

Diethyl 2-[[2-(trifluoromethyl)anilino]-methylidene]propanedioate

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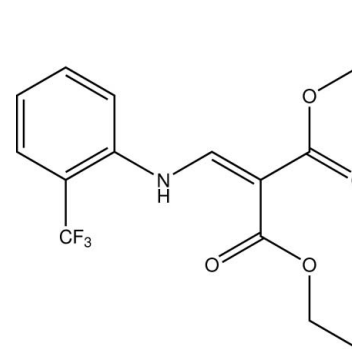
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Key indicators: single-crystal X-ray study; $T = 200$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.044; wR factor = 0.128; data-to-parameter ratio = 18.2.

The title compound, $\text{C}_{15}\text{H}_{16}\text{F}_3\text{NO}_4$, is an *N*-substituted derivative of *ortho*-trifluoromethylaniline featuring a twofold Michael system. The least-squares planes defined by the atoms of the phenyl ring and the atoms of the Michael system enclose an angle of $15.52(5)^\circ$. Apart from classical intramolecular $\text{N}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{F}$ hydrogen bonds, intermolecular $\text{C}-\text{H}\cdots\text{O}$ contacts are observed, the latter connecting the molecules into chains along $[110]$. The shortest intercentroid distance between two aromatic systems is $3.6875(9)$ Å.

Related literature

For the crystal structure of another *ortho*-trifluoromethyl aniline derivative featuring a Michael system as substituent, see: Schweinfurth *et al.* (2011). For general information on Michael systems, see: McMurry (1992). For general pharmaceutical background to derivatives of the title compound, see: Kaur *et al.* (2010); Eswaran *et al.* (2010); Chou *et al.* (2010); Chen *et al.* (2004); Shingalapur *et al.* (2009). For the preparation of the title compound, see: Eswaran *et al.* (2009). For the graph-set analysis of hydrogen bonds, see: Etter *et al.* (1990); Bernstein *et al.* (1995).



Experimental

Crystal data

$\text{C}_{15}\text{H}_{16}\text{F}_3\text{NO}_4$
 $M_r = 331.29$
 Triclinic, $P\bar{1}$
 $a = 7.8080(2)$ Å
 $b = 10.1485(3)$ Å
 $c = 10.5265(3)$ Å
 $\alpha = 95.193(1)^\circ$
 $\beta = 109.183(1)^\circ$

$\gamma = 99.405(1)^\circ$
 $V = 767.84(4)$ Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 0.13$ mm⁻¹
 $T = 200$ K
 $0.55 \times 0.39 \times 0.09$ mm

Data collection

Bruker APEXII CCD diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2008)
 $T_{\min} = 0.946$, $T_{\max} = 1.000$

13616 measured reflections
 3825 independent reflections
 3240 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.014$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$
 $wR(F^2) = 0.128$
 $S = 1.05$
 3825 reflections

210 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.39$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.34$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1}\cdots\text{F2}$	0.88	2.35	2.9330 (15)	124
$\text{N1}-\text{H1}\cdots\text{F3}$	0.88	2.45	2.9242 (15)	114
$\text{N1}-\text{H1}\cdots\text{O3}$	0.88	1.99	2.6399 (14)	130
$\text{C4}-\text{H4}\cdots\text{O1}^i$	0.95	2.60	3.2766 (17)	129

Symmetry code: (i) $x - 1, y - 1, z$.

Data collection: APEX2 (Bruker, 2010); cell refinement: SAINT (Bruker, 2010); data reduction: SAINT; program(s) used to solve structure: SIR97 (Altomare *et al.*, 1999); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 (Farrugia, 1997) and Mercury (Macrae *et al.*, 2008); software used to prepare material for publication: SHELXL97 and PLATON (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BG2441).

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Acta Cryst. (2012). E68, o514–o515 [doi:10.1107/S1600536812002590]

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S1. Comment

Esters of trifluoroaniline are very important intermediates to synthesize 8-fluoroquinoline derivatives, this moiety is of great importance to chemists as well as biologists as it is one of the key building elements for many naturally occurring compounds. Members of this family have a wide range of applications in pharmaceuticals as antimalarial (Kaur *et al.*, 2010), anti-tuberculosis (Eswaran *et al.*, 2010), antitumor (Chou *et al.*, 2010), anticancer (Chen *et al.*, 2004) and antiviral (Shingalapur *et al.*, 2009) agents. In view of the biological importance, we have synthesized the title compound to study its crystal structure.

Resonance between the aromatic system and the *N*-bonded twofold Michael system (an α,β -unsaturated carbonyl compound moiety, see for instance, McMurry, 1992) renders the carbon–nitrogen backbone of the molecule nearly planar, with the least-squares planes defined by the carbon atoms of the phenyl ring on the one hand and the non-hydrogen atoms of the twofold Michael system on the other hand enclosing an angle of only 15.52 (5) ° (Fig. 1). In the crystal, classical intramolecular hydrogen bridges of the N–H \cdots O and N–H \cdots F type can be observed between the secondary amine group and two of the fluorine atoms of the trifluoromethyl group and one of the double-bonded oxygen atoms. In addition, an intramolecular C–H \cdots F contact ($d_{\text{C}\cdots\text{F}}$: 2.6805 (19) Å) involving one of the hydrogen atoms on the aromatic systems is obvious that explains the in-plane conformation of one of the trifluoromethyl group's fluorine atoms with the plane defined by the carbon atoms of the phenyl group. Intermolecular C–H \cdots O contacts whose range falls by more than 0.1 Å below the sum of van-der-Waals radii of the atoms participating in them are present in the crystal structure. In terms of graph-set analysis (Etter *et al.*, 1990; Bernstein *et al.*, 1995), the descriptor for the latter type of contacts is $C^1_1(10)$ on the unitary level. In total, the molecules are connected to infinite chains along [1 1 0]. Information about metrical parameters of these interactions can be found in Table 1. The shortest intercentroid distance between two aromatic systems was measured at 3.6875 (9) Å (Fig. 2).

S2. Experimental

A suspension of 2-(trifluoromethyl) aniline (1.0 g, 0.0062 mol) and diethyl(ethoxymethylene) malonate (4.02 g, 0.0186 mol) was heated to 110 °C for 4 h. The reaction mixture was cooled. The solid product obtained was filtered, washed with pet ether and recrystallized using ethanol. Yield: 1.81 g, 88.29%, m. p. 357–358 K, (Eswaran *et al.*, 2009).

S3. Refinement

Carbon-bound H atoms were placed in calculated positions (C–H 0.95 Å for aromatic and vinylic C atoms, C–H 0.99 Å for methylene groups) and were included in the refinement in the riding model approximation, with $U(\text{H})$ set to $1.2U_{\text{eq}}(\text{C})$. The H atoms of the methyl groups were allowed to rotate with a fixed angle around the C–C bond to best fit the experimental electron density (HFIX 137 in the *SHELX* program suite (Sheldrick, 2008)), with $U(\text{H})$ set to $1.5U_{\text{eq}}(\text{C})$.

The nitrogen-bound H atom was placed in a calculated position (N—H 0.88 Å) and was included in the refinement in the riding model approximation, with $U(\text{H})$ set to $1.2U_{\text{eq}}(\text{N})$

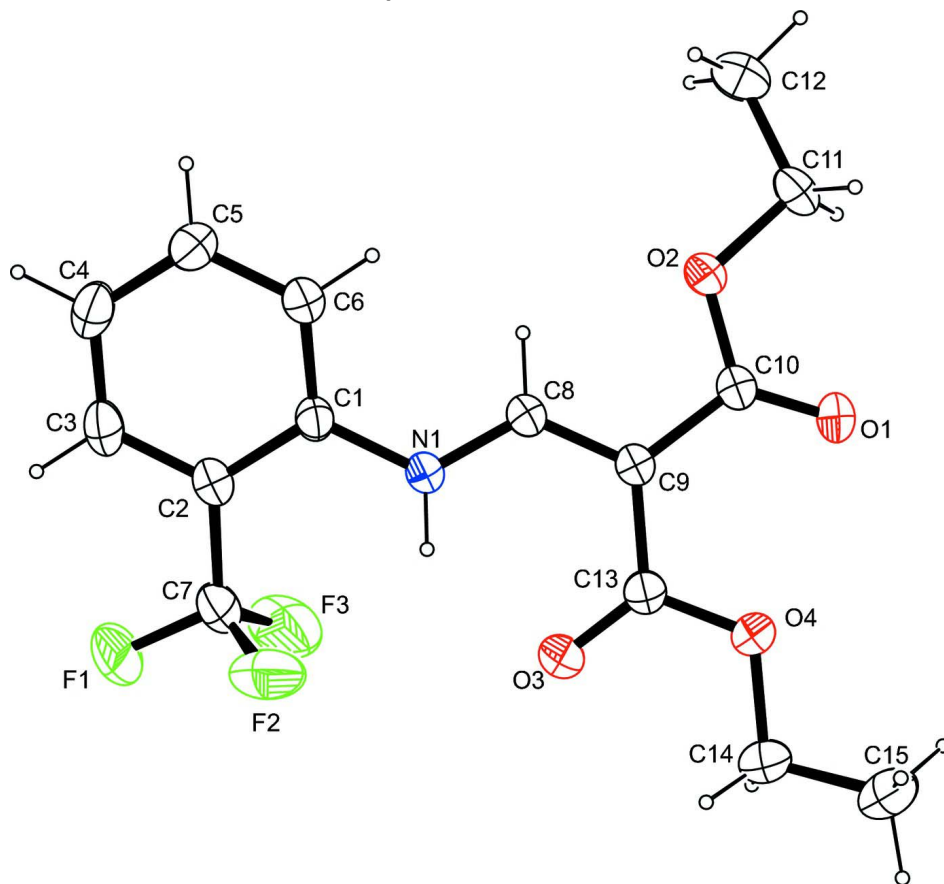
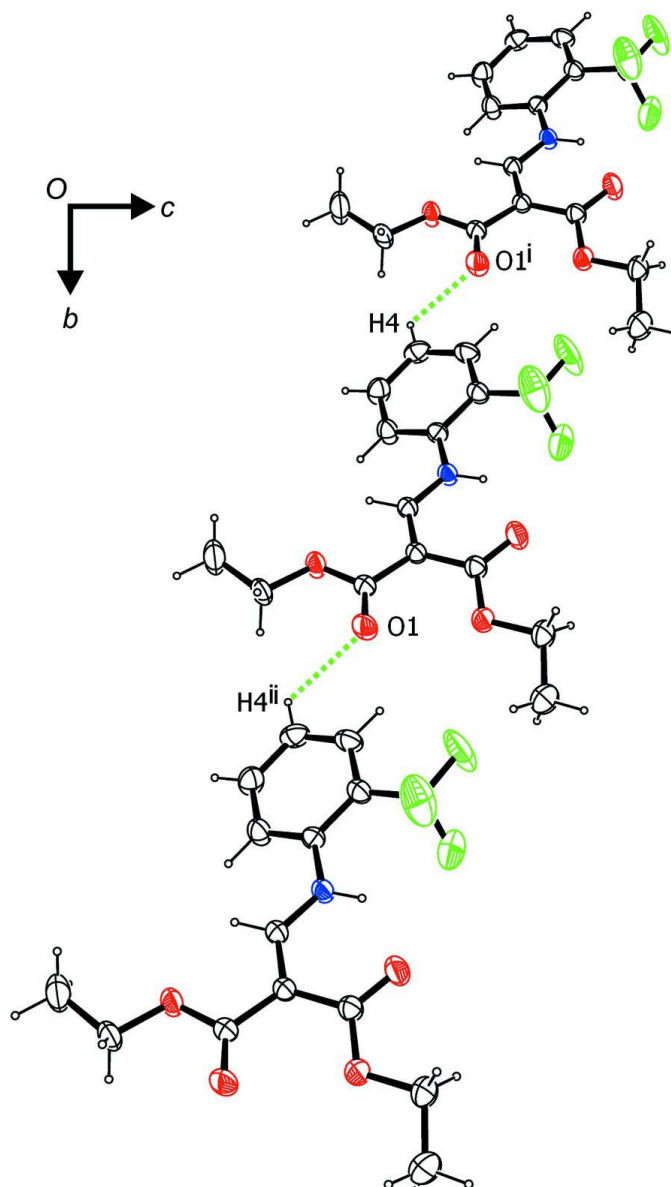


Figure 1

The molecular structure of the title compound, with atom labels and anisotropic displacement ellipsoids (drawn at 50% probability level).

**Figure 2**

Intermolecular C–H···O contacts, viewed along $[-1\ 0\ 0]$. Symmetry operators: ⁱ $x - 1, y - 1, z$; ⁱⁱ $x + 1, y + 1, z$.

Diethyl 2-[[2-(trifluoromethyl)anilino]methylidene]propanedioate

Crystal data

$C_{15}H_{16}F_3NO_4$

$M_r = 331.29$

Triclinic, $P\bar{1}$

Hall symbol: $-P\ 1$

$a = 7.8080\ (2)\ \text{\AA}$

$b = 10.1485\ (3)\ \text{\AA}$

$c = 10.5265\ (3)\ \text{\AA}$

$\alpha = 95.193\ (1)^\circ$

$\beta = 109.183\ (1)^\circ$

$\gamma = 99.405\ (1)^\circ$

$V = 767.84\ (4)\ \text{\AA}^3$

$Z = 2$

$F(000) = 344$

$D_x = 1.433\ \text{Mg m}^{-3}$

Melting point: $357(1)\ \text{K}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 7916 reflections

$\theta = 2.8\text{--}28.3^\circ$

$\mu = 0.13 \text{ mm}^{-1}$
 $T = 200 \text{ K}$

Platelet, colourless
 $0.55 \times 0.39 \times 0.09 \text{ mm}$

Data collection

Bruker APEXII CCD
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 φ and ω scans
 Absorption correction: multi-scan
 (SADABS; Bruker, 2008)
 $T_{\min} = 0.946$, $T_{\max} = 1.000$

13616 measured reflections
 3825 independent reflections
 3240 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.014$
 $\theta_{\max} = 28.4^\circ$, $\theta_{\min} = 2.1^\circ$
 $h = -10 \rightarrow 10$
 $k = -13 \rightarrow 13$
 $l = -14 \rightarrow 14$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.044$
 $wR(F^2) = 0.128$
 $S = 1.05$
 3825 reflections
 210 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
 $w = 1/[\sigma^2(F_o^2) + (0.0646P)^2 + 0.2686P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.39 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.34 \text{ e } \text{Å}^{-3}$

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
F1	0.3636 (2)	-0.04352 (13)	0.35986 (11)	0.0857 (5)
F2	0.3880 (2)	0.16548 (13)	0.34858 (11)	0.0744 (4)
F3	0.60489 (16)	0.07240 (15)	0.33735 (11)	0.0755 (4)
O1	0.86819 (16)	0.61635 (10)	0.03165 (10)	0.0418 (3)
O2	0.68442 (13)	0.44254 (9)	-0.12584 (9)	0.0335 (2)
O3	0.71583 (16)	0.42107 (11)	0.32438 (10)	0.0431 (3)
O4	0.82996 (15)	0.61975 (10)	0.27787 (9)	0.0374 (2)
N1	0.49938 (14)	0.23376 (10)	0.11834 (10)	0.0264 (2)
H1	0.5378	0.2582	0.2071	0.032*
C1	0.36699 (16)	0.11270 (12)	0.06191 (12)	0.0251 (2)
C2	0.32968 (18)	0.02013 (13)	0.14539 (13)	0.0295 (3)
C3	0.20078 (19)	-0.10035 (13)	0.08848 (15)	0.0345 (3)
H3	0.1757	-0.1621	0.1459	0.041*
C4	0.10908 (19)	-0.13115 (13)	-0.05021 (15)	0.0360 (3)
H4	0.0239	-0.2147	-0.0890	0.043*
C5	0.1430 (2)	-0.03872 (15)	-0.13194 (15)	0.0376 (3)
H5	0.0788	-0.0587	-0.2275	0.045*
C6	0.26878 (19)	0.08245 (14)	-0.07735 (13)	0.0335 (3)
H6	0.2882	0.1454	-0.1353	0.040*
C7	0.4211 (2)	0.05075 (16)	0.29631 (15)	0.0439 (4)
C8	0.57087 (16)	0.31427 (12)	0.04683 (12)	0.0247 (2)
H8	0.5348	0.2837	-0.0480	0.030*
C9	0.69146 (16)	0.43685 (12)	0.09707 (12)	0.0246 (2)

C10	0.75905 (16)	0.51016 (12)	0.00254 (12)	0.0263 (2)
C11	0.7483 (2)	0.50110 (15)	-0.22660 (14)	0.0353 (3)
H11A	0.8853	0.5197	-0.1952	0.042*
H11B	0.7065	0.5872	-0.2421	0.042*
C12	0.6691 (3)	0.4020 (2)	-0.35413 (17)	0.0588 (5)
H12A	0.5338	0.3800	-0.3810	0.088*
H12B	0.7180	0.3196	-0.3391	0.088*
H12C	0.7030	0.4411	-0.4265	0.088*
C13	0.74669 (17)	0.48967 (13)	0.24184 (13)	0.0287 (3)
C14	0.8920 (2)	0.67197 (15)	0.42245 (14)	0.0432 (4)
H14A	1.0000	0.6351	0.4733	0.052*
H14B	0.7915	0.6459	0.4588	0.052*
C15	0.9441 (4)	0.82171 (18)	0.43692 (19)	0.0652 (6)
H15A	1.0332	0.8459	0.3913	0.098*
H15B	1.0003	0.8600	0.5337	0.098*
H15C	0.8332	0.8578	0.3953	0.098*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.1160 (11)	0.0754 (8)	0.0410 (6)	-0.0340 (7)	0.0158 (6)	0.0275 (6)
F2	0.1112 (10)	0.0703 (8)	0.0401 (6)	0.0085 (7)	0.0324 (6)	-0.0040 (5)
F3	0.0501 (6)	0.1190 (11)	0.0426 (6)	0.0030 (6)	-0.0001 (5)	0.0263 (6)
O1	0.0542 (6)	0.0302 (5)	0.0357 (5)	-0.0117 (4)	0.0186 (5)	0.0039 (4)
O2	0.0392 (5)	0.0344 (5)	0.0256 (4)	-0.0053 (4)	0.0160 (4)	0.0038 (4)
O3	0.0599 (7)	0.0368 (5)	0.0243 (5)	-0.0072 (5)	0.0115 (4)	0.0062 (4)
O4	0.0508 (6)	0.0286 (5)	0.0254 (5)	-0.0056 (4)	0.0111 (4)	0.0000 (4)
N1	0.0288 (5)	0.0256 (5)	0.0226 (5)	-0.0010 (4)	0.0092 (4)	0.0048 (4)
C1	0.0255 (5)	0.0230 (5)	0.0274 (6)	0.0017 (4)	0.0115 (4)	0.0046 (4)
C2	0.0320 (6)	0.0278 (6)	0.0307 (6)	0.0035 (5)	0.0137 (5)	0.0088 (5)
C3	0.0378 (7)	0.0252 (6)	0.0434 (7)	0.0022 (5)	0.0186 (6)	0.0106 (5)
C4	0.0340 (6)	0.0251 (6)	0.0459 (8)	-0.0017 (5)	0.0156 (6)	-0.0011 (5)
C5	0.0372 (7)	0.0377 (7)	0.0313 (7)	-0.0039 (6)	0.0108 (5)	-0.0015 (5)
C6	0.0350 (6)	0.0341 (7)	0.0278 (6)	-0.0034 (5)	0.0112 (5)	0.0058 (5)
C7	0.0511 (8)	0.0435 (8)	0.0324 (7)	-0.0059 (6)	0.0140 (6)	0.0135 (6)
C8	0.0260 (5)	0.0246 (5)	0.0245 (5)	0.0034 (4)	0.0108 (4)	0.0051 (4)
C9	0.0265 (5)	0.0236 (5)	0.0239 (6)	0.0030 (4)	0.0098 (4)	0.0050 (4)
C10	0.0282 (5)	0.0247 (5)	0.0270 (6)	0.0036 (4)	0.0111 (5)	0.0061 (4)
C11	0.0411 (7)	0.0387 (7)	0.0315 (7)	0.0042 (6)	0.0197 (6)	0.0133 (5)
C12	0.0817 (13)	0.0610 (11)	0.0340 (8)	-0.0048 (9)	0.0308 (9)	0.0038 (7)
C13	0.0301 (6)	0.0277 (6)	0.0254 (6)	0.0014 (5)	0.0080 (5)	0.0046 (5)
C14	0.0556 (9)	0.0387 (8)	0.0251 (6)	-0.0028 (6)	0.0083 (6)	-0.0011 (5)
C15	0.1017 (16)	0.0404 (9)	0.0358 (8)	-0.0061 (9)	0.0137 (9)	-0.0067 (7)

Geometric parameters (Å, °)

F1—C7	1.3179 (16)	C5—C6	1.3832 (18)
F2—C7	1.342 (2)	C5—H5	0.9500

F3—C7	1.329 (2)	C6—H6	0.9500
O1—C10	1.2042 (15)	C8—C9	1.3728 (16)
O2—C10	1.3492 (15)	C8—H8	0.9500
O2—C11	1.4464 (14)	C9—C13	1.4630 (17)
O3—C13	1.2207 (16)	C9—C10	1.4741 (16)
O4—C13	1.3327 (15)	C11—C12	1.484 (2)
O4—C14	1.4551 (16)	C11—H11A	0.9900
N1—C8	1.3357 (14)	C11—H11B	0.9900
N1—C1	1.4071 (15)	C12—H12A	0.9800
N1—H1	0.8800	C12—H12B	0.9800
C1—C6	1.3908 (17)	C12—H12C	0.9800
C1—C2	1.4029 (16)	C14—C15	1.488 (2)
C2—C3	1.3913 (18)	C14—H14A	0.9900
C2—C7	1.4909 (19)	C14—H14B	0.9900
C3—C4	1.378 (2)	C15—H15A	0.9800
C3—H3	0.9500	C15—H15B	0.9800
C4—C5	1.380 (2)	C15—H15C	0.9800
C4—H4	0.9500		
C10—O2—C11	116.56 (10)	C8—C9—C10	118.54 (11)
C13—O4—C14	115.84 (10)	C13—C9—C10	122.74 (10)
C8—N1—C1	124.71 (10)	O1—C10—O2	122.13 (11)
C8—N1—H1	117.6	O1—C10—C9	126.34 (12)
C1—N1—H1	117.6	O2—C10—C9	111.52 (10)
C6—C1—C2	118.40 (11)	O2—C11—C12	107.34 (12)
C6—C1—N1	120.99 (11)	O2—C11—H11A	110.2
C2—C1—N1	120.61 (11)	C12—C11—H11A	110.2
C3—C2—C1	120.22 (12)	O2—C11—H11B	110.2
C3—C2—C7	118.76 (12)	C12—C11—H11B	110.2
C1—C2—C7	120.98 (11)	H11A—C11—H11B	108.5
C4—C3—C2	120.77 (12)	C11—C12—H12A	109.5
C4—C3—H3	119.6	C11—C12—H12B	109.5
C2—C3—H3	119.6	H12A—C12—H12B	109.5
C3—C4—C5	118.95 (12)	C11—C12—H12C	109.5
C3—C4—H4	120.5	H12A—C12—H12C	109.5
C5—C4—H4	120.5	H12B—C12—H12C	109.5
C4—C5—C6	121.24 (13)	O3—C13—O4	121.74 (12)
C4—C5—H5	119.4	O3—C13—C9	122.85 (11)
C6—C5—H5	119.4	O4—C13—C9	115.40 (10)
C5—C6—C1	120.37 (12)	O4—C14—C15	107.04 (12)
C5—C6—H6	119.8	O4—C14—H14A	110.3
C1—C6—H6	119.8	C15—C14—H14A	110.3
F1—C7—F3	108.38 (14)	O4—C14—H14B	110.3
F1—C7—F2	105.40 (15)	C15—C14—H14B	110.3
F3—C7—F2	103.63 (14)	H14A—C14—H14B	108.6
F1—C7—C2	113.24 (13)	C14—C15—H15A	109.5
F3—C7—C2	113.39 (13)	C14—C15—H15B	109.5
F2—C7—C2	112.06 (13)	H15A—C15—H15B	109.5

N1—C8—C9	126.33 (11)	C14—C15—H15C	109.5
N1—C8—H8	116.8	H15A—C15—H15C	109.5
C9—C8—H8	116.8	H15B—C15—H15C	109.5
C8—C9—C13	118.72 (10)		
C8—N1—C1—C6	-14.70 (19)	C1—C2—C7—F2	57.59 (18)
C8—N1—C1—C2	165.95 (11)	C1—N1—C8—C9	175.78 (12)
C6—C1—C2—C3	1.70 (19)	N1—C8—C9—C13	-1.73 (19)
N1—C1—C2—C3	-178.93 (11)	N1—C8—C9—C10	178.62 (11)
C6—C1—C2—C7	-175.94 (13)	C11—O2—C10—O1	2.65 (19)
N1—C1—C2—C7	3.43 (19)	C11—O2—C10—C9	-176.71 (10)
C1—C2—C3—C4	0.5 (2)	C8—C9—C10—O1	-177.98 (13)
C7—C2—C3—C4	178.20 (13)	C13—C9—C10—O1	2.4 (2)
C2—C3—C4—C5	-1.9 (2)	C8—C9—C10—O2	1.34 (16)
C3—C4—C5—C6	1.0 (2)	C13—C9—C10—O2	-178.29 (11)
C4—C5—C6—C1	1.2 (2)	C10—O2—C11—C12	172.35 (13)
C2—C1—C6—C5	-2.6 (2)	C14—O4—C13—O3	3.8 (2)
N1—C1—C6—C5	178.08 (12)	C14—O4—C13—C9	-177.67 (12)
C3—C2—C7—F1	-1.0 (2)	C8—C9—C13—O3	12.48 (19)
C1—C2—C7—F1	176.66 (14)	C10—C9—C13—O3	-167.89 (13)
C3—C2—C7—F3	123.02 (15)	C8—C9—C13—O4	-166.01 (11)
C1—C2—C7—F3	-59.31 (19)	C10—C9—C13—O4	13.62 (18)
C3—C2—C7—F2	-120.09 (15)	C13—O4—C14—C15	-168.58 (15)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1...F2	0.88	2.35	2.9330 (15)	124
N1—H1...F3	0.88	2.45	2.9242 (15)	114
N1—H1...O3	0.88	1.99	2.6399 (14)	130
C3—H3...F1	0.95	2.33	2.6805 (19)	101
C4—H4...O1 ⁱ	0.95	2.60	3.2766 (17)	129

Symmetry code: (i) $x-1, y-1, z$.