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2,2-Diethyl 3,4-dimethyl 5-(4-cyanophenyl)pyrrolidine-2,2,3,4-tetracarboxylate

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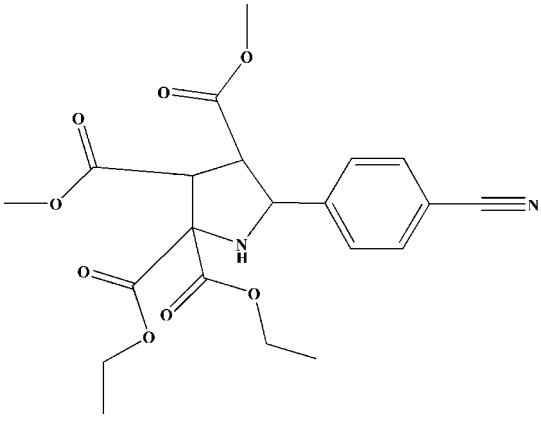
Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.005 Å; R factor = 0.050; wR factor = 0.141; data-to-parameter ratio = 8.9.

The title compound, $C_{21}H_{24}N_2O_8$, was synthesized by a 1,3-dipolar cycloaddition reaction of dimethyl fumarate, diethyl 2-aminomalonate and 4-cyanobenzaldehyde. Both methyl ester groups display a *trans* configuration and the pyrrolidine ring possesses an envelope conformation, with the C atom in the 3-position as the flap. In the crystal, N—H···N hydrogen bonds and weak C—H···O interactions occur.

Related literature

For the biological activity of pyrrolidine derivatives, see: Coldham & Hufton (2005); Pandey *et al.* (2006); Schreiber *et al.* (2000). For a related structure, see: He *et al.* (2010).



Experimental

Crystal data

$C_{21}H_{24}N_2O_8$
M_r = 432.42
Orthorhombic, $P2_12_12_1$
a = 8.4720 (2) Å
b = 10.3043 (2) Å
c = 25.8774 (5) Å
V = 2259.05 (8) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 0.83 mm^{-1}
T = 291 K
0.38 × 0.30 × 0.30 mm

Data collection

Oxford Diffraction Gemini S Ultra diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2007)
T_{min} = 0.744, T_{max} = 0.789
26556 measured reflections
2565 independent reflections
2050 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.141
S = 1.03
2565 reflections
288 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H1···N1^{i}	0.86 (4)	2.47 (4)	3.240 (5)	148 (4)
C8—H8···O1^{ii}	0.98	2.39	3.333 (4)	161 (1)
C13—H13C···O7^{ii}	0.96	2.46	3.388 (6)	163
C18—H18C···O5^{iii}	0.96	2.55	3.453 (9)	157

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

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2007); 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5557).

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supporting information

Acta Cryst. (2012). E**68**, o2363 [https://doi.org/10.1107/S1600536812029625]

2,2-Diethyl 3,4-dimethyl 5-(4-cyanophenyl)pyrrolidine-2,2,3,4-tetracarboxylate

Long He

S1. Comment

Facile synthesis of structurally diverse heterocycles is important in chemical biology. Five-membered pyrrolidines compounds is an important class of heterocyclic compounds with wide spread applications to the synthesis of biologically active compounds and natural alkaloids (Coldham & Hufton, 2005; Pandey *et al.*, 2006; Schreiber *et al.*, 2000). Its crystal structure is reported here.

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 crystal packing is stabilized by C—H···O and N—H···N hydrogen bonds (Table 1).

S2. Experimental

To a solution of 4-cyanobenzaldehyde (0.065 g, 0.5 mmol), sodium sulflate (0.1 g) and diethyl 2-aminomalonate (0.070 g, 0.4 mmol) in chloroform (4 ml) was added dimethyl fumarate (0.145 g, 1 mmol). The mixture was stirred at 323 K for 2 d and then cooled to room temperature, the solvent was evaporated under reduced pressure and the residues was purified by flash chromatograghy on silica gel. The colourless powder was obtained. Single crystals suitable for X-ray diffraction were obtained by slow evaporation of an ethyl acetate solution.

S3. Refinement

H atoms on N atoms were located in a difference Fourier map and refined isotropically. 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 U_{iso}(H) =1.2U_{eq}(C) for the others. As no significant anomalous scatterings Friedel pairs were merged.

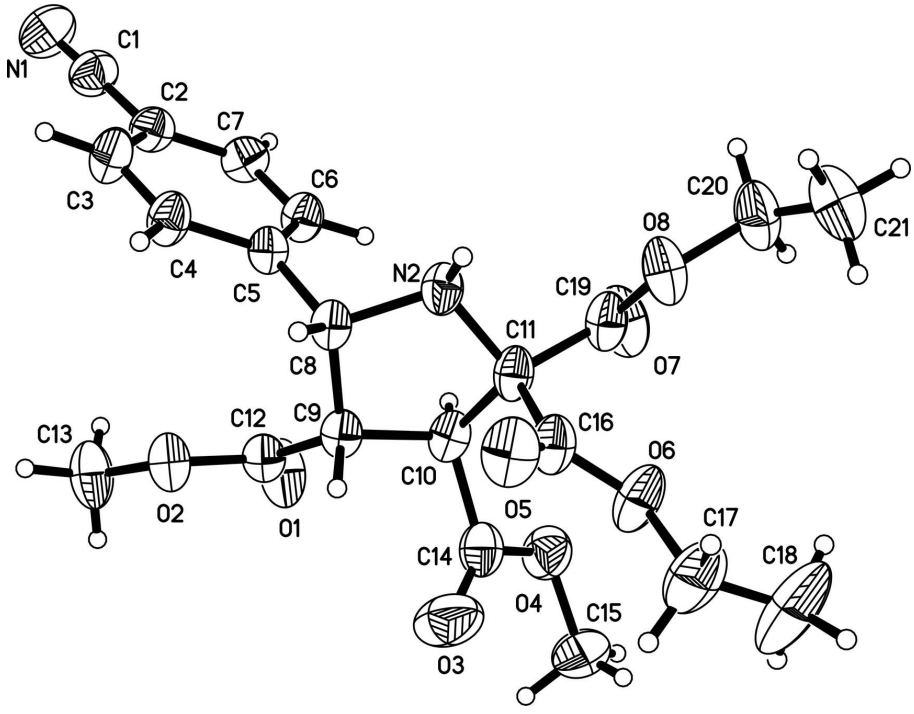

Figure 1

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

2,2-Diethyl 3,4-dimethyl 5-(4-cyanophenyl)pyrrolidine-2,2,3,4-tetracarboxylate

Crystal data

$C_{21}H_{24}N_2O_8$
$M_r = 432.42$
Orthorhombic, $P2_12_12_1$
$a = 8.4720\ (2)$ Å
$b = 10.3043\ (2)$ Å
$c = 25.8774\ (5)$ Å
$V = 2259.05\ (8)$ Å^3
$Z = 4$
$F(000) = 912$
$D_x = 1.271$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 10771 reflections
$\theta = 1.7$–$72.3°$
$\mu = 0.83$ mm^{-1}
$T = 291$ K
Block, colorless
0.38 × 0.30 × 0.30 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, 2007)
$T_{min} = 0.744$, $T_{max} = 0.789$
26556 measured reflections
2565 independent reflections
2050 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 72.6°$, $\theta_{min} = 3.4°$
$h = -10\rightarrow10$
$k = -12\rightarrow10$
$l = -31\rightarrow31$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.141$
$S = 1.03$
2565 reflections
288 parameters
2 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0724P)^2 + 0.5988P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.40$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0015 (4)

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}
O2	1.0668 (3)	0.9425 (2)	0.20700 (8)	0.0707 (7)
O5	1.0369 (4)	0.7873 (2)	0.40309 (11)	0.0880 (9)
O8	0.8498 (4)	1.0544 (3)	0.46182 (9)	0.0865 (9)
N2	0.8356 (4)	0.9769 (3)	0.35733 (10)	0.0622 (7)
O1	1.1491 (5)	1.1320 (3)	0.23785 (10)	0.0928 (10)
C9	1.0464 (4)	0.9727 (3)	0.29624 (11)	0.0548 (7)
H9	1.1073	0.8930	0.3016	0.066*
O6	1.1628 (4)	0.9441 (3)	0.44645 (10)	0.0898 (9)
O4	1.3158 (4)	1.1691 (3)	0.37339 (11)	0.0874 (8)
O7	0.9598 (5)	1.2258 (3)	0.42249 (12)	0.1046 (12)
C5	0.7612 (4)	1.0072 (3)	0.26561 (11)	0.0542 (7)
C2	0.5727 (4)	1.1296 (3)	0.19214 (13)	0.0632 (9)
C8	0.8696 (4)	0.9382 (3)	0.30325 (11)	0.0530 (7)
H8	0.8559	0.8442	0.2997	0.064*
C12	1.0926 (4)	1.0266 (3)	0.24488 (12)	0.0597 (8)
C6	0.7360 (4)	1.1396 (3)	0.26702 (13)	0.0620 (8)
H6	0.7825	1.1885	0.2931	0.074*
C10	1.0857 (4)	1.0649 (3)	0.34090 (12)	0.0592 (8)
H10	1.0539	1.1535	0.3320	0.071*
C4	0.6885 (5)	0.9354 (4)	0.22681 (13)	0.0668 (9)
H4	0.7047	0.8462	0.2251	0.080*
C11	0.9802 (4)	1.0124 (3)	0.38427 (12)	0.0602 (8)
C7	0.6433 (4)	1.2009 (3)	0.23057 (14)	0.0658 (9)

H7	0.6287	1.2903	0.2320	0.079*
C19	0.9328 (5)	1.1127 (3)	0.42459 (13)	0.0716 (10)
C16	1.0612 (5)	0.8987 (3)	0.41186 (13)	0.0683 (10)
C3	0.5921 (5)	0.9951 (4)	0.19062 (15)	0.0754 (11)
H3	0.5409	0.9460	0.1656	0.091*
C14	1.2688 (5)	1.0600 (4)	0.35092 (14)	0.0719 (10)
C13	1.0929 (7)	0.9876 (5)	0.15484 (13)	0.0955 (15)
H13A	1.2035	1.0033	0.1497	0.115*
H13B	1.0352	1.0665	0.1493	0.115*
H13C	1.0575	0.9228	0.1308	0.115*
C20	0.7915 (8)	1.1323 (5)	0.50465 (15)	0.1021 (16)
H20A	0.8668	1.2001	0.5128	0.122*
H20B	0.6923	1.1729	0.4952	0.122*
C17	1.2540 (8)	0.8528 (5)	0.47718 (19)	0.1127 (19)
H17A	1.1848	0.7871	0.4916	0.135*
H17B	1.3318	0.8100	0.4556	0.135*
C15	1.4850 (5)	1.1640 (7)	0.3860 (2)	0.1114 (17)
H15A	1.5435	1.1382	0.3559	0.167*
H15B	1.5020	1.1022	0.4132	0.167*
H15C	1.5198	1.2481	0.3971	0.167*
C21	0.7690 (9)	1.0492 (6)	0.54894 (18)	0.121 (2)
H21A	0.8670	1.0078	0.5575	0.145*
H21B	0.6912	0.9845	0.5410	0.145*
H21C	0.7337	1.1003	0.5778	0.145*
C18	1.3299 (11)	0.9207 (8)	0.5172 (3)	0.189 (4)
H18A	1.2602	0.9860	0.5306	0.227*
H18B	1.4238	0.9610	0.5040	0.227*
H18C	1.3575	0.8613	0.5443	0.227*
O3	1.3504 (4)	0.9759 (4)	0.33931 (18)	0.1249 (14)
N1	0.4052 (5)	1.2386 (4)	0.12122 (16)	0.0982 (12)
C1	0.4786 (5)	1.1909 (4)	0.15304 (15)	0.0752 (10)
H1	0.780 (5)	0.924 (4)	0.3757 (16)	0.113*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O2	0.1013 (19)	0.0581 (13)	0.0527 (11)	−0.0096 (14)	0.0014 (12)	−0.0020 (11)
O5	0.126 (2)	0.0517 (14)	0.0863 (18)	0.0007 (16)	−0.0135 (18)	0.0065 (12)
O8	0.134 (3)	0.0700 (15)	0.0558 (12)	−0.0049 (18)	0.0108 (16)	−0.0103 (12)
N2	0.0743 (17)	0.0655 (17)	0.0469 (13)	−0.0087 (16)	−0.0029 (13)	0.0032 (12)
O1	0.148 (3)	0.0629 (16)	0.0677 (15)	−0.0344 (19)	0.0105 (18)	0.0011 (13)
C9	0.0710 (19)	0.0417 (15)	0.0516 (15)	0.0009 (15)	−0.0052 (15)	0.0015 (13)
O6	0.121 (2)	0.0700 (15)	0.0782 (16)	−0.0008 (18)	−0.0441 (17)	0.0122 (13)
O4	0.0833 (17)	0.094 (2)	0.0853 (17)	−0.0168 (17)	−0.0061 (16)	−0.0093 (16)
O7	0.170 (4)	0.0548 (16)	0.089 (2)	−0.0019 (19)	0.017 (2)	−0.0071 (13)
C5	0.0664 (18)	0.0482 (16)	0.0479 (15)	−0.0017 (15)	−0.0027 (14)	−0.0003 (13)
C2	0.0652 (19)	0.064 (2)	0.0606 (18)	0.0040 (18)	−0.0029 (16)	0.0074 (16)
C8	0.0706 (19)	0.0414 (14)	0.0472 (14)	−0.0014 (15)	−0.0051 (13)	0.0015 (13)

C12	0.074 (2)	0.0487 (16)	0.0569 (17)	0.0013 (16)	−0.0020 (16)	0.0020 (14)
C6	0.077 (2)	0.0460 (17)	0.0625 (18)	−0.0003 (17)	−0.0093 (17)	−0.0035 (15)
C10	0.078 (2)	0.0439 (15)	0.0556 (16)	−0.0060 (16)	−0.0124 (15)	0.0046 (14)
C4	0.085 (2)	0.0502 (17)	0.0648 (18)	0.0030 (18)	−0.0177 (19)	−0.0071 (16)
C11	0.088 (2)	0.0463 (16)	0.0465 (14)	−0.0032 (17)	−0.0107 (16)	0.0025 (13)
C7	0.069 (2)	0.0536 (18)	0.075 (2)	0.0036 (17)	−0.0033 (19)	0.0058 (17)
C19	0.106 (3)	0.0555 (19)	0.0537 (18)	0.001 (2)	−0.013 (2)	0.0007 (15)
C16	0.096 (3)	0.0555 (19)	0.0534 (17)	−0.0025 (19)	−0.0080 (19)	0.0078 (15)
C3	0.090 (3)	0.069 (2)	0.068 (2)	0.001 (2)	−0.024 (2)	−0.0083 (18)
C14	0.087 (3)	0.065 (2)	0.065 (2)	−0.011 (2)	−0.0043 (19)	−0.0079 (19)
C13	0.143 (4)	0.092 (3)	0.0511 (18)	−0.016 (3)	0.009 (2)	−0.0015 (19)
C20	0.147 (5)	0.094 (3)	0.064 (2)	0.014 (3)	0.006 (3)	−0.020 (2)
C17	0.140 (5)	0.094 (3)	0.104 (3)	0.014 (4)	−0.046 (4)	0.027 (3)
C15	0.064 (2)	0.148 (5)	0.122 (4)	−0.008 (3)	−0.014 (3)	−0.017 (4)
C21	0.158 (5)	0.123 (4)	0.081 (3)	−0.002 (4)	0.032 (3)	−0.016 (3)
C18	0.220 (9)	0.184 (7)	0.164 (6)	0.007 (7)	−0.125 (7)	0.040 (6)
O3	0.095 (2)	0.101 (2)	0.179 (4)	0.024 (2)	−0.037 (3)	−0.040 (3)
N1	0.092 (2)	0.103 (3)	0.099 (3)	0.012 (2)	−0.028 (2)	0.020 (2)
C1	0.070 (2)	0.080 (2)	0.075 (2)	0.004 (2)	−0.008 (2)	0.008 (2)

Geometric parameters (Å, º)

O2—C12	1.327 (4)	C10—C14	1.573 (6)
O2—C13	1.445 (4)	C10—H10	0.9800
O5—C16	1.188 (4)	C4—C3	1.386 (5)
O8—C19	1.335 (5)	C4—H4	0.9300
O8—C20	1.455 (5)	C11—C19	1.523 (5)
N2—C11	1.456 (5)	C11—C16	1.534 (5)
N2—C8	1.484 (4)	C7—H7	0.9300
N2—H1	0.86 (2)	C3—H3	0.9300
O1—C12	1.201 (4)	C14—O3	1.149 (5)
C9—C12	1.493 (4)	C13—H13A	0.9600
C9—C10	1.532 (4)	C13—H13B	0.9600
C9—C8	1.550 (5)	C13—H13C	0.9600
C9—H9	0.9800	C20—C21	1.443 (7)
O6—C16	1.327 (5)	C20—H20A	0.9700
O6—C17	1.454 (5)	C20—H20B	0.9700
O4—C14	1.327 (5)	C17—C18	1.405 (8)
O4—C15	1.471 (5)	C17—H17A	0.9700
O7—C19	1.189 (5)	C17—H17B	0.9700
C5—C6	1.381 (4)	C15—H15A	0.9600
C5—C4	1.391 (5)	C15—H15B	0.9600
C5—C8	1.516 (4)	C15—H15C	0.9600
C2—C7	1.374 (5)	C21—H21A	0.9600
C2—C3	1.396 (5)	C21—H21B	0.9600
C2—C1	1.435 (5)	C21—H21C	0.9600
C8—H8	0.9800	C18—H18A	0.9600
C6—C7	1.380 (5)	C18—H18B	0.9600

C6—H6	0.9300	C18—H18C	0.9600
C10—C11	1.534 (5)	N1—C1	1.143 (5)
C12—O2—C13	117.1 (3)	O7—C19—O8	125.1 (4)
C19—O8—C20	118.7 (3)	O7—C19—C11	125.7 (4)
C11—N2—C8	110.8 (3)	O8—C19—C11	109.1 (3)
C11—N2—H1	111 (3)	O5—C16—O6	125.6 (4)
C8—N2—H1	117 (3)	O5—C16—C11	124.8 (4)
C12—C9—C10	112.6 (3)	O6—C16—C11	109.6 (3)
C12—C9—C8	116.3 (3)	C4—C3—C2	119.4 (4)
C10—C9—C8	105.3 (3)	C4—C3—H3	120.3
C12—C9—H9	107.4	C2—C3—H3	120.3
C10—C9—H9	107.4	O3—C14—O4	124.9 (4)
C8—C9—H9	107.4	O3—C14—C10	125.1 (4)
C16—O6—C17	119.1 (3)	O4—C14—C10	110.0 (4)
C14—O4—C15	111.1 (4)	O2—C13—H13A	109.5
C6—C5—C4	118.4 (3)	O2—C13—H13B	109.5
C6—C5—C8	122.7 (3)	H13A—C13—H13B	109.5
C4—C5—C8	118.8 (3)	O2—C13—H13C	109.5
C7—C2—C3	120.0 (3)	H13A—C13—H13C	109.5
C7—C2—C1	121.1 (3)	H13B—C13—H13C	109.5
C3—C2—C1	118.9 (4)	C21—C20—O8	108.8 (4)
N2—C8—C5	111.2 (3)	C21—C20—H20A	109.9
N2—C8—C9	103.7 (3)	O8—C20—H20A	109.9
C5—C8—C9	113.7 (3)	C21—C20—H20B	109.9
N2—C8—H8	109.4	O8—C20—H20B	109.9
C5—C8—H8	109.4	H20A—C20—H20B	108.3
C9—C8—H8	109.4	C18—C17—O6	108.9 (5)
O1—C12—O2	123.0 (3)	C18—C17—H17A	109.9
O1—C12—C9	125.2 (3)	O6—C17—H17A	109.9
O2—C12—C9	111.8 (3)	C18—C17—H17B	109.9
C7—C6—C5	121.5 (3)	O6—C17—H17B	109.9
C7—C6—H6	119.3	H17A—C17—H17B	108.3
C5—C6—H6	119.3	O4—C15—H15A	109.5
C9—C10—C11	101.9 (3)	O4—C15—H15B	109.5
C9—C10—C14	108.6 (3)	H15A—C15—H15B	109.5
C11—C10—C14	116.3 (3)	O4—C15—H15C	109.5
C9—C10—H10	109.9	H15A—C15—H15C	109.5
C11—C10—H10	109.9	H15B—C15—H15C	109.5
C14—C10—H10	109.9	C20—C21—H21A	109.5
C3—C4—C5	120.8 (3)	C20—C21—H21B	109.5
C3—C4—H4	119.6	H21A—C21—H21B	109.5
C5—C4—H4	119.6	C20—C21—H21C	109.5
N2—C11—C19	106.1 (3)	H21A—C21—H21C	109.5
N2—C11—C10	103.2 (2)	H21B—C21—H21C	109.5
C19—C11—C10	114.6 (3)	C17—C18—H18A	109.5
N2—C11—C16	114.0 (3)	C17—C18—H18B	109.5
C19—C11—C16	108.5 (3)	H18A—C18—H18B	109.5

C10—C11—C16	110.4 (3)	C17—C18—H18C	109.5
C2—C7—C6	119.8 (3)	H18A—C18—H18C	109.5
C2—C7—H7	120.1	H18B—C18—H18C	109.5
C6—C7—H7	120.1	N1—C1—C2	178.8 (5)
C11—N2—C8—C5	−130.9 (3)	C3—C2—C7—C6	1.3 (6)
C11—N2—C8—C9	−8.3 (3)	C1—C2—C7—C6	−178.6 (4)
C6—C5—C8—N2	48.9 (4)	C5—C6—C7—C2	0.6 (6)
C4—C5—C8—N2	−133.6 (3)	C20—O8—C19—O7	−2.4 (7)
C6—C5—C8—C9	−67.7 (4)	C20—O8—C19—C11	−179.8 (4)
C4—C5—C8—C9	109.8 (4)	N2—C11—C19—O7	−106.8 (5)
C12—C9—C8—N2	−142.0 (3)	C10—C11—C19—O7	6.4 (6)
C10—C9—C8—N2	−16.5 (3)	C16—C11—C19—O7	130.3 (5)
C12—C9—C8—C5	−21.1 (4)	N2—C11—C19—O8	70.6 (4)
C10—C9—C8—C5	104.4 (3)	C10—C11—C19—O8	−176.2 (3)
C13—O2—C12—O1	−6.8 (6)	C16—C11—C19—O8	−52.3 (4)
C13—O2—C12—C9	174.4 (4)	C17—O6—C16—O5	−0.3 (7)
C10—C9—C12—O1	−2.6 (6)	C17—O6—C16—C11	−179.8 (4)
C8—C9—C12—O1	119.0 (4)	N2—C11—C16—O5	18.0 (6)
C10—C9—C12—O2	176.1 (3)	C19—C11—C16—O5	136.0 (4)
C8—C9—C12—O2	−62.2 (4)	C10—C11—C16—O5	−97.7 (5)
C4—C5—C6—C7	−1.1 (6)	N2—C11—C16—O6	−162.5 (3)
C8—C5—C6—C7	176.5 (3)	C19—C11—C16—O6	−44.5 (4)
C12—C9—C10—C11	161.4 (3)	C10—C11—C16—O6	81.8 (4)
C8—C9—C10—C11	33.7 (3)	C5—C4—C3—C2	2.3 (6)
C12—C9—C10—C14	−75.3 (4)	C7—C2—C3—C4	−2.7 (6)
C8—C9—C10—C14	157.0 (3)	C1—C2—C3—C4	177.1 (4)
C6—C5—C4—C3	−0.5 (6)	C15—O4—C14—O3	−5.2 (7)
C8—C5—C4—C3	−178.1 (4)	C15—O4—C14—C10	176.6 (4)
C8—N2—C11—C19	150.5 (3)	C9—C10—C14—O3	−22.5 (6)
C8—N2—C11—C10	29.7 (3)	C11—C10—C14—O3	91.7 (5)
C8—N2—C11—C16	−90.1 (3)	C9—C10—C14—O4	155.7 (3)
C9—C10—C11—N2	−38.3 (3)	C11—C10—C14—O4	−90.1 (4)
C14—C10—C11—N2	−156.2 (3)	C19—O8—C20—C21	−155.3 (5)
C9—C10—C11—C19	−153.2 (3)	C16—O6—C17—C18	−169.0 (6)
C14—C10—C11—C19	88.9 (4)	C7—C2—C1—N1	126 (23)
C9—C10—C11—C16	83.9 (3)	C3—C2—C1—N1	−54 (23)
C14—C10—C11—C16	−34.0 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H1···N1^{i}	0.86 (4)	2.47 (4)	3.240 (5)	148 (4)
C8—H8···O1^{ii}	0.98	2.39	3.333 (4)	161 (1)
C13—H13*C*···O7^{ii}	0.96	2.46	3.388 (6)	163
C18—H18*C*···O5^{iii}	0.96	2.55	3.453 (9)	157

Symmetry codes: (i) −*x*+1, *y*−1/2, −*z*+1/2; (ii) −*x*+2, *y*−1/2, −*z*+1/2; (iii) *x*+1/2, −*y*+3/2, −*z*+1.