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Trimethyl 5-(2-chloro-4-fluorophenyl)-2-phenylpyrrolidine-2,3,4-tricarboxylate

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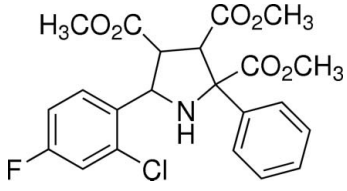
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.002 Å; disorder in main residue; R factor = 0.024; wR factor = 0.060; data-to-parameter ratio = 11.9.

The title compound, $C_{22}H_{21}ClFNO_6$, was synthesized by the 1,3-dipolar cycloaddition reaction of dimethyl maleate, methyl 2-amino-2-phenylacetate and 2-chloro-4-fluorobenzaldehyde. The pyrrolidine ring possesses an envelope conformation and the two benzene rings are oriented at a dihedral angle of 68.28 (7)°. Weak intermolecular C—H···O hydrogen bonding is present in the crystal structure. One methyl group is disordered over two positions with a site-occupancy ratio of 0.651 (12):0.349 (12).

Related literature

For the biological activity of pyrrolidine derivatives, see: Coldham & Hufton (2005); Nair & Suja (2007); Pandey *et al.* (2006); Sardina & Rapoport (1996); Witherup *et al.* (1995). For a related structure, see: Yu *et al.* (2007).



Experimental

Crystal data

$C_{22}H_{21}ClFNO_6$
M_r = 449.85
Orthorhombic, $P2_12_12_1$
a = 9.474 (3) Å
b = 15.057 (8) Å
c = 15.182 (5) Å
V = 2165.7 (15) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 1.98 mm^{-1}
T = 298 K
0.38 × 0.36 × 0.03 mm

Data collection

Oxford Diffraction Gemini S Ultra diffractometer
Absorption correction: multi-scan (*CrysAlis Pro*; Oxford Diffraction, 2009)
T_{min} = 0.520, T_{max} = 0.943
32943 measured reflections
3442 independent reflections
3332 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.060
S = 1.05
3442 reflections
290 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Absolute structure: Flack (1983), 1456 Friedel pairs
Flack parameter: −0.001 (11)

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

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6···O5^{i}	0.93	2.56	3.380 (3)	147

Symmetry code: (i) $x+\frac{1}{2}, -y+\frac{3}{2}, -z+1$.

Data collection: *CrysAlis Pro* (Oxford Diffraction, 2009); cell refinement: *CrysAlis Pro*; data reduction: *CrysAlis Pro*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

The diffraction measurements were made at Sichuan University. We acknowledge financial support from China West Normal University.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2655).

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supporting information

Acta Cryst. (2009). E**65**, o2927 [https://doi.org/10.1107/S1600536809044274]

Trimethyl 5-(2-chloro-4-fluorophenyl)-2-phenylpyrrolidine-2,3,4-tricarboxylate

Long He and Lian-Mei Chen

S1. Comment

Substituted pyrrolidine compound is an important class of heterocyclic compounds with wide spread applications to the synthesis of biologically active compounds and natural products (Coldham *et al.*, 2005; Nair *et al.*, 2007; Pandey *et al.*, 2006; Sardina *et al.*, 1996; Witherup *et al.* 1995).

The molecular structure of (I) is shown in Fig. 1. Bond lengths and angles in (I) are normal. The pyrrolidine ring possesses an envelope conformation. The dihedral angle between the C1—C6 and C11—C16 benzene planes is 68.28 (7)°.

S2. Experimental

2-Chloro-4-fluorobenzaldehyde (0.063 g, 0.4 mmol), anhydrous sodium sulfate (200 mg) and dimethyl maleate (0.029 g, 0.2 mmol) were added to a solution of methyl 2-amino-2-phenylacetate(0.049 g, 0.3 mmol) in chloroform (2 ml). To the stirred mixture, acetic acid (0.012 g, 0.2 mmol) was added. After the mixture had been stirred at 323k for 10 h, the reaction was quenched with a saturated solution of sodium bicarbonate (5 ml). The mixture was extracted with diethyl ether, evaporated and separated by flash chromatograghy. A colourless powder was obtained. Single crystals suitable for X-ray diffraction were obtained by slow evaporation of an ethyl acetate solution.

S3. Refinement

Imino H atom was located in a difference Fourier map and positional parameters were refined, U_{iso}(H) = 1.2U_{eq}(N). The carbon-bound hydrogen atoms were placed in calculated positions, with C—H = 0.93–0.98 Å, and refined using a riding model, with U_{iso}(H) =1.5U_{eq}(C) for methyl H atoms and 1.2U_{eq}(C) for the others. One of methyl groups is disordered over two positions, site occupancy factors were refined to 0.651 (12):0.349 (12).

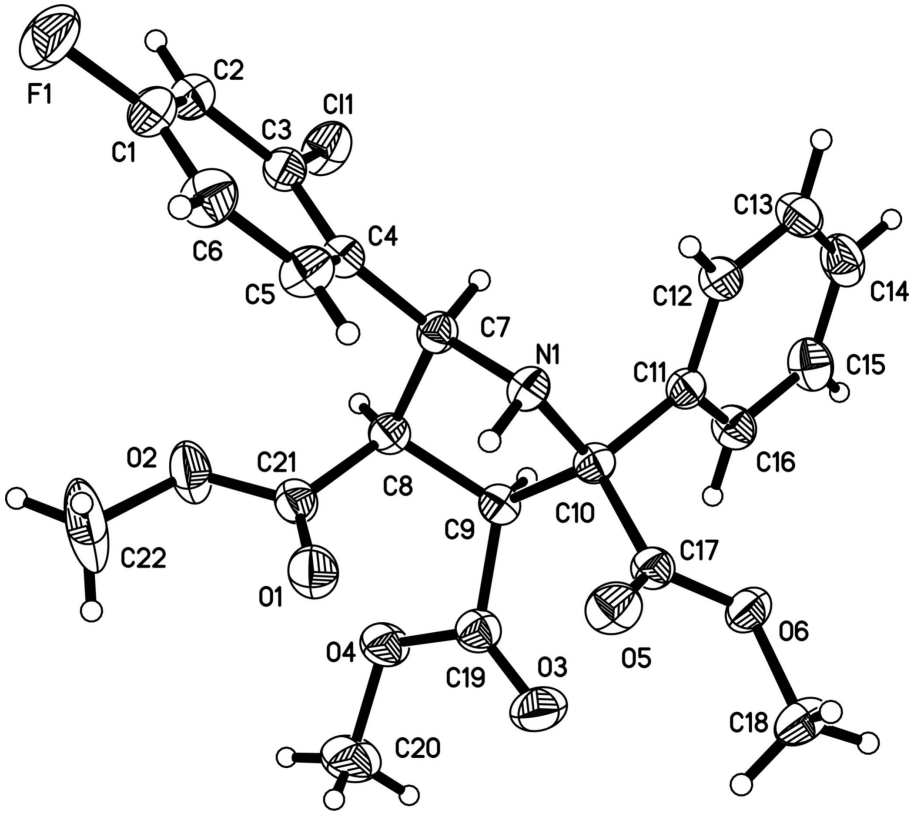

Figure 1

The molecular structure of (I) with 30% probability displacement ellipsoids (arbitrary spheres for H atoms).

Trimethyl 5-(2-chloro-4-fluorophenyl)-2-phenylpyrrolidine-2,3,4-tricarboxylate

Crystal data

$C_{22}H_{21}ClFNO_6$
$M_r = 449.85$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 9.474\ (3)$ Å
$b = 15.057\ (8)$ Å
$c = 15.182\ (5)$ Å
$V = 2165.7\ (15)$ Å^3
$Z = 4$
$F(000) = 936$
$D_x = 1.380$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 23516 reflections
$\theta = 2.9–62.8°$
$\mu = 1.98$ mm^{-1}
$T = 298$ K
Platelet, colourless
0.38 × 0.36 × 0.03 mm

Data collection

Oxford Diffraction Gemini S Ultra diffractometer
Radiation source: Enhance Ultra (Cu) X-ray Source
Mirror monochromator
Detector resolution: 15.9149 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2009)
$T_{min} = 0.520$, $T_{max} = 0.943$
32943 measured reflections
3442 independent reflections
3332 reflections with $I > 2\sigma(I)$
$R_{int} = 0.025$
$\theta_{max} = 62.6°$, $\theta_{min} = 4.1°$
$h = -10\rightarrow10$
$k = -17\rightarrow17$
$l = -17\rightarrow17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.024$
$wR(F^2) = 0.060$
$S = 1.05$
3442 reflections
290 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0318P)^2 + 0.3155P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.12$ e Å^{-3}
$\Delta\rho_{min} = -0.16$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0055 (2)
Absolute structure: Flack (1983), 1456 Friedel pairs
Absolute structure parameter: −0.001 (11)

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}	Occ. (<1)
Cl1	0.87819 (6)	0.40571 (3)	0.69190 (3)	0.06039 (14)	
F1	1.10815 (14)	0.42498 (8)	0.39470 (7)	0.0789 (4)	
O1	0.64936 (15)	0.70388 (7)	0.55124 (8)	0.0578 (3)	
O2	0.59938 (18)	0.55992 (8)	0.53671 (8)	0.0715 (4)	
O3	0.50206 (14)	0.80786 (9)	0.73253 (11)	0.0735 (4)	
O4	0.41937 (11)	0.67622 (8)	0.68953 (9)	0.0568 (3)	
O5	0.79799 (13)	0.86655 (7)	0.67762 (8)	0.0553 (3)	
O6	0.74501 (13)	0.87248 (7)	0.82137 (7)	0.0509 (3)	
N1	0.89048 (14)	0.69866 (8)	0.69034 (9)	0.0390 (3)	
H1	0.870 (2)	0.7278 (11)	0.6438 (12)	0.047*	
C1	1.0498 (2)	0.46980 (13)	0.46390 (11)	0.0546 (4)	
C2	1.00115 (19)	0.42137 (12)	0.53368 (11)	0.0500 (4)	
H2	1.0094	0.3598	0.5350	0.060*	
C3	0.93922 (17)	0.46752 (10)	0.60228 (10)	0.0417 (4)	
C4	0.92532 (15)	0.55915 (10)	0.60251 (10)	0.0386 (3)	
C5	0.98023 (19)	0.60471 (12)	0.53049 (11)	0.0509 (4)	
H5	0.9745	0.6664	0.5293	0.061*	
C6	1.0431 (2)	0.56086 (13)	0.46054 (12)	0.0588 (5)	
H6	1.0796	0.5921	0.4128	0.071*	
C7	0.85178 (16)	0.60535 (9)	0.67778 (9)	0.0379 (3)	
H7	0.8783	0.5738	0.7318	0.045*	
C8	0.68738 (15)	0.60534 (10)	0.67347 (10)	0.0393 (3)	

H8	0.6491	0.5473	0.6902	0.047*	
C9	0.65018 (15)	0.67691 (10)	0.74326 (10)	0.0390 (3)	
H9	0.6296	0.6450	0.7980	0.047*	
C10	0.79423 (16)	0.72962 (10)	0.75935 (10)	0.0372 (3)	
C11	0.85468 (17)	0.70603 (9)	0.85036 (10)	0.0386 (3)	
C12	0.99477 (17)	0.68067 (11)	0.86012 (11)	0.0468 (4)	
H12	1.0537	0.6788	0.8111	0.056*	
C13	1.0475 (2)	0.65805 (12)	0.94231 (13)	0.0589 (5)	
H13	1.1410	0.6401	0.9477	0.071*	
C14	0.9633 (2)	0.66181 (12)	1.01602 (12)	0.0604 (5)	
H14	0.9992	0.6464	1.0710	0.073*	
C15	0.8256 (2)	0.68856 (12)	1.00731 (12)	0.0565 (5)	
H15	0.7685	0.6928	1.0569	0.068*	
C16	0.77124 (19)	0.70915 (11)	0.92558 (11)	0.0478 (4)	
H16	0.6769	0.7254	0.9207	0.057*	
C17	0.77865 (16)	0.83067 (10)	0.74725 (11)	0.0409 (3)	
C18	0.7123 (2)	0.96590 (11)	0.81177 (15)	0.0666 (5)	
H18A	0.7012	0.9922	0.8690	0.100*	
H18B	0.6263	0.9724	0.7790	0.100*	
H18C	0.7878	0.9950	0.7810	0.100*	
C19	0.51912 (18)	0.73007 (12)	0.72121 (11)	0.0462 (4)	
C20	0.2893 (2)	0.71857 (16)	0.66086 (19)	0.0810 (7)	
H20A	0.2229	0.6739	0.6429	0.121*	
H20B	0.3088	0.7573	0.6121	0.121*	
H20C	0.2502	0.7524	0.7086	0.121*	
C21	0.64215 (18)	0.63050 (10)	0.58152 (10)	0.0442 (4)	
C22A	0.5863 (10)	0.5705 (4)	0.4390 (3)	0.0813 (19)	0.651 (12)
H22A	0.6715	0.5505	0.4112	0.122*	0.651 (12)
H22B	0.5708	0.6319	0.4251	0.122*	0.651 (12)
H22C	0.5081	0.5358	0.4181	0.122*	0.651 (12)
C22B	0.5167 (16)	0.5848 (8)	0.4569 (7)	0.0813 (19)	0.349 (12)
H22D	0.4260	0.6070	0.4744	0.122*	0.349 (12)
H22E	0.5046	0.5335	0.4201	0.122*	0.349 (12)
H22F	0.5664	0.6300	0.4248	0.122*	0.349 (12)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0909 (3)	0.0400 (2)	0.0503 (2)	0.0019 (2)	0.0084 (2)	0.00626 (17)
F1	0.0926 (9)	0.0837 (8)	0.0604 (6)	0.0170 (7)	0.0228 (6)	−0.0173 (6)
O1	0.0762 (9)	0.0468 (7)	0.0505 (7)	−0.0035 (6)	−0.0099 (6)	0.0073 (5)
O2	0.1081 (12)	0.0502 (7)	0.0562 (7)	−0.0174 (7)	−0.0325 (7)	0.0010 (6)
O3	0.0495 (7)	0.0544 (8)	0.1167 (13)	0.0096 (6)	−0.0117 (7)	−0.0141 (8)
O4	0.0371 (6)	0.0568 (7)	0.0766 (8)	−0.0006 (5)	−0.0088 (6)	−0.0026 (7)
O5	0.0692 (8)	0.0449 (6)	0.0519 (7)	0.0018 (5)	0.0049 (6)	0.0112 (5)
O6	0.0651 (8)	0.0368 (5)	0.0507 (6)	0.0065 (5)	−0.0016 (6)	−0.0044 (5)
N1	0.0423 (7)	0.0352 (6)	0.0393 (6)	−0.0019 (5)	0.0035 (6)	−0.0011 (5)
C1	0.0541 (10)	0.0627 (11)	0.0468 (10)	0.0104 (9)	0.0073 (8)	−0.0100 (8)

C2	0.0556 (10)	0.0442 (9)	0.0502 (9)	0.0081 (8)	−0.0030 (8)	−0.0062 (7)
C3	0.0452 (9)	0.0401 (8)	0.0399 (8)	0.0012 (7)	−0.0021 (7)	0.0014 (6)
C4	0.0372 (8)	0.0399 (8)	0.0387 (8)	0.0003 (6)	−0.0026 (6)	0.0005 (6)
C5	0.0582 (10)	0.0446 (9)	0.0499 (9)	0.0028 (8)	0.0104 (8)	0.0041 (7)
C6	0.0666 (12)	0.0620 (12)	0.0478 (10)	0.0016 (10)	0.0155 (9)	0.0046 (8)
C7	0.0412 (8)	0.0349 (7)	0.0376 (7)	−0.0011 (6)	0.0000 (6)	0.0004 (6)
C8	0.0409 (8)	0.0351 (7)	0.0419 (8)	−0.0020 (6)	−0.0008 (6)	0.0021 (6)
C9	0.0374 (8)	0.0407 (8)	0.0389 (8)	−0.0026 (7)	0.0007 (7)	0.0022 (6)
C10	0.0365 (8)	0.0372 (8)	0.0380 (8)	0.0002 (6)	0.0017 (6)	0.0001 (6)
C11	0.0420 (8)	0.0329 (7)	0.0410 (8)	−0.0010 (6)	−0.0030 (7)	0.0002 (6)
C12	0.0405 (9)	0.0459 (9)	0.0541 (10)	−0.0006 (7)	−0.0036 (7)	−0.0052 (7)
C13	0.0521 (11)	0.0548 (10)	0.0697 (12)	0.0039 (9)	−0.0210 (10)	0.0005 (9)
C14	0.0737 (14)	0.0547 (11)	0.0529 (11)	−0.0088 (10)	−0.0200 (10)	0.0087 (9)
C15	0.0655 (12)	0.0618 (10)	0.0421 (9)	−0.0092 (9)	−0.0007 (8)	0.0058 (8)
C16	0.0450 (9)	0.0541 (9)	0.0443 (9)	0.0005 (7)	0.0005 (7)	0.0030 (7)
C17	0.0386 (8)	0.0377 (8)	0.0465 (9)	−0.0006 (6)	−0.0021 (7)	0.0002 (7)
C18	0.0837 (13)	0.0395 (9)	0.0766 (13)	0.0132 (9)	−0.0060 (12)	−0.0070 (9)
C19	0.0404 (9)	0.0466 (10)	0.0517 (9)	0.0001 (7)	0.0018 (7)	−0.0010 (7)
C20	0.0436 (11)	0.0816 (15)	0.1177 (19)	0.0075 (10)	−0.0185 (12)	−0.0039 (14)
C21	0.0432 (9)	0.0443 (9)	0.0450 (8)	−0.0014 (7)	−0.0051 (7)	−0.0002 (7)
C22A	0.126 (6)	0.070 (2)	0.0474 (18)	−0.010 (3)	−0.027 (2)	−0.0089 (17)
C22B	0.126 (6)	0.070 (2)	0.0474 (18)	−0.010 (3)	−0.027 (2)	−0.0089 (17)

Geometric parameters (Å, º)

Cl1—C3	1.7469 (16)	C8—H8	0.9800
F1—C1	1.366 (2)	C9—C19	1.515 (2)
O1—C21	1.1987 (19)	C9—C10	1.597 (2)
O2—C21	1.325 (2)	C9—H9	0.9800
O2—C22B	1.490 (7)	C10—C11	1.537 (2)
O2—C22A	1.497 (4)	C10—C17	1.540 (2)
O3—C19	1.195 (2)	C11—C12	1.389 (2)
O4—C19	1.335 (2)	C11—C16	1.390 (2)
O4—C20	1.454 (2)	C12—C13	1.387 (3)
O5—C17	1.201 (2)	C12—H12	0.9300
O6—C17	1.3283 (19)	C13—C14	1.375 (3)
O6—C18	1.448 (2)	C13—H13	0.9300
N1—C7	1.4645 (18)	C14—C15	1.371 (3)
N1—C10	1.465 (2)	C14—H14	0.9300
N1—H1	0.855 (18)	C15—C16	1.379 (2)
C1—C2	1.366 (3)	C15—H15	0.9300
C1—C6	1.373 (3)	C16—H16	0.9300
C2—C3	1.383 (2)	C18—H18A	0.9600
C2—H2	0.9300	C18—H18B	0.9600
C3—C4	1.386 (2)	C18—H18C	0.9600
C4—C5	1.392 (2)	C20—H20A	0.9600
C4—C7	1.508 (2)	C20—H20B	0.9600
C5—C6	1.385 (2)	C20—H20C	0.9600

C5—H5	0.9300	C22A—H22A	0.9600
C6—H6	0.9300	C22A—H22B	0.9600
C7—C8	1.559 (2)	C22A—H22C	0.9600
C7—H7	0.9800	C22B—H22D	0.9600
C8—C21	1.509 (2)	C22B—H22E	0.9600
C8—C9	1.552 (2)	C22B—H22F	0.9600

C21—O2—C22B	112.1 (5)	C12—C11—C10	121.02 (14)
C21—O2—C22A	116.7 (3)	C16—C11—C10	121.26 (14)
C22B—O2—C22A	28.9 (4)	C13—C12—C11	120.50 (17)
C19—O4—C20	116.19 (15)	C13—C12—H12	119.7
C17—O6—C18	115.25 (14)	C11—C12—H12	119.7
C7—N1—C10	104.04 (11)	C14—C13—C12	120.89 (17)
C7—N1—H1	109.1 (12)	C14—C13—H13	119.6
C10—N1—H1	106.6 (12)	C12—C13—H13	119.6
F1—C1—C2	117.99 (17)	C15—C14—C13	119.02 (17)
F1—C1—C6	118.91 (17)	C15—C14—H14	120.5
C2—C1—C6	123.10 (16)	C13—C14—H14	120.5
C1—C2—C3	117.32 (16)	C14—C15—C16	120.55 (18)
C1—C2—H2	121.3	C14—C15—H15	119.7
C3—C2—H2	121.3	C16—C15—H15	119.7
C2—C3—C4	122.85 (15)	C15—C16—C11	121.28 (17)
C2—C3—Cl1	117.35 (12)	C15—C16—H16	119.4
C4—C3—Cl1	119.79 (12)	C11—C16—H16	119.4
C3—C4—C5	116.95 (14)	O5—C17—O6	124.68 (14)
C3—C4—C7	120.33 (13)	O5—C17—C10	122.33 (14)
C5—C4—C7	122.72 (14)	O6—C17—C10	112.97 (13)
C6—C5—C4	121.88 (16)	O6—C18—H18A	109.5
C6—C5—H5	119.1	O6—C18—H18B	109.5
C4—C5—H5	119.1	H18A—C18—H18B	109.5
C1—C6—C5	117.86 (17)	O6—C18—H18C	109.5
C1—C6—H6	121.1	H18A—C18—H18C	109.5
C5—C6—H6	121.1	H18B—C18—H18C	109.5
N1—C7—C4	115.18 (12)	O3—C19—O4	123.47 (16)
N1—C7—C8	104.82 (12)	O3—C19—C9	126.67 (16)
C4—C7—C8	115.45 (12)	O4—C19—C9	109.81 (14)
N1—C7—H7	107.0	O4—C20—H20A	109.5
C4—C7—H7	107.0	O4—C20—H20B	109.5
C8—C7—H7	107.0	H20A—C20—H20B	109.5
C21—C8—C9	113.13 (12)	O4—C20—H20C	109.5
C21—C8—C7	108.82 (12)	H20A—C20—H20C	109.5
C9—C8—C7	101.43 (11)	H20B—C20—H20C	109.5
C21—C8—H8	111.0	O1—C21—O2	124.03 (15)
C9—C8—H8	111.0	O1—C21—C8	124.77 (14)
C7—C8—H8	111.0	O2—C21—C8	111.13 (13)
C19—C9—C8	113.72 (13)	O2—C22A—H22A	109.5
C19—C9—C10	118.14 (13)	O2—C22A—H22B	109.5
C8—C9—C10	104.80 (12)	H22A—C22A—H22B	109.5

C19—C9—H9	106.5	O2—C22A—H22C	109.5
C8—C9—H9	106.5	H22A—C22A—H22C	109.5
C10—C9—H9	106.5	H22B—C22A—H22C	109.5
N1—C10—C11	109.71 (12)	O2—C22B—H22D	109.5
N1—C10—C17	106.78 (12)	O2—C22B—H22E	109.5
C11—C10—C17	111.80 (12)	H22D—C22B—H22E	109.5
N1—C10—C9	105.33 (12)	O2—C22B—H22F	109.5
C11—C10—C9	109.94 (12)	H22D—C22B—H22F	109.5
C17—C10—C9	113.00 (12)	H22E—C22B—H22F	109.5
C12—C11—C16	117.72 (15)		

F1—C1—C2—C3	−178.45 (16)	N1—C10—C11—C12	14.85 (19)
C6—C1—C2—C3	1.8 (3)	C17—C10—C11—C12	−103.41 (16)
C1—C2—C3—C4	0.0 (3)	C9—C10—C11—C12	130.23 (15)
C1—C2—C3—Cl1	−179.53 (14)	N1—C10—C11—C16	−165.02 (14)
C2—C3—C4—C5	−1.5 (2)	C17—C10—C11—C16	76.71 (18)
Cl1—C3—C4—C5	177.95 (12)	C9—C10—C11—C16	−49.64 (18)
C2—C3—C4—C7	177.68 (14)	C16—C11—C12—C13	1.0 (2)
Cl1—C3—C4—C7	−2.8 (2)	C10—C11—C12—C13	−178.87 (15)
C3—C4—C5—C6	1.5 (3)	C11—C12—C13—C14	−1.2 (3)
C7—C4—C5—C6	−177.73 (16)	C12—C13—C14—C15	−0.1 (3)
F1—C1—C6—C5	178.39 (17)	C13—C14—C15—C16	1.7 (3)
C2—C1—C6—C5	−1.9 (3)	C14—C15—C16—C11	−1.9 (3)
C4—C5—C6—C1	0.2 (3)	C12—C11—C16—C15	0.6 (2)
C10—N1—C7—C4	173.79 (12)	C10—C11—C16—C15	−179.57 (15)
C10—N1—C7—C8	45.80 (14)	C18—O6—C17—O5	8.4 (2)
C3—C4—C7—N1	156.57 (14)	C18—O6—C17—C10	−172.84 (15)
C5—C4—C7—N1	−24.3 (2)	N1—C10—C17—O5	24.6 (2)
C3—C4—C7—C8	−80.97 (18)	C11—C10—C17—O5	144.60 (15)
C5—C4—C7—C8	98.19 (18)	C9—C10—C17—O5	−90.74 (18)
N1—C7—C8—C21	81.72 (14)	N1—C10—C17—O6	−154.16 (13)
C4—C7—C8—C21	−46.10 (17)	C11—C10—C17—O6	−34.15 (18)
N1—C7—C8—C9	−37.78 (14)	C9—C10—C17—O6	90.51 (15)
C4—C7—C8—C9	−165.60 (12)	C20—O4—C19—O3	5.6 (3)
C21—C8—C9—C19	30.15 (18)	C20—O4—C19—C9	−176.82 (16)
C7—C8—C9—C19	146.52 (13)	C8—C9—C19—O3	−140.85 (18)
C21—C8—C9—C10	−100.31 (14)	C10—C9—C19—O3	−17.4 (3)
C7—C8—C9—C10	16.07 (14)	C8—C9—C19—O4	41.67 (18)
C7—N1—C10—C11	84.15 (14)	C10—C9—C19—O4	165.13 (13)
C7—N1—C10—C17	−154.52 (12)	C22B—O2—C21—O1	−19.4 (7)
C7—N1—C10—C9	−34.13 (14)	C22A—O2—C21—O1	12.0 (5)
C19—C9—C10—N1	−117.73 (14)	C22B—O2—C21—C8	163.6 (7)
C8—C9—C10—N1	10.09 (15)	C22A—O2—C21—C8	−165.1 (5)
C19—C9—C10—C11	124.14 (14)	C9—C8—C21—O1	38.7 (2)
C8—C9—C10—C11	−108.04 (13)	C7—C8—C21—O1	−73.2 (2)
C19—C9—C10—C17	−1.53 (19)	C9—C8—C21—O2	−144.27 (15)
C8—C9—C10—C17	126.29 (13)	C7—C8—C21—O2	103.82 (16)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···O5^{i}	0.93	2.56	3.380 (3)	147

Symmetry code: (i) *x*+1/2, −*y*+3/2, −*z*+1.