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Ethyl 9-dicyanomethylene-2,5,7-trinitrofluorene-4-carboxylate

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The title compound, $C_{19}H_9N_5O_8$, has a warped fluorene ring system due to steric repulsion between the 4-ethoxycarbonyl and 5-nitro groups.

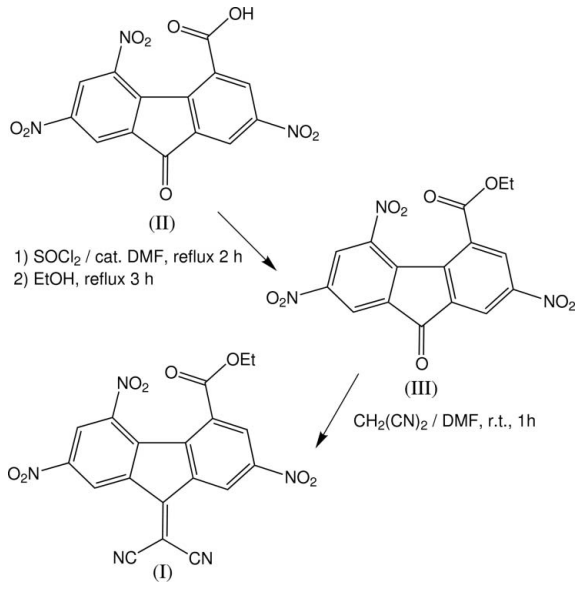
Key indicators

Single-crystal X-ray study
T = 110 K
Mean σ(C–C) = 0.007 Å
R factor = 0.067
wR factor = 0.172
Data-to-parameter ratio = 10.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

The title compound, (I), has been obtained in the course of our studies of electron acceptors of the fluorene series and their charge-transfer complexes and radical ion salts with π-electron donors, *e.g.* tetrathiafulvalene family donors (Perepichka *et al.*, 1998, 2000, 2002; Batsanov *et al.*, 2001, 2002; Kuz'mina *et al.*, 2002).



The molecular structure of (I) is strongly influenced by steric repulsion between the nitro and ethoxycarbonyl groups in positions 4 and 5, respectively. This overcrowding, indicated, for example, by the short (intramolecular) non-bonding distances N4···O5 [2.730 (5) Å] and C17···O4 [2.711 (6) Å], causes the above-mentioned substituents to tilt out of the fluorene plane in opposite directions. Furthermore, the fluorene aromatic system itself loses planarity and adopts a warped (twisted) conformation, the deviations (Å) of its C atoms from the mean plane being: C1 −0.186 (4), C2 −0.149 (4), C3 0.135 (4), C4 0.245 (4), C5 −0.226 (4), C6 −0.145 (4), C7 0.070 (4), C8 0.138 (4), C9 0.033 (4), C10 −0.039 (4), C11 0.060 (5), C12 −0.013 (5) and C13 0.078 (4). Similar distortions are typical for other fluorene derivatives with bulky substituents in positions 4 and 5, *e.g.* 9-dicyanomethylene-2,4,5,7-tetranitrofluorene (Silverman *et al.*, 1974;

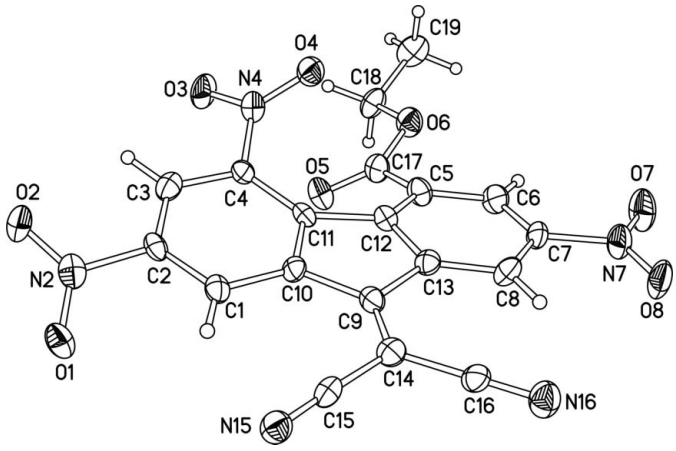


Figure 1
The molecular structure of (I). Atomic displacement ellipsoids are drawn at the 50% probability level.

Batsanov *et al.*, 2001), 9-dicyanomethylene-4,5-dinitrofluorene-2,7-disulfonamide (Batsanov & Perepichka, 2004) or 9-dicyanomethylene-4-bromo-2,5,7-trinitrofluorene (Perepichka *et al.*, 2002).

Experimental

To a suspension of 2,5,7-trinitro-9-oxofluorene-4-carboxylic acid (10.0 g, 27.8 mmol), (II), in thionyl chloride (50 ml) a catalytic amount of *N*,*N*-dimethylformamide (2 drops) was added. The mixture was refluxed for 2 h (full dissolution occurring in 15–20 min) and then evaporated under reduced pressure until dry. Ethanol (50 ml) was added to the residue, the mixture was refluxed for 3 h and left to cool for crystallization. The precipitate was filtered off, washed with cold ethanol (2 × 20 ml) and dried to yield crude compound (III) (9.1 g, 84%; m.p. 426–430 K). This was dissolved in boiling dioxane (25 ml), filtered hot and the filtrate was diluted with hot ethanol (100 ml). On cooling, the solid was filtered off, washed with ethanol and dried. This procedure was repeated to give pure compound (III) (7.2 g, 67%) as yellow crystals (m.p. 434–435 K). ^{1}H NMR (200 MHz, acetone-d$_6$): δ 8.99 (1H, *d*, *J* = 2.0 Hz, H-3), 8.83 (1H, *d*, *J* = 2.0 Hz, H-6), 8.78 (1H, *d*, *J* = 2.2 Hz, H-8), 8.69 (1H, *d*, *J* = 2.2 Hz, H-1), 4.42 (2H, *q*, *J* = 7.2 Hz, CH2), 1.43 (3H, *t*, *J* = 7.2 Hz, CH3). ^{13}C NMR (50 MHz, acetone-d_6): δ 186.21 (C═O), 165.44 (–CO_2–), 150.71, 150.56, 147.36, 144.19, 140.30, 140.08, 139.18, 132.90, 131.05, 126.38, 122.95, 122.00, 63.84 (CH_2), 14.32 (CH_3). MS (EI): *m*/*z* 387 (M^+, 100%). HRMS (EI): m/*z* 387.03447; calculated exact mass: 387.03388. Analysis found: C 49.52, H 2.40, N 10.89%; $C_{16}H_9N_3O_9$ requires: C 49.62, H 2.34, N 10.85%.

Compound (III) (5.0 g, 12.9 mmol) was dissolved in *N*,*N*-dimethylformamide (25 ml), malonitrile (2.2 g, 33.3 mmol) was added to this solution and the mixture was stirred at room temperature for 1 h (the product began to precipitate in 30 min). 2-Propanol (100 ml) was added to the mixture and it was allowed to stand at 273 K for 1–2 h. The solid was filtered off, washed with 2-propanol and dried to yield crude compound (I) (5.2 g, 93%; m.p. 539–543 K). It was dissolved in boiling dioxane (75 ml), hot 2-propanol was added to the solution and the product left to crystallize. The solid was filtered off, washed with 2-propanol and dried. The purification procedure was repeated once more, to afford pure compound (I) (4.8 g, 85%) as bright-yellow crystals (m.p. 543–545 K). ^{1}H NMR (200 MHz, acetone-d_6 + half a drop CF_3CO_2D): δ 9.69 (1H, *d*, *J* = 2.0 Hz, H-8), 9.60 (1H, *d*, *J* = 2.0 Hz, H-1), 9.04 (1H, *d*, *J* = 2.0 Hz, H-6), 8.88 (1H, *d* *J* = 2.0 Hz, H-3), 4.43 (2H, *q*, *J* = 7.2 Hz, CH2), 1.44 (3H, *t*, *J* = 7.2 Hz, CH3). ^{13}C NMR (100 MHz, acetone-d_6 + 0.5 drop CF_3CO_2D): δ 165.30 (–CO_2–), 154.81, 141.14, 140.14, 139.25, 137.49, 133.05, 130.49, 125.76, 124.75, 123.70, 121.52, 117.74, 113.96, 113.37, 113.32, 110.17, 63.96 (CH_2), 14.36 (CH_3). MS (EI): *m*/*z* 435 (M^+, 100%). Analysis found: C 52.52, H 2.03, N 16.15%; $C_{19}H_9N_5O_8$ requires: C 52.42, H 2.08, N 16.09%. Compound (I) was dissolved in hot acetonitrile and left to cool slowly to yield single crystals of X-ray quality.

Crystal data

$C_{19}H_9N_5O_8$	D_x = 1.615 Mg m^{-3}
M_r = 435.31	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 1107 reflections
a = 19.481 (2) Å	θ = 3.1–22.6°
b = 8.620 (1) Å	μ = 0.13 mm^{-1}
c = 10.814 (1) Å	T = 110 (2) K
β = 99.40 (1)°	Prism, yellow
V = 1791.6 (3) Å^3	0.18 × 0.07 × 0.07 mm
Z = 4	

Data collection

Bruker SMART 1 K CCD area-detector diffractometer	1518 reflections with $I > 2\sigma(I)$
	R_{int} = 0.126
ω scans	θ_{max} = 25.0°
Absorption correction: none	$h = -13 \rightarrow 22$
7993 measured reflections	$k = -9 \rightarrow 10$
3079 independent reflections	$l = -12 \rightarrow 12$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0632P)^2 + 0.5406P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.067	
$wR(F^2)$ = 0.172	
S = 1.03	$(\Delta/\sigma)_{max} < 0.001$
3079 reflections	$\Delta\rho_{max}$ = 0.41 e Å^{-3}
292 parameters	$\Delta\rho_{min}$ = −0.37 e Å^{-3}
H-atom parameters constrained	Extinction correction: *SHELXTL*
	Extinction coefficient: 0.018 (2)

Table 1
Selected geometric parameters (Å, °).

N15—C15	1.152 (6)	C10—C11	1.412 (6)
N16—C16	1.147 (6)	C11—C12	1.502 (7)
C9—C14	1.352 (7)	C12—C13	1.397 (7)
C9—C10	1.465 (7)	C14—C16	1.444 (7)
C9—C13	1.495 (6)	C14—C15	1.446 (7)
C2—C1—C10	116.9 (4)	C6—C5—C12	117.7 (5)
C3—C2—C1	123.3 (4)	C7—C6—C5	119.2 (5)
C2—C3—C4	118.0 (5)	C6—C7—C8	124.3 (5)
C3—C4—C11	121.4 (4)	C7—C8—C13	116.0 (5)

The diffraction was rather weak, with a mean $I/\sigma(I)$ ratio of 5.2. The methyl group was refined as a rigid body (C—H = 0.98 Å) rotating around the C—C bond, with a common (refined) U_{iso} value for all three H atoms. Other H atoms were treated as riding in idealized positions, with Csp^3—H = 0.99 Å and Csp^2—H = 0.95 Å, and U_{iso}(H) = 1.3U_{eq}(C) and 1.2U_{eq}(C), respectively.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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supporting information

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$V = 1791.6$ (3) Å^3
$Z = 4$
$F(000) = 888$
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$\theta_{max} = 25.0°$, $\theta_{min} = 2.1°$
$h = -13 \rightarrow 22$
$k = -9 \rightarrow 10$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.067$
$wR(F^2) = 0.172$
$S = 1.03$
3079 reflections
292 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
$w = 1/[\sigma^2(F_o^2) + (0.0632P)^2 + 0.5406P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.41$ e Å^{-3}
$\Delta\rho_{min} = -0.37$ e Å^{-3}
Extinction correction: SHELXTL, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.018 (2)

Special details

Experimental. The data collection nominally covered over a hemisphere of reciprocal space, by a combination of 4 sets of ω scans; each set at different φ and/or 2θ angles and each scan (46.5 sec exposure) covering 0.3° in ω. Crystal to detector distance 6.03 cm.

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.53613 (17)	0.2322 (4)	0.2237 (4)	0.0309 (9)
O2	0.55693 (17)	0.2996 (4)	0.4205 (3)	0.0321 (10)
O3	0.34804 (18)	0.4135 (4)	0.6081 (3)	0.0330 (10)
O4	0.28475 (17)	0.5939 (4)	0.5031 (3)	0.0293 (9)
O5	0.21531 (18)	0.2915 (4)	0.4372 (3)	0.0323 (10)
O6	0.13270 (17)	0.4589 (4)	0.4749 (3)	0.0302 (10)
O7	−0.00294 (18)	0.6632 (5)	0.0855 (4)	0.0407 (11)
O8	0.04476 (17)	0.6932 (5)	−0.0813 (4)	0.0385 (11)
N2	0.5196 (2)	0.2939 (5)	0.3175 (5)	0.0264 (11)
N4	0.3258 (2)	0.4838 (5)	0.5107 (4)	0.0271 (11)
N7	0.0466 (2)	0.6547 (5)	0.0287 (4)	0.0309 (12)
N15	0.4359 (2)	0.4704 (5)	−0.1171 (4)	0.0264 (11)
N16	0.2283 (2)	0.6451 (5)	−0.2394 (4)	0.0317 (12)
C1	0.4162 (2)	0.4011 (6)	0.1842 (5)	0.0216 (12)
H1	0.4389	0.3905	0.1133	0.026*
C2	0.4497 (2)	0.3643 (5)	0.3044 (5)	0.0198 (12)
C3	0.4208 (3)	0.3903 (6)	0.4109 (5)	0.0246 (12)
H3	0.4471	0.3747	0.4920	0.029*
C4	0.3523 (2)	0.4397 (5)	0.3960 (5)	0.0202 (12)
C5	0.1783 (3)	0.4889 (6)	0.2876 (5)	0.0245 (13)
C6	0.1152 (2)	0.5433 (6)	0.2194 (5)	0.0272 (13)
H6	0.0741	0.5440	0.2561	0.033*
C7	0.1136 (2)	0.5954 (6)	0.0985 (5)	0.0225 (12)
C8	0.1697 (2)	0.5900 (6)	0.0349 (5)	0.0252 (13)
H8	0.1657	0.6200	−0.0506	0.030*
C9	0.3006 (2)	0.5089 (6)	0.0615 (5)	0.0206 (12)
C10	0.3476 (2)	0.4543 (5)	0.1728 (4)	0.0190 (12)
C11	0.3132 (2)	0.4608 (5)	0.2780 (4)	0.0191 (12)
C12	0.2382 (3)	0.4980 (6)	0.2302 (5)	0.0237 (12)
C13	0.2325 (2)	0.5381 (6)	0.1038 (5)	0.0204 (12)
C14	0.3158 (2)	0.5339 (6)	−0.0545 (5)	0.0222 (12)
C15	0.3831 (3)	0.4987 (6)	−0.0877 (5)	0.0224 (12)
C16	0.2665 (3)	0.5949 (6)	−0.1573 (5)	0.0235 (12)
C17	0.1782 (2)	0.4036 (6)	0.4085 (5)	0.0261 (13)
C18	0.1288 (3)	0.3746 (6)	0.5921 (4)	0.0294 (14)
H181	0.1753	0.3702	0.6450	0.038*
H182	0.1123	0.2671	0.5734	0.038*

C19	0.0786 (3)	0.4606 (7)	0.6594 (5)	0.0380 (15)
H191	0.0952	0.5670	0.6766	0.049 (10)*
H192	0.0753	0.4081	0.7385	0.049 (10)*
H193	0.0327	0.4629	0.6066	0.049 (10)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.025 (2)	0.034 (2)	0.036 (2)	0.0071 (17)	0.0116 (17)	0.0019 (19)
O2	0.022 (2)	0.039 (3)	0.031 (2)	0.0037 (18)	−0.0046 (18)	0.0040 (18)
O3	0.034 (2)	0.044 (3)	0.019 (2)	0.0073 (19)	−0.0010 (16)	0.0118 (19)
O4	0.023 (2)	0.038 (2)	0.028 (2)	0.0037 (19)	0.0053 (16)	−0.0030 (18)
O5	0.030 (2)	0.028 (2)	0.040 (2)	0.0083 (19)	0.0079 (17)	0.0064 (18)
O6	0.022 (2)	0.038 (2)	0.031 (2)	0.0013 (17)	0.0063 (17)	0.0005 (18)
O7	0.019 (2)	0.058 (3)	0.046 (3)	0.0080 (19)	0.0083 (19)	0.014 (2)
O8	0.023 (2)	0.062 (3)	0.028 (2)	0.0122 (19)	−0.0031 (17)	0.006 (2)
N2	0.022 (2)	0.021 (3)	0.036 (3)	−0.001 (2)	0.004 (2)	0.006 (2)
N4	0.022 (3)	0.030 (3)	0.029 (3)	0.004 (2)	0.001 (2)	0.004 (2)
N7	0.020 (3)	0.039 (3)	0.033 (3)	0.005 (2)	0.001 (2)	0.005 (2)
N15	0.021 (3)	0.033 (3)	0.025 (3)	−0.001 (2)	0.005 (2)	−0.001 (2)
N16	0.025 (3)	0.040 (3)	0.030 (3)	0.003 (2)	0.004 (2)	0.003 (2)
C1	0.019 (3)	0.015 (3)	0.030 (3)	0.000 (2)	0.001 (2)	0.000 (2)
C2	0.019 (3)	0.013 (3)	0.028 (3)	0.003 (2)	0.005 (2)	0.003 (2)
C3	0.025 (3)	0.022 (3)	0.024 (3)	−0.002 (2)	−0.002 (2)	0.003 (2)
C4	0.021 (3)	0.015 (3)	0.023 (3)	0.002 (2)	0.001 (2)	−0.001 (2)
C5	0.019 (3)	0.028 (3)	0.028 (3)	0.003 (2)	0.008 (2)	0.001 (2)
C6	0.015 (3)	0.036 (4)	0.031 (3)	0.000 (2)	0.005 (2)	−0.003 (3)
C7	0.015 (3)	0.029 (3)	0.023 (3)	0.003 (2)	−0.001 (2)	0.000 (2)
C8	0.021 (3)	0.033 (3)	0.020 (3)	−0.004 (2)	−0.001 (2)	−0.001 (2)
C9	0.013 (3)	0.019 (3)	0.030 (3)	−0.002 (2)	0.003 (2)	−0.005 (2)
C10	0.014 (3)	0.019 (3)	0.023 (3)	−0.003 (2)	0.000 (2)	−0.002 (2)
C11	0.018 (3)	0.016 (3)	0.024 (3)	0.004 (2)	0.006 (2)	−0.001 (2)
C12	0.022 (3)	0.023 (3)	0.025 (3)	0.005 (2)	0.001 (2)	−0.002 (2)
C13	0.020 (3)	0.019 (3)	0.022 (3)	−0.002 (2)	0.001 (2)	−0.001 (2)
C14	0.021 (3)	0.022 (3)	0.024 (3)	−0.003 (2)	0.003 (2)	0.000 (2)
C15	0.023 (3)	0.025 (3)	0.018 (3)	−0.002 (2)	−0.002 (2)	0.003 (2)
C16	0.018 (3)	0.029 (3)	0.023 (3)	−0.001 (2)	0.001 (2)	−0.005 (3)
C17	0.012 (3)	0.032 (4)	0.034 (3)	−0.003 (3)	0.004 (2)	−0.002 (3)
C18	0.028 (3)	0.040 (4)	0.020 (3)	−0.004 (3)	0.001 (2)	0.007 (3)
C19	0.036 (3)	0.052 (4)	0.027 (3)	−0.007 (3)	0.010 (3)	0.002 (3)

Geometric parameters (Å, º)

O1—N2	1.234 (5)	C5—C12	1.412 (7)
O2—N2	1.228 (5)	C5—C17	1.500 (7)
O3—N4	1.231 (5)	C6—C7	1.378 (7)
O4—N4	1.235 (5)	C6—H6	0.9500
O5—C17	1.217 (6)	C7—C8	1.384 (6)

O6—C17	1.317 (6)	C8—C13	1.398 (7)
O6—C18	1.474 (6)	C8—H8	0.9500
O7—N7	1.228 (5)	C9—C14	1.352 (7)
O8—N7	1.230 (5)	C9—C10	1.465 (7)
N2—C2	1.476 (6)	C9—C13	1.495 (6)
N4—C4	1.469 (6)	C10—C11	1.412 (6)
N7—C7	1.487 (6)	C11—C12	1.502 (7)
N15—C15	1.152 (6)	C12—C13	1.397 (7)
N16—C16	1.147 (6)	C14—C16	1.444 (7)
C1—C2	1.393 (6)	C14—C15	1.446 (7)
C1—C10	1.401 (6)	C18—C19	1.505 (7)
C1—H1	0.9501	C18—H181	0.9900
C2—C3	1.381 (6)	C18—H182	0.9900
C3—C4	1.385 (6)	C19—H191	0.9800
C3—H3	0.9500	C19—H192	0.9800
C4—C11	1.388 (6)	C19—H193	0.9799
C5—C6	1.407 (7)		

C17—O6—C18	115.1 (4)	C14—C9—C13	126.6 (4)
O2—N2—O1	124.6 (4)	C10—C9—C13	105.6 (4)
O2—N2—C2	118.1 (4)	C1—C10—C11	120.9 (4)
O1—N2—C2	117.3 (4)	C1—C10—C9	129.5 (4)
O3—N4—O4	124.7 (4)	C11—C10—C9	109.6 (4)
O3—N4—C4	118.1 (4)	C4—C11—C10	118.3 (4)
O4—N4—C4	117.1 (4)	C4—C11—C12	134.5 (4)
O7—N7—O8	124.5 (4)	C10—C11—C12	107.1 (4)
O7—N7—C7	117.6 (4)	C13—C12—C5	120.3 (4)
O8—N7—C7	117.9 (4)	C13—C12—C11	107.9 (4)
C2—C1—C10	116.9 (4)	C5—C12—C11	131.6 (5)
C2—C1—H1	121.5	C12—C13—C8	121.6 (4)
C10—C1—H1	121.7	C12—C13—C9	108.9 (4)
C3—C2—C1	123.3 (4)	C8—C13—C9	129.4 (5)
C3—C2—N2	118.8 (4)	C9—C14—C16	123.6 (4)
C1—C2—N2	117.9 (4)	C9—C14—C15	122.7 (4)
C2—C3—C4	118.0 (5)	C16—C14—C15	113.7 (4)
C2—C3—H3	121.0	N15—C15—C14	178.3 (5)
C4—C3—H3	121.0	N16—C16—C14	178.7 (6)
C3—C4—C11	121.4 (4)	O5—C17—O6	125.0 (5)
C3—C4—N4	116.5 (4)	O5—C17—C5	121.7 (5)
C11—C4—N4	121.8 (4)	O6—C17—C5	113.2 (5)
C6—C5—C12	117.7 (5)	O6—C18—C19	107.6 (4)
C6—C5—C17	119.7 (4)	O6—C18—H181	110.2
C12—C5—C17	122.0 (4)	C19—C18—H181	110.1
C7—C6—C5	119.2 (5)	O6—C18—H182	110.3
C7—C6—H6	120.3	C19—C18—H182	110.2
C5—C6—H6	120.4	H181—C18—H182	108.5
C6—C7—C8	124.3 (5)	C18—C19—H191	109.5
C6—C7—N7	118.4 (4)	C18—C19—H192	109.6

C8—C7—N7	117.2 (5)	H191—C19—H192	109.5
C7—C8—C13	116.0 (5)	C18—C19—H193	109.4
C7—C8—H8	122.0	H191—C19—H193	109.5
C13—C8—H8	122.0	H192—C19—H193	109.5
C14—C9—C10	127.7 (4)		

C10—C1—C2—C3	−5.7 (7)	C1—C10—C11—C4	11.1 (7)
C10—C1—C2—N2	174.2 (4)	C9—C10—C11—C4	−169.1 (4)
O2—N2—C2—C3	−19.5 (6)	C1—C10—C11—C12	−171.2 (4)
O1—N2—C2—C3	160.0 (4)	C9—C10—C11—C12	8.6 (5)
O2—N2—C2—C1	160.6 (4)	C6—C5—C12—C13	−9.2 (7)
O1—N2—C2—C1	−19.9 (6)	C17—C5—C12—C13	161.8 (5)
C1—C2—C3—C4	7.2 (7)	C6—C5—C12—C11	175.6 (5)
N2—C2—C3—C4	−172.7 (4)	C17—C5—C12—C11	−13.4 (9)
C2—C3—C4—C11	0.8 (7)	C4—C11—C12—C13	167.2 (5)
C2—C3—C4—N4	−173.0 (4)	C10—C11—C12—C13	−9.9 (5)
O3—N4—C4—C3	−33.3 (6)	C4—C11—C12—C5	−17.1 (10)
O4—N4—C4—C3	143.6 (4)	C10—C11—C12—C5	165.8 (5)
O3—N4—C4—C11	152.9 (5)	C5—C12—C13—C8	8.5 (8)
O4—N4—C4—C11	−30.2 (6)	C11—C12—C13—C8	−175.2 (4)
C12—C5—C6—C7	3.2 (8)	C5—C12—C13—C9	−169.0 (4)
C17—C5—C6—C7	−168.0 (5)	C11—C12—C13—C9	7.2 (5)
C5—C6—C7—C8	4.0 (8)	C7—C8—C13—C12	−1.5 (7)
C5—C6—C7—N7	−179.1 (4)	C7—C8—C13—C9	175.5 (5)
O7—N7—C7—C6	4.4 (7)	C14—C9—C13—C12	−179.7 (5)
O8—N7—C7—C6	−176.2 (5)	C10—C9—C13—C12	−2.0 (5)
O7—N7—C7—C8	−178.4 (5)	C14—C9—C13—C8	3.0 (8)
O8—N7—C7—C8	1.0 (7)	C10—C9—C13—C8	−179.4 (5)
C6—C7—C8—C13	−4.8 (8)	C10—C9—C14—C16	−177.0 (5)
N7—C7—C8—C13	178.2 (4)	C13—C9—C14—C16	0.1 (8)
C2—C1—C10—C11	−3.7 (7)	C10—C9—C14—C15	4.3 (8)
C2—C1—C10—C9	176.5 (5)	C13—C9—C14—C15	−178.5 (5)
C14—C9—C10—C1	−6.9 (9)	C18—O6—C17—O5	0.3 (7)
C13—C9—C10—C1	175.5 (5)	C18—O6—C17—C5	177.6 (4)
C14—C9—C10—C11	173.3 (5)	C6—C5—C17—O5	140.5 (5)
C13—C9—C10—C11	−4.3 (5)	C12—C5—C17—O5	−30.4 (8)
C3—C4—C11—C10	−9.7 (7)	C6—C5—C17—O6	−36.9 (7)
N4—C4—C11—C10	163.8 (4)	C12—C5—C17—O6	152.2 (5)
C3—C4—C11—C12	173.5 (5)	C17—O6—C18—C19	175.8 (4)
N4—C4—C11—C12	−13.0 (8)		