.0445 (10)
H3A	0.8152	0.4614	0.4230	0.053*

C3	0.0711 (8)	0.7408 (8)	0.0371 (4)	0.0676 (17)
C4	0.2289 (8)	0.8143 (8)	0.0214 (4)	0.0734 (18)
H4A	0.2481	0.8853	−0.0216	0.088*
C5	0.3555 (7)	0.7772 (7)	0.0724 (3)	0.0605 (15)
C6	0.3333 (6)	0.6713 (6)	0.1366 (3)	0.0456 (12)
C7	0.4737 (6)	0.6405 (6)	0.1930 (3)	0.0425 (12)
C8	0.5168 (6)	0.4976 (6)	0.1943 (3)	0.0449 (12)
C9	0.8628 (7)	0.4159 (6)	0.3118 (4)	0.0504 (14)
H9A	0.9642	0.3854	0.3204	0.060*
C10	0.6445 (6)	0.4938 (6)	0.3394 (3)	0.0402 (12)
H10A	0.5645	0.5261	0.3681	0.048*
C11	0.4530 (8)	0.3536 (7)	0.1346 (4)	0.0704 (18)
H11A	0.3712	0.3784	0.0935	0.106*
H11B	0.4040	0.2615	0.1645	0.106*
H11C	0.5432	0.3289	0.1077	0.106*
C12	0.4855 (7)	0.8956 (6)	0.2671 (3)	0.0530 (14)
H12A	0.3866	0.8547	0.2941	0.064*
H12B	0.4542	0.9490	0.2172	0.064*
C13	0.6100 (7)	1.0130 (6)	0.3248 (3)	0.0530 (14)
H13A	0.5696	1.1117	0.3321	0.064*
H13B	0.7131	1.0415	0.2997	0.064*
C14	0.6429 (7)	0.9442 (6)	0.4086 (3)	0.0528 (14)
H14A	0.5411	0.9168	0.4348	0.063*
H14B	0.6851	0.8463	0.4023	0.063*
C15	0.8075 (6)	1.0270 (6)	0.5371 (3)	0.0446 (12)
C16	0.9266 (7)	1.1447 (6)	0.5838 (4)	0.0536 (14)
H16A	0.9712	1.2414	0.5598	0.064*
C17	0.9793 (7)	1.1207 (7)	0.6646 (4)	0.0565 (15)
H17A	1.0592	1.2002	0.6952	0.068*
C18	0.9121 (7)	0.9758 (7)	0.7007 (3)	0.0545 (14)
C19	0.7945 (7)	0.8595 (7)	0.6547 (4)	0.0562 (15)
H19A	0.7494	0.7628	0.6786	0.067*
C20	0.7436 (7)	0.8845 (6)	0.5745 (4)	0.0568 (15)
H20A	0.6643	0.8041	0.5441	0.068*
O4	0.8591 (4)	0.5077 (4)	0.5425 (2)	0.0537 (9)
N4	0.7288 (6)	0.4501 (5)	0.5751 (3)	0.0502 (11)
O3	0.7345 (5)	0.4623 (5)	0.6516 (3)	0.0658 (11)
O5	0.6014 (5)	0.3882 (5)	0.5316 (3)	0.0775 (13)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br	0.0977 (6)	0.0922 (6)	0.0618 (4)	0.0230 (4)	−0.0016 (4)	0.0056 (4)
O1	0.048 (2)	0.042 (2)	0.061 (2)	0.0136 (17)	−0.0076 (18)	−0.0066 (17)
N1	0.039 (2)	0.039 (2)	0.048 (3)	0.0110 (19)	0.005 (2)	−0.0007 (19)
F1	0.083 (3)	0.147 (4)	0.093 (3)	0.038 (3)	−0.022 (2)	0.035 (3)
C1	0.055 (4)	0.059 (4)	0.051 (3)	0.004 (3)	−0.001 (3)	0.010 (3)
O2	0.057 (2)	0.034 (2)	0.065 (2)	0.0005 (17)	−0.0107 (19)	−0.0009 (18)
F2	0.066 (2)	0.120 (3)	0.091 (3)	−0.004 (2)	0.007 (2)	0.046 (2)
N2	0.045 (3)	0.059 (3)	0.054 (3)	0.017 (2)	0.010 (2)	−0.003 (2)

C2	0.046 (4)	0.089 (5)	0.065 (4)	0.012 (3)	−0.013 (3)	0.003 (4)
N3	0.036 (2)	0.041 (2)	0.054 (3)	0.006 (2)	−0.003 (2)	0.004 (2)
C3	0.065 (4)	0.091 (5)	0.049 (3)	0.032 (4)	−0.016 (3)	0.010 (3)
C4	0.077 (5)	0.080 (5)	0.061 (4)	0.014 (4)	−0.004 (4)	0.024 (3)
C5	0.055 (4)	0.070 (4)	0.050 (3)	0.002 (3)	−0.002 (3)	0.014 (3)
C6	0.046 (3)	0.048 (3)	0.040 (3)	0.008 (3)	−0.003 (2)	−0.003 (2)
C7	0.041 (3)	0.046 (3)	0.038 (3)	0.007 (3)	−0.002 (2)	0.002 (2)
C8	0.042 (3)	0.045 (3)	0.045 (3)	0.008 (2)	−0.003 (2)	−0.003 (2)
C9	0.038 (3)	0.052 (3)	0.066 (4)	0.018 (3)	0.013 (3)	−0.003 (3)
C10	0.031 (3)	0.040 (3)	0.051 (3)	0.010 (2)	0.009 (2)	0.003 (2)
C11	0.070 (4)	0.062 (4)	0.075 (4)	0.016 (3)	−0.009 (3)	−0.020 (3)
C12	0.051 (3)	0.044 (3)	0.063 (3)	0.015 (3)	−0.009 (3)	0.002 (3)
C13	0.058 (4)	0.032 (3)	0.065 (4)	0.006 (3)	−0.005 (3)	−0.004 (3)
C14	0.047 (3)	0.036 (3)	0.070 (4)	0.001 (3)	0.002 (3)	−0.005 (3)
C15	0.043 (3)	0.029 (3)	0.062 (3)	0.010 (2)	0.004 (3)	−0.005 (2)
C16	0.050 (3)	0.033 (3)	0.074 (4)	0.006 (3)	0.000 (3)	0.002 (3)
C17	0.051 (3)	0.050 (4)	0.067 (4)	0.014 (3)	−0.006 (3)	−0.004 (3)
C18	0.051 (3)	0.057 (4)	0.058 (3)	0.019 (3)	0.006 (3)	−0.005 (3)
C19	0.063 (4)	0.044 (3)	0.061 (4)	0.009 (3)	0.011 (3)	0.003 (3)
C20	0.054 (4)	0.034 (3)	0.077 (4)	0.000 (3)	0.002 (3)	−0.007 (3)
O4	0.035 (2)	0.057 (2)	0.067 (2)	0.0034 (17)	0.0080 (18)	0.0052 (19)
N4	0.042 (3)	0.038 (3)	0.074 (3)	0.012 (2)	0.012 (3)	0.007 (2)
O3	0.074 (3)	0.071 (3)	0.056 (3)	0.016 (2)	0.020 (2)	0.011 (2)
O5	0.038 (2)	0.091 (3)	0.091 (3)	−0.010 (2)	0.003 (2)	0.000 (3)

Geometric parameters (Å, º)

Br—C18	1.884 (6)	C9—H9A	0.9300
O1—C7	1.364 (6)	C10—H10A	0.9300
O1—C12	1.448 (6)	C11—H11A	0.9600
N1—C10	1.309 (6)	C11—H11B	0.9600
N1—N2	1.379 (5)	C11—H11C	0.9600
N1—C8	1.445 (6)	C12—C13	1.500 (7)
F1—C3	1.368 (6)	C12—H12A	0.9700
C1—C2	1.386 (7)	C12—H12B	0.9700
C1—C6	1.392 (7)	C13—C14	1.507 (7)
C1—H1A	0.9300	C13—H13A	0.9700
O2—C15	1.352 (6)	C13—H13B	0.9700
O2—C14	1.430 (6)	C14—H14A	0.9700
F2—C5	1.347 (6)	C14—H14B	0.9700
N2—C9	1.304 (6)	C15—C20	1.383 (7)
C2—C3	1.346 (8)	C15—C16	1.391 (7)
C2—H2B	0.9300	C16—C17	1.368 (7)
N3—C10	1.300 (6)	C16—H16A	0.9300
N3—C9	1.303 (6)	C17—C18	1.395 (8)
N3—H3A	0.8600	C17—H17A	0.9300
C3—C4	1.375 (8)	C18—C19	1.374 (7)
C4—C5	1.363 (8)	C19—C20	1.356 (7)
C4—H4A	0.9300	C19—H19A	0.9300
C5—C6	1.382 (7)	C20—H20A	0.9300

C6—C7	1.476 (7)	O4—N4	1.259 (5)
C7—C8	1.327 (7)	N4—O5	1.219 (5)
C8—C11	1.504 (7)	N4—O3	1.235 (5)
C7—O1—C12	118.7 (4)	C8—C11—H11C	109.5
C10—N1—N2	110.9 (4)	H11A—C11—H11C	109.5
C10—N1—C8	128.3 (4)	H11B—C11—H11C	109.5
N2—N1—C8	120.8 (4)	O1—C12—C13	107.4 (4)
C2—C1—C6	122.1 (5)	O1—C12—H12A	110.2
C2—C1—H1A	119.0	C13—C12—H12A	110.2
C6—C1—H1A	119.0	O1—C12—H12B	110.2
C15—O2—C14	117.2 (4)	C13—C12—H12B	110.2
C9—N2—N1	103.0 (4)	H12A—C12—H12B	108.5
C3—C2—C1	117.5 (6)	C12—C13—C14	112.4 (4)
C3—C2—H2B	121.3	C12—C13—H13A	109.1
C1—C2—H2B	121.3	C14—C13—H13A	109.1
C10—N3—C9	110.5 (4)	C12—C13—H13B	109.1
C10—N3—H3A	124.8	C14—C13—H13B	109.1
C9—N3—H3A	124.8	H13A—C13—H13B	107.9
C2—C3—F1	118.0 (6)	O2—C14—C13	107.9 (4)
C2—C3—C4	124.0 (6)	O2—C14—H14A	110.1
F1—C3—C4	118.0 (6)	C13—C14—H14A	110.1
C5—C4—C3	116.3 (6)	O2—C14—H14B	110.1
C5—C4—H4A	121.9	C13—C14—H14B	110.1
C3—C4—H4A	121.9	H14A—C14—H14B	108.4
F2—C5—C4	118.2 (5)	O2—C15—C20	125.7 (5)
F2—C5—C6	117.8 (5)	O2—C15—C16	116.1 (5)
C4—C5—C6	124.0 (6)	C20—C15—C16	118.1 (5)
C5—C6—C1	116.0 (5)	C17—C16—C15	121.1 (5)
C5—C6—C7	122.3 (5)	C17—C16—H16A	119.4
C1—C6—C7	121.7 (5)	C15—C16—H16A	119.4
C8—C7—O1	118.6 (4)	C16—C17—C18	119.4 (5)
C8—C7—C6	122.5 (5)	C16—C17—H17A	120.3
O1—C7—C6	118.8 (4)	C18—C17—H17A	120.3
C7—C8—N1	117.9 (4)	C19—C18—C17	119.6 (5)
C7—C8—C11	127.8 (5)	C19—C18—Br	120.1 (4)
N1—C8—C11	114.4 (4)	C17—C18—Br	120.3 (4)
N3—C9—N2	110.4 (5)	C20—C19—C18	120.5 (5)
N3—C9—H9A	124.8	C20—C19—H19A	119.7
N2—C9—H9A	124.8	C18—C19—H19A	119.7
N3—C10—N1	105.2 (4)	C19—C20—C15	121.3 (5)
N3—C10—H10A	127.4	C19—C20—H20A	119.4
N1—C10—H10A	127.4	C15—C20—H20A	119.4
C8—C11—H11A	109.5	O5—N4—O3	121.9 (5)
C8—C11—H11B	109.5	O5—N4—O4	120.4 (5)
H11A—C11—H11B	109.5	O3—N4—O4	117.7 (5)
C10—N1—N2—C9	−1.1 (5)	C10—N1—C8—C7	55.1 (7)
C8—N1—N2—C9	−179.7 (4)	N2—N1—C8—C7	−126.5 (5)

C6—C1—C2—C3	−0.2 (9)	C10—N1—C8—C11	−126.2 (5)
C1—C2—C3—F1	−179.8 (5)	N2—N1—C8—C11	52.2 (6)
C1—C2—C3—C4	3.1 (10)	C10—N3—C9—N2	0.0 (6)
C2—C3—C4—C5	−3.4 (10)	N1—N2—C9—N3	0.6 (6)
F1—C3—C4—C5	179.6 (6)	C9—N3—C10—N1	−0.7 (5)
C3—C4—C5—F2	−178.2 (6)	N2—N1—C10—N3	1.1 (5)
C3—C4—C5—C6	0.6 (10)	C8—N1—C10—N3	179.6 (4)
F2—C5—C6—C1	−179.2 (5)	C7—O1—C12—C13	−179.3 (4)
C4—C5—C6—C1	2.0 (9)	O1—C12—C13—C14	−68.8 (6)
F2—C5—C6—C7	1.8 (8)	C15—O2—C14—C13	179.2 (4)
C4—C5—C6—C7	−177.0 (6)	C12—C13—C14—O2	180.0 (4)
C2—C1—C6—C5	−2.2 (8)	C14—O2—C15—C20	−2.6 (7)
C2—C1—C6—C7	176.7 (5)	C14—O2—C15—C16	178.6 (4)
C12—O1—C7—C8	−158.1 (5)	O2—C15—C16—C17	178.8 (5)
C12—O1—C7—C6	21.7 (6)	C20—C15—C16—C17	−0.1 (8)
C5—C6—C7—C8	−113.0 (6)	C15—C16—C17—C18	−0.2 (8)
C1—C6—C7—C8	68.1 (7)	C16—C17—C18—C19	0.2 (8)
C5—C6—C7—O1	67.2 (7)	C16—C17—C18—Br	179.5 (4)
C1—C6—C7—O1	−111.7 (6)	C17—C18—C19—C20	0.1 (8)
O1—C7—C8—N1	7.3 (7)	Br—C18—C19—C20	−179.3 (4)
C6—C7—C8—N1	−172.5 (4)	C18—C19—C20—C15	−0.3 (9)
O1—C7—C8—C11	−171.2 (5)	O2—C15—C20—C19	−178.5 (5)
C6—C7—C8—C11	9.0 (9)	C16—C15—C20—C19	0.3 (8)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3*A*···O4	0.86	1.95	2.790 (6)	167
C9—H9*A*···O3^{i}	0.93	2.55	3.271 (7)	135
C10—H10*A*···O3^{ii}	0.93	2.49	3.263 (7)	140
C10—H10*A*···O5^{ii}	0.93	2.42	3.340 (7)	168
C19—H19*A*···O3	0.93	2.54	3.276 (7)	137

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