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Crystal structure of ethyl 2-[2-(4-methylbenzoyl)-5-*p*-tolyl-1*H*-imidazol-1-yl]acetate

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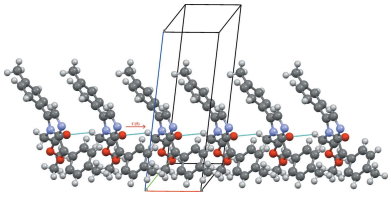
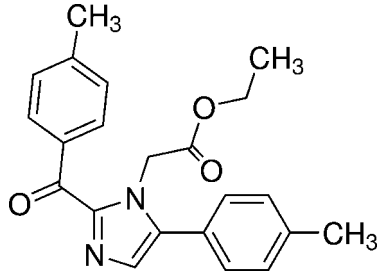
In the title compound, $C_{22}H_{22}N_2O_3$, the plane of the five-membered ring is oriented at dihedral angles of 45.4 (1) and 52.5 (1)° to the phenyl rings. Furthermore, this ring makes an angle of 85.2 (2)° with the plane of the ethyl acetate substituent. The molecular structure is affected by an intramolecular C—H···O hydrogen bond between an H atom from the *p*-tolyl group and the carbonyl O atom of the acetate. The methyl group of the ethyl acetate residue is disordered over two sites with equal occupancies. The crystal structure features intermolecular C—H···O and C—H···N interactions. One of the C—H···O hydrogen bonds forms a $C(5)$ chain motif extending along the *a* axis. In addition, C—H···N contacts form inversion dimers with $R_2^2(12)$ ring motifs, linking the imidazole ring system to the benzene ring of the *p*-tolyl substituent.

1. Chemical context

Imidazole and its derivatives have numerous pharmaceutical applications including uses as antifungal (Shingalapur *et al.* 2009), antimicrobial (Sharma *et al.* 2009), anti-inflammatory (Puratchikody *et al.* 2007), analgesic (Achar *et al.* 2010), antitubercular (Pandey *et al.* 2009), antidepressant (Hadizadeh *et al.* 2008), antileishmanial (Bhandari *et al.* 2009) and anticancer agents (Ozkay *et al.* 2010). We are interested in the synthesis of active pharmaceutical ingredients (APIs) based on imidazoles and we report here the synthesis and crystal structure of the title imidazole derivative.





2. Structural commentary

The molecular structure of the title compound is shown in Fig. 1. The C—N bond lengths within the imidazole ring are 1.373 (3) Å (C10—N2), 1.372 (3) Å (C8—N2), 1.349 (3) Å (C9—N1) and 1.329 (3) Å (C10—N1). These bond distances

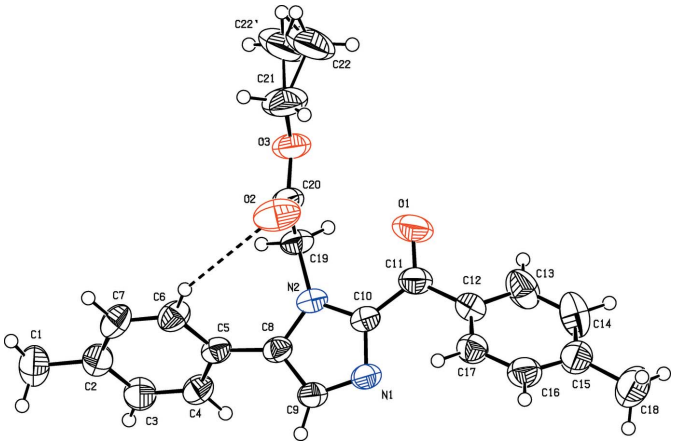


Figure 1
The molecular structure of the title compound, showing the atom-numbering scheme and 50% probability displacement ellipsoids. The methyl group (C22) of the side chain is disordered over two positions each with 0.5 occupancy.

are shorter than the single-bond length (1.443 Å) and longer than the accepted double-bond length (1.269 Å) due to electron delocalization in the central imidazole ring. The phenyl rings and the plane of the imidazole ring are inclined at angles of 45.4 (1)° (with the C12–C17 ring) and 52.5 (1)° (with the C2–C7 ring). The phenyl rings are oriented to each other with a dihedral angle of 88.1 (1)°. Further, the imidazole ring is inclined at an angle of 85.2 (2)° to the best-fit plane through atoms C19, C20, O3, C21 and C22 of the ethyl acetate substituent. The molecular structure is also influenced by the formation of an intramolecular C6—H6···O2 hydrogen bond, Table 1, which generates an *S*(8) ring motif (Bernstein *et al.*, 1995).

3. Supramolecular features

The N-bound methylene group of the side chain is connected with the carbonyl oxygen of an adjacent molecule through a C19—H19*A*···O2 hydrogen bond, forming a linear *C*(5) chain motif along the *a* axis, Table 1 and Fig. 2. The phenyl and imidazole rings are linked through inversion-dimer formation involving C4—H4···N1 hydrogen bonds that generate $R_2^2(12)$ ring motifs. A second inversion dimer to an adjacent molecule results from C1—H1···O2 contacts, forming ring $R_2^2(22)$ **[OK?]** rings, Fig. 3.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···O2	0.93	2.91	3.723 (4)	147
C1—H1*A*···O2^{i}	0.96	2.71	3.605 (4)	155
C4—H4···N1^{ii}	0.93	2.83	3.724 (3)	161
C19—H19*A*···O2^{iii}	0.97	2.51	3.309 (3)	140

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

4. Database survey

The Cambridge Structural Database (Groom & Allen, 2014) reveals only five structures of imidazole derivatives with a $CH_2COOCH_2CH_3$ substituent on nitrogen (Cai *et al.*, 2014; Bahnous *et al.*, 2013; Zaprutko *et al.*, 2012). Imidazoles with benzoyl substituents are slightly more common with eight occurrences (Xue *et al.*, 2014; Nagaraj *et al.*, 2012; Samanta *et al.*, 2013), while the structures of only six *p*-tolyl-substituted imidazoles are found (Bu *et al.*, 1996; Fridman *et al.*, 2006, 2009). These searches also reveal the unique nature of the molecule reported here.

5. Synthesis and crystallization

The title compound was synthesized from a mixture of 2-(4-methoxyphenyl)-2-oxoacetaldehyde (1 mmol), glycine methyl ester hydrochloride (1 mmol) and selenium dioxide (1 mmol) in a basic environment in acetonitrile at 373 K. Crystals suitable for X-ray investigation were obtained by solvent evaporation from the resulting solution in 33% yield.

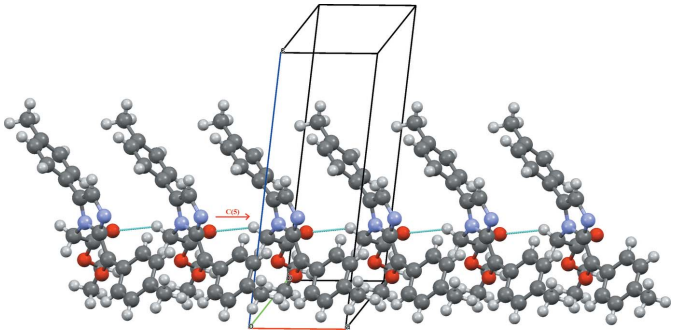


Figure 2
Linear *C*(5) chains formed by a C—H···O intermolecular interaction extending along the *a* axis of the unit cell.

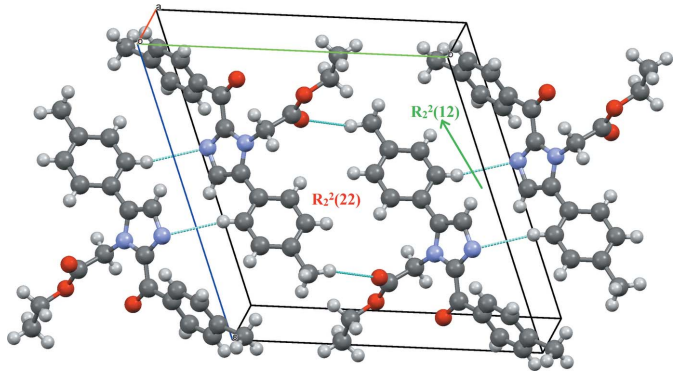


Figure 3
Inversion dimers with $R_2^2(12)$ and $R_2^2(22)$ ring motifs resulting from C—H···N and C—H···O hydrogen bonds.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93 −0.97 Å and U_{iso}(H) = 1.2–1.5U_{eq}(parent C atom). The methyl group C22 of the side chain is disordered over two positions, each with a site-occupancy factor of 0.5. The atomic displacement parameters of these two C atoms are restrained to be equivalent and the C21—C22 and C21—C22′ bond distances were restrained during the refinement using DFIX commands.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{22}H_{22}N_2O_3$
M_r	362.41
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	293
a, b, c (Å)	5.0968 (5), 13.8189 (15), 14.6993 (17)
α, β, γ (°)	71.484 (5), 84.018 (5), 82.531 (5)
V (Å^3)	971.20 (18)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.08
Crystal size (mm)	0.21 × 0.19 × 0.16
Data collection	
Diffractometer	Bruker SMART APEX CCD area-detector
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	18453, 3405, 2354
R_{int}	0.055
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.054, 0.168, 1.07
No. of reflections	3405
No. of parameters	251
No. of restraints	2
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.52, −0.31

Computer programs: *SMART* (Bruker, 2001), *SAINT* (Bruker, 2001), *SHELXTL/PC* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

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Computing details

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT* (Bruker, 2001); program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL/PC* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXTL/PC* (Sheldrick, 2008).

Ethyl 2-[2-(4-methylbenzoyl)-5-*p*-tolyl-1*H*-imidazol-1-yl]acetate

Crystal data

$C_{22}H_{22}N_2O_3$
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Triclinic, $P\overline{1}$
$a = 5.0968$ (5) Å
$b = 13.8189$ (15) Å
$c = 14.6993$ (17) Å
$\alpha = 71.484$ (5)°
$\beta = 84.018$ (5)°
$\gamma = 82.531$ (5)°
$V = 971.20$ (18) Å^3
$Z = 2$
$F(000) = 384$
$D_x = 1.239$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2986 reflections
$\theta = 2.1$–$24.4°$
$\mu = 0.08$ mm^{-1}
$T = 293$ K
Block, colourless
0.21 × 0.19 × 0.16 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
ω scans
18453 measured reflections
3405 independent reflections
2354 reflections with $I > 2\sigma(I)$
$R_{int} = 0.055$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.5°$
$h = -6 \rightarrow 6$
$k = -16 \rightarrow 16$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.168$
$S = 1.07$
3405 reflections
251 parameters
2 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0636P)^2 + 0.5608P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.52$ e Å^{-3}
$\Delta\rho_{min} = -0.30$ 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	−0.6633 (7)	0.3819 (3)	0.6826 (2)	0.0754 (9)	
H1A	−0.6105	0.4476	0.6779	0.113*	
H1B	−0.6498	0.3372	0.7474	0.113*	
H1C	−0.8433	0.3901	0.6655	0.113*	
C2	−0.4852 (5)	0.3362 (2)	0.61545 (19)	0.0530 (7)	
C3	−0.5093 (5)	0.2386 (2)	0.6123 (2)	0.0568 (7)	
H3	−0.6403	0.2015	0.6517	0.068*	
C4	−0.3441 (5)	0.19537 (19)	0.55220 (19)	0.0501 (6)	
H4	−0.3636	0.1293	0.5524	0.060*	
C5	−0.1486 (5)	0.24912 (17)	0.49132 (17)	0.0426 (6)	
C6	−0.1244 (5)	0.34725 (19)	0.4938 (2)	0.0536 (7)	
H6	0.0048	0.3851	0.4539	0.064*	
C7	−0.2906 (5)	0.3891 (2)	0.5551 (2)	0.0573 (7)	
H7	−0.2705	0.4548	0.5557	0.069*	
C8	0.0360 (5)	0.19884 (17)	0.43313 (17)	0.0419 (6)	
C9	0.1875 (5)	0.10614 (18)	0.46091 (18)	0.0466 (6)	
H9	0.1835	0.0613	0.5233	0.056*	
C10	0.2885 (5)	0.16918 (17)	0.31157 (17)	0.0428 (6)	
C11	0.4033 (5)	0.1806 (2)	0.21394 (19)	0.0539 (7)	
C12	0.6016 (5)	0.0980 (2)	0.19780 (17)	0.0480 (6)	
C17	0.8022 (5)	0.0507 (2)	0.25858 (19)	0.0514 (7)	
H17	0.8186	0.0717	0.3117	0.062*	
C16	0.9772 (6)	−0.0268 (2)	0.2414 (2)	0.0614 (8)	
H16	1.1136	−0.0560	0.2821	0.074*	
C15	0.9556 (6)	−0.0622 (2)	0.1653 (2)	0.0629 (8)	
C14	0.7592 (7)	−0.0130 (3)	0.1034 (2)	0.0777 (10)	
H14	0.7426	−0.0345	0.0505	0.093*	
C13	0.5874 (6)	0.0671 (3)	0.1182 (2)	0.0709 (9)	
H13	0.4612	0.1004	0.0742	0.085*	
C18	1.1384 (8)	−0.1519 (3)	0.1513 (3)	0.0990 (13)	
H18A	1.0844	−0.1703	0.0988	0.149*	
H18B	1.1306	−0.2092	0.2089	0.149*	
H18C	1.3167	−0.1334	0.1372	0.149*	
C19	−0.0310 (5)	0.33098 (18)	0.27136 (18)	0.0488 (6)	
H19A	−0.1902	0.3538	0.3044	0.059*	
H19B	−0.0836	0.3150	0.2167	0.059*	
C20	0.1464 (5)	0.41606 (19)	0.23635 (19)	0.0510 (7)	
C21	0.2175 (8)	0.5710 (2)	0.1156 (2)	0.0923 (12)	
H21A	0.4038	0.5456	0.1198	0.111*	

H21B	0.1794	0.6196	0.1517	0.111*	
C22	0.161 (3)	0.6255 (15)	0.0101 (4)	0.119 (4)	0.5
H22A	0.2473	0.5850	−0.0289	0.179*	0.5
H22B	0.2273	0.6915	−0.0105	0.179*	0.5
H22C	−0.0267	0.6340	0.0036	0.179*	0.5
C22′	0.047 (3)	0.6433 (15)	0.0364 (5)	0.119 (4)	0.5
H22D	0.0075	0.6068	−0.0055	0.179*	0.5
H22E	0.1418	0.7005	0.0001	0.179*	0.5
H22F	−0.1156	0.6677	0.0651	0.179*	0.5
N1	0.3431 (4)	0.08765 (14)	0.38698 (14)	0.0464 (5)	
N2	0.1006 (4)	0.23871 (14)	0.33651 (14)	0.0434 (5)	
O1	0.3314 (5)	0.25504 (18)	0.14648 (15)	0.0913 (8)	
O2	0.3358 (4)	0.42182 (16)	0.27496 (17)	0.0774 (7)	
O3	0.0563 (4)	0.48554 (13)	0.15783 (13)	0.0663 (6)	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.077 (2)	0.081 (2)	0.077 (2)	0.0039 (17)	−0.0022 (17)	−0.0423 (18)
C2	0.0517 (15)	0.0546 (16)	0.0558 (16)	0.0006 (13)	−0.0118 (13)	−0.0211 (13)
C3	0.0558 (16)	0.0555 (17)	0.0601 (17)	−0.0134 (13)	0.0026 (13)	−0.0185 (14)
C4	0.0553 (15)	0.0386 (13)	0.0585 (16)	−0.0095 (12)	−0.0050 (13)	−0.0155 (12)
C5	0.0453 (13)	0.0361 (12)	0.0458 (14)	−0.0031 (10)	−0.0120 (11)	−0.0094 (11)
C6	0.0531 (15)	0.0392 (14)	0.0681 (18)	−0.0111 (12)	−0.0003 (13)	−0.0149 (13)
C7	0.0617 (17)	0.0408 (14)	0.0759 (19)	−0.0036 (13)	−0.0105 (15)	−0.0259 (14)
C8	0.0475 (14)	0.0331 (12)	0.0450 (14)	−0.0075 (10)	−0.0073 (11)	−0.0092 (11)
C9	0.0595 (16)	0.0362 (13)	0.0405 (14)	−0.0052 (11)	−0.0059 (12)	−0.0060 (11)
C10	0.0459 (14)	0.0346 (12)	0.0447 (14)	−0.0027 (10)	−0.0072 (11)	−0.0071 (11)
C11	0.0557 (16)	0.0496 (15)	0.0467 (15)	−0.0009 (12)	−0.0049 (12)	−0.0025 (12)
C12	0.0499 (15)	0.0515 (15)	0.0401 (14)	−0.0078 (12)	−0.0016 (11)	−0.0099 (11)
C17	0.0535 (15)	0.0524 (15)	0.0478 (15)	−0.0058 (13)	−0.0060 (12)	−0.0136 (12)
C16	0.0600 (17)	0.0582 (17)	0.0561 (17)	0.0031 (14)	−0.0026 (13)	−0.0077 (14)
C15	0.0662 (19)	0.0526 (17)	0.0669 (19)	−0.0124 (14)	0.0146 (15)	−0.0175 (15)
C14	0.074 (2)	0.109 (3)	0.070 (2)	−0.016 (2)	0.0051 (17)	−0.055 (2)
C13	0.0578 (18)	0.106 (3)	0.0529 (17)	0.0029 (17)	−0.0105 (14)	−0.0325 (17)
C18	0.116 (3)	0.063 (2)	0.112 (3)	−0.003 (2)	0.032 (2)	−0.032 (2)
C19	0.0471 (14)	0.0397 (13)	0.0500 (15)	0.0019 (11)	−0.0091 (11)	−0.0010 (11)
C20	0.0540 (16)	0.0381 (14)	0.0522 (15)	0.0030 (12)	−0.0057 (13)	−0.0041 (12)
C21	0.133 (3)	0.0462 (18)	0.085 (2)	−0.0256 (19)	−0.012 (2)	0.0059 (17)
C22	0.199 (13)	0.099 (7)	0.050 (4)	−0.063 (7)	0.004 (6)	0.004 (6)
C22′	0.199 (13)	0.099 (7)	0.050 (4)	−0.063 (7)	0.004 (6)	0.004 (6)
N1	0.0566 (13)	0.0345 (11)	0.0443 (12)	−0.0018 (9)	−0.0086 (10)	−0.0061 (9)
N2	0.0462 (11)	0.0331 (10)	0.0448 (12)	−0.0018 (9)	−0.0081 (9)	−0.0029 (9)
O1	0.1031 (18)	0.0828 (16)	0.0517 (12)	0.0282 (14)	0.0041 (12)	0.0128 (11)
O2	0.0675 (13)	0.0612 (13)	0.0934 (16)	−0.0175 (11)	−0.0261 (12)	0.0010 (11)
O3	0.0886 (14)	0.0412 (10)	0.0577 (12)	−0.0074 (10)	−0.0135 (10)	0.0037 (9)

Geometric parameters (Å, º)

C1—C2	1.503 (4)	C16—C15	1.375 (4)
C1—H1A	0.9600	C16—H16	0.9300
C1—H1B	0.9600	C15—C14	1.383 (4)
C1—H1C	0.9600	C15—C18	1.506 (4)
C2—C7	1.377 (4)	C14—C13	1.379 (4)
C2—C3	1.386 (4)	C14—H14	0.9300
C3—C4	1.377 (4)	C13—H13	0.9300
C3—H3	0.9300	C18—H18A	0.9600
C4—C5	1.389 (3)	C18—H18B	0.9600
C4—H4	0.9300	C18—H18C	0.9600
C5—C6	1.390 (3)	C19—N2	1.459 (3)
C5—C8	1.463 (3)	C19—C20	1.503 (4)
C6—C7	1.381 (4)	C19—H19A	0.9700
C6—H6	0.9300	C19—H19B	0.9700
C7—H7	0.9300	C20—O2	1.193 (3)
C8—C9	1.371 (3)	C20—O3	1.325 (3)
C8—N2	1.372 (3)	C21—O3	1.462 (4)
C9—N1	1.349 (3)	C21—C22	1.534 (2)
C9—H9	0.9300	C21—C22′	1.535 (2)
C10—N1	1.329 (3)	C21—H21A	0.9700
C10—N2	1.373 (3)	C21—H21B	0.9700
C10—C11	1.460 (4)	C22—H22A	0.9600
C11—O1	1.227 (3)	C22—H22B	0.9600
C11—C12	1.484 (4)	C22—H22C	0.9600
C12—C13	1.379 (4)	C22′—H22D	0.9600
C12—C17	1.384 (3)	C22′—H22E	0.9600
C17—C16	1.373 (4)	C22′—H22F	0.9600
C17—H17	0.9300		
C2—C1—H1A	109.5	C14—C15—C18	121.8 (3)
C2—C1—H1B	109.5	C13—C14—C15	121.5 (3)
H1A—C1—H1B	109.5	C13—C14—H14	119.2
C2—C1—H1C	109.5	C15—C14—H14	119.2
H1A—C1—H1C	109.5	C12—C13—C14	120.4 (3)
H1B—C1—H1C	109.5	C12—C13—H13	119.8
C7—C2—C3	117.3 (3)	C14—C13—H13	119.8
C7—C2—C1	121.5 (3)	C15—C18—H18A	109.5
C3—C2—C1	121.2 (3)	C15—C18—H18B	109.5
C4—C3—C2	121.6 (3)	H18A—C18—H18B	109.5
C4—C3—H3	119.2	C15—C18—H18C	109.5
C2—C3—H3	119.2	H18A—C18—H18C	109.5
C3—C4—C5	120.8 (2)	H18B—C18—H18C	109.5
C3—C4—H4	119.6	N2—C19—C20	111.7 (2)
C5—C4—H4	119.6	N2—C19—H19A	109.3
C4—C5—C6	117.8 (2)	C20—C19—H19A	109.3
C4—C5—C8	119.7 (2)	N2—C19—H19B	109.3

C6—C5—C8	122.3 (2)	C20—C19—H19B	109.3
C7—C6—C5	120.5 (2)	H19A—C19—H19B	107.9
C7—C6—H6	119.7	O2—C20—O3	124.9 (2)
C5—C6—H6	119.7	O2—C20—C19	125.1 (2)
C2—C7—C6	121.9 (2)	O3—C20—C19	109.9 (2)
C2—C7—H7	119.1	O3—C21—C22	111.1 (9)
C6—C7—H7	119.1	O3—C21—C22′	102.4 (9)
C9—C8—N2	104.8 (2)	O3—C21—H21A	109.4
C9—C8—C5	129.3 (2)	C22—C21—H21A	109.4
N2—C8—C5	125.8 (2)	O3—C21—H21B	109.4
N1—C9—C8	112.0 (2)	C22—C21—H21B	109.4
N1—C9—H9	124.0	H21A—C21—H21B	108.0
C8—C9—H9	124.0	C21—C22—H22A	109.5
N1—C10—N2	111.2 (2)	C21—C22—H22B	109.5
N1—C10—C11	124.2 (2)	H22A—C22—H22B	109.5
N2—C10—C11	124.5 (2)	C21—C22—H22C	109.5
O1—C11—C10	120.8 (2)	H22A—C22—H22C	109.5
O1—C11—C12	120.8 (2)	H22B—C22—H22C	109.5
C10—C11—C12	118.3 (2)	C21—C22′—H22D	109.5
C13—C12—C17	118.2 (3)	C21—C22′—H22E	109.5
C13—C12—C11	118.7 (2)	H22D—C22′—H22E	109.5
C17—C12—C11	123.2 (2)	C21—C22′—H22F	109.5
C16—C17—C12	120.8 (3)	H22D—C22′—H22F	109.5
C16—C17—H17	119.6	H22E—C22′—H22F	109.5
C12—C17—H17	119.6	C10—N1—C9	105.0 (2)
C17—C16—C15	121.5 (3)	C8—N2—C10	106.94 (18)
C17—C16—H16	119.2	C8—N2—C19	125.8 (2)
C15—C16—H16	119.2	C10—N2—C19	126.8 (2)
C16—C15—C14	117.4 (3)	C20—O3—C21	114.8 (2)
C16—C15—C18	120.8 (3)		

C7—C2—C3—C4	0.7 (4)	C17—C16—C15—C14	3.4 (4)
C1—C2—C3—C4	−178.8 (3)	C17—C16—C15—C18	−176.0 (3)
C2—C3—C4—C5	−0.9 (4)	C16—C15—C14—C13	−1.4 (5)
C3—C4—C5—C6	0.6 (4)	C18—C15—C14—C13	178.0 (3)
C3—C4—C5—C8	175.8 (2)	C17—C12—C13—C14	3.8 (4)
C4—C5—C6—C7	0.0 (4)	C11—C12—C13—C14	−176.6 (3)
C8—C5—C6—C7	−175.1 (2)	C15—C14—C13—C12	−2.3 (5)
C3—C2—C7—C6	−0.2 (4)	N2—C19—C20—O2	−20.6 (4)
C1—C2—C7—C6	179.4 (3)	N2—C19—C20—O3	161.4 (2)
C5—C6—C7—C2	−0.2 (4)	N2—C10—N1—C9	−0.2 (3)
C4—C5—C8—C9	−51.0 (4)	C11—C10—N1—C9	−177.1 (2)
C6—C5—C8—C9	124.0 (3)	C8—C9—N1—C10	−0.3 (3)
C4—C5—C8—N2	131.5 (3)	C9—C8—N2—C10	−0.7 (3)
C6—C5—C8—N2	−53.5 (4)	C5—C8—N2—C10	177.3 (2)
N2—C8—C9—N1	0.7 (3)	C9—C8—N2—C19	172.2 (2)
C5—C8—C9—N1	−177.2 (2)	C5—C8—N2—C19	−9.8 (4)
N1—C10—C11—O1	175.3 (3)	N1—C10—N2—C8	0.6 (3)

N2—C10—C11—O1	−1.2 (4)	C11—C10—N2—C8	177.5 (2)
N1—C10—C11—C12	−2.5 (4)	N1—C10—N2—C19	−172.3 (2)
N2—C10—C11—C12	−179.0 (2)	C11—C10—N2—C19	4.7 (4)
O1—C11—C12—C13	−39.9 (4)	C20—C19—N2—C8	111.7 (3)
C10—C11—C12—C13	137.9 (3)	C20—C19—N2—C10	−76.8 (3)
O1—C11—C12—C17	139.7 (3)	O2—C20—O3—C21	3.5 (4)
C10—C11—C12—C17	−42.6 (4)	C19—C20—O3—C21	−178.5 (2)
C13—C12—C17—C16	−1.8 (4)	C22—C21—O3—C20	160.0 (6)
C11—C12—C17—C16	178.6 (2)	C22′—C21—O3—C20	−172.9 (6)
C12—C17—C16—C15	−1.9 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···O2	0.93	2.91	3.723 (4)	147
C1—H1*A*···O2^{i}	0.96	2.71	3.605 (4)	155
C4—H4···N1^{ii}	0.93	2.83	3.724 (3)	161
C19—H19*A*···O2^{iii}	0.97	2.51	3.309 (3)	140

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