

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

3-Fluoro-4-nitrophenyl 4-methylbenzenesulfonate

Wei Ang,^a You-Fu Luo^b and Yong Deng^{a*}

^aKey Laboratory of Drug Targeting and Drug Delivery System of the Education Ministry, Department of Medicinal Chemistry, West China School of Pharmacy, Sichuan University, Chengdu 610041, People's Republic of China, and ^bState Key Laboratory of Biotherapy, West China Hospital, Sichuan University, Chengdu 610041, People's Republic of China

Correspondence e-mail: dengyongy@sohu.com

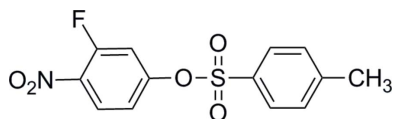
Received 16 December 2010; accepted 17 February 2011

Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.034; wR factor = 0.083; data-to-parameter ratio = 11.8.

In the title compound, $\text{C}_{13}\text{H}_{10}\text{FNO}_5\text{S}$, the dihedral angle between the benzene rings is 47.63 (14)°. In the crystal, π - π stacking occurs between nearly parallel benzene rings of adjacent molecules, the centroid-centroid distance being 3.7806 (16) Å. Weak intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonding is also present in the crystal structure.

Related literature

For related compounds and their biological activity, see: Cho *et al.* (2003); Marson *et al.* (2007).



Experimental

Crystal data

 $\text{C}_{13}\text{H}_{10}\text{FNO}_5\text{S}$ $M_r = 311.28$ Orthorhombic, $Pna2_1$ $a = 14.2596$ (5) Å $b = 11.4800$ (3) Å $c = 8.3602$ (2) Å $V = 1368.57$ (7) Å³ $Z = 4$ Mo $K\alpha$ radiation $\mu = 0.27$ mm⁻¹ $T = 293$ K $0.30 \times 0.30 \times 0.20$ mm

Data collection

Oxford Diffraction Xcalibur Eos diffractometer

Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2006) $T_{\min} = 0.979$, $T_{\max} = 1.0$

10802 measured reflections

2251 independent reflections

1855 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.025$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.034$ $wR(F^2) = 0.083$ $S = 1.05$

2251 reflections

191 parameters

1 restraint

H-atom parameters constrained

 $\Delta\rho_{\max} = 0.12$ e Å⁻³ $\Delta\rho_{\min} = -0.23$ e Å⁻³

Absolute structure: Flack (1983),

752 Friedel pairs

Flack parameter: -0.06 (9)

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C6}-\text{H6}\cdots\text{O2}^i$	0.93	2.55	3.224 (4)	129

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

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2006); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2*.

We thank the Analytical and Testing Center of Sichuan University for the X-ray measurements.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5126).

References

- Cho, C. H., Yun, H. S. & Park, K. Y. (2003). *J. Org. Chem.* **68**, 3017–3025.
 Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
 Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
 Marson, C. M., Mahadevan, T., Dines, J., Sengmany, S., Morrel, J. M., Alao, J. P., Joel, S. P., Vigushin, D. M. & Coombes, R. C. (2007). *Bioorg. Med. Chem. Lett.* **17**, 136–141.
 Oxford Diffraction (2006). *CrysAlis PRO*. Oxford Diffraction Ltd, Abingdon, England.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

supporting information

Acta Cryst. (2011). E67, o750 [doi:10.1107/S1600536811005903]

3-Fluoro-4-nitrophenyl 4-methylbenzenesulfonate

Wei Ang, You-Fu Luo and Yong Deng

S1. Comment

Aryloxyalkanoic acid hydroxyamides are potent inhibitors of histone deacetylase (Marson *et al.*, 2007; Cho *et al.*, 2003). 3-Fluoro-4-nitrophenyl 4-methylbenzenesulfonate is one of the key intermediates to synthesize the aryloxyalkanoic acid hydroxyamides derivatives. We report here its crystal structure. In the title compound (Fig. 1), the dihedral angle between the 3-fluoro-4-nitrophenyl ring and the 4-methylbenzene ring is 47.63 (14)°. In the crystal, intermolecular π - π stacking [centroid-centroid distance = 3.7806 (16) Å] stabilizes the structure (Fig. 2). Weak C—H \cdots O hydrogen bonding is present in the crystal structure (Table 1).

S2. Experimental

To the 3-fluoro-4-nitrophenol (19.10 mmol) in chloroform (20 ml) at 273 K were added pyridine (3.70 ml, 45.84 mmol) dropwise over a period of 20 min and *p*-toluenesulfonyl chloride (22.92 mmol) in small portions. This reaction mixture was stirred at room temperature for 12 h and diluted with dichloromethane and then 10% aqueous HCl. The separated organic layer was washed with 10% aqueous HCl, water and saturated aqueous NaCl; dried over NaSO₄; and concentrated *in vacuo*. The crude 3-fluoro-4-nitrophenyl 4-methylbenzenesulfonate were purified by recrystallization. Crystals suitable for X-ray analysis were obtained by slow evaporation from a solution of ethanol.

S3. Refinement

H atoms were positioned geometrically and refined using a riding model with C—H = 0.93 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for aromatic H atoms and C—H = 0.96 Å and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl H atoms.

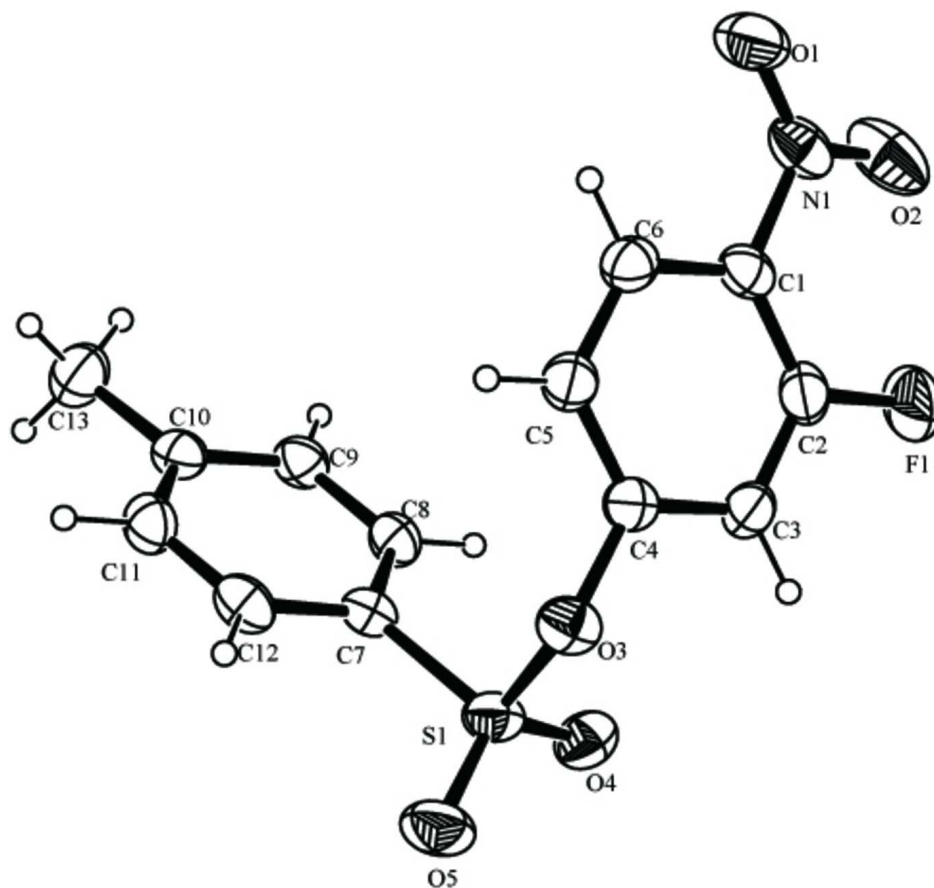
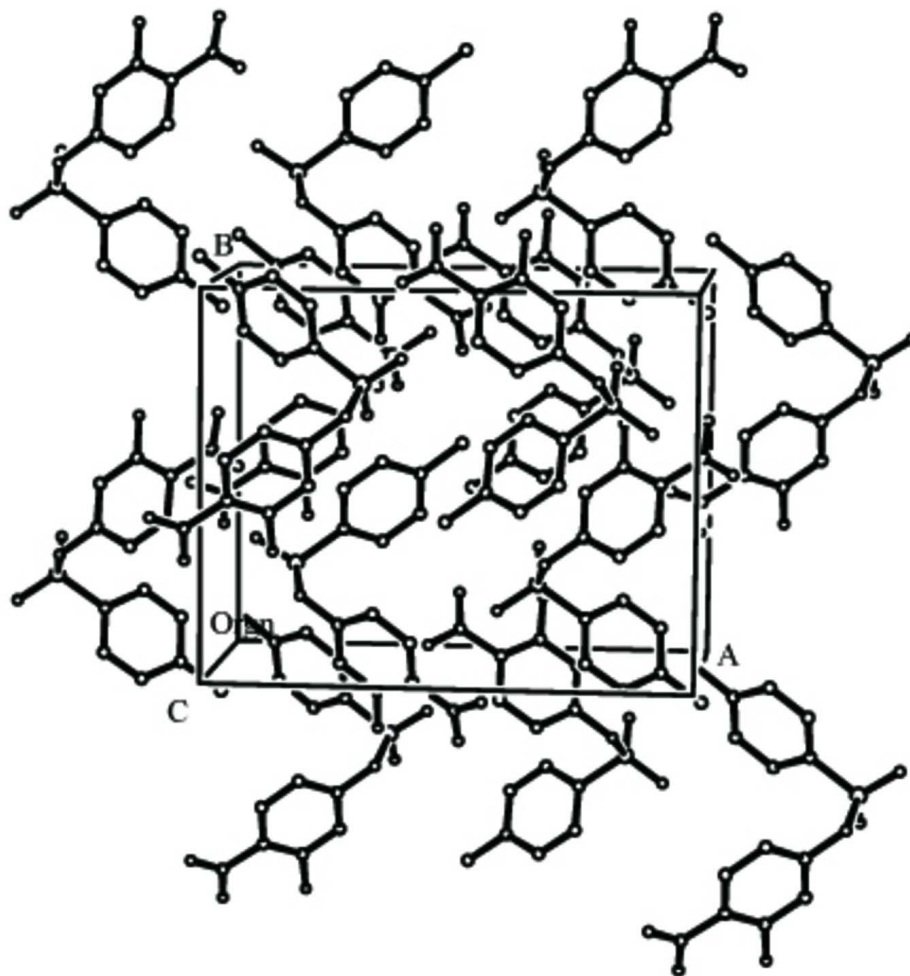


Figure 1

The molecular structure of the title compound, with displacement ellipsoids drawn at the 30% probability level.

**Figure 2**

The cell packing of the title compound.

3-Fluoro-4-nitrophenyl 4-methylbenzenesulfonate

Crystal data

$C_{13}H_{10}FNO_5S$

$M_r = 311.28$

Orthorhombic, $Pna2_1$

Hall symbol: $P\ 2c\ -2n$

$a = 14.2596\ (5)\ \text{\AA}$

$b = 11.4800\ (3)\ \text{\AA}$

$c = 8.3602\ (2)\ \text{\AA}$

$V = 1368.57\ (7)\ \text{\AA}^3$

$Z = 4$

$F(000) = 640$

$D_x = 1.511\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.7107\ \text{\AA}$

Cell parameters from 4234 reflections

$\theta = 2.9\text{--}29.1^\circ$

$\mu = 0.27\ \text{mm}^{-1}$

$T = 293\ \text{K}$

Block, colorless

$0.30 \times 0.30 \times 0.20\ \text{mm}$

Data collection

Oxford Diffraction Xcalibur Eos
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: $16.0874\ \text{pixels mm}^{-1}$

ω scans

Absorption correction: multi-scan

(*CrysAlis PRO*; Oxford Diffraction, 2006)

$T_{\min} = 0.979$, $T_{\max} = 1.0$

10802 measured reflections

2251 independent reflections
 1855 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.025$
 $\theta_{\text{max}} = 26.4^\circ$, $\theta_{\text{min}} = 2.9^\circ$

$h = -17 \rightarrow 17$
 $k = -14 \rightarrow 14$
 $l = -7 \rightarrow 10$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.034$
 $wR(F^2) = 0.083$
 $S = 1.05$
 2251 reflections
 191 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-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.039P)^2 + 0.1799P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.025$
 $\Delta\rho_{\text{max}} = 0.12 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.23 \text{ e } \text{\AA}^{-3}$
 Absolute structure: Flack (1983), 752 Friedel
 pairs
 Absolute structure parameter: -0.06 (9)

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.16636 (4)	0.27076 (5)	0.58998 (10)	0.05567 (19)
F1	0.34171 (15)	-0.14722 (14)	0.6292 (3)	0.1007 (7)
O1	0.56870 (17)	-0.0441 (2)	0.8899 (4)	0.0906 (8)
O2	0.51366 (19)	-0.1649 (2)	0.7187 (3)	0.1041 (9)
O3	0.18775 (13)	0.20401 (17)	0.7566 (2)	0.0574 (5)
O4	0.16341 (14)	0.18606 (19)	0.4665 (3)	0.0675 (6)
O5	0.08578 (12)	0.33777 (18)	0.6260 (3)	0.0788 (7)
N1	0.5080 (2)	-0.0778 (2)	0.7989 (3)	0.0675 (7)
C1	0.4217 (2)	-0.0077 (2)	0.7863 (4)	0.0523 (7)
C2	0.3442 (2)	-0.0442 (2)	0.7026 (4)	0.0601 (8)
C3	0.2660 (2)	0.0248 (2)	0.6906 (4)	0.0580 (7)
H3	0.2136	-0.0003	0.6341	0.070*
C4	0.26682 (18)	0.1317 (2)	0.7636 (3)	0.0474 (6)
C5	0.3427 (2)	0.1698 (2)	0.8491 (4)	0.0548 (7)
H5	0.3417	0.2424	0.8987	0.066*
C6	0.4204 (2)	0.0995 (2)	0.8609 (4)	0.0566 (7)
H6	0.4722	0.1243	0.9192	0.068*
C7	0.26414 (17)	0.3597 (2)	0.5642 (3)	0.0468 (6)
C8	0.33831 (18)	0.3209 (2)	0.4704 (4)	0.0532 (7)

H8	0.3359	0.2483	0.4213	0.064*
C9	0.41530 (18)	0.3917 (2)	0.4512 (4)	0.0538 (7)
H9	0.4659	0.3650	0.3914	0.065*
C10	0.41940 (19)	0.5008 (2)	0.5179 (3)	0.0519 (7)
C11	0.34570 (19)	0.5373 (2)	0.6128 (4)	0.0584 (7)
H11	0.3483	0.6103	0.6609	0.070*
C12	0.26858 (19)	0.4679 (2)	0.6375 (3)	0.0551 (7)
H12	0.2198	0.4931	0.7028	0.066*
C13	0.5028 (2)	0.5786 (3)	0.4867 (5)	0.0752 (9)
H13A	0.4860	0.6370	0.4096	0.113*
H13B	0.5537	0.5327	0.4460	0.113*
H13C	0.5215	0.6155	0.5847	0.113*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0420 (3)	0.0668 (4)	0.0582 (4)	0.0045 (3)	-0.0005 (4)	0.0083 (4)
F1	0.1299 (17)	0.0508 (9)	0.1214 (18)	0.0063 (10)	-0.0321 (16)	-0.0197 (11)
O1	0.0640 (15)	0.0999 (18)	0.108 (2)	0.0104 (14)	-0.0161 (15)	0.0259 (17)
O2	0.135 (2)	0.0934 (17)	0.0836 (18)	0.0580 (16)	-0.0025 (17)	-0.0028 (15)
O3	0.0489 (11)	0.0689 (12)	0.0543 (12)	0.0047 (9)	0.0104 (10)	0.0087 (10)
O4	0.0635 (14)	0.0765 (13)	0.0625 (13)	-0.0099 (10)	-0.0104 (11)	-0.0038 (11)
O5	0.0452 (11)	0.0901 (14)	0.1011 (19)	0.0158 (9)	0.0081 (13)	0.0152 (14)
N1	0.0737 (19)	0.0709 (17)	0.0579 (17)	0.0151 (14)	0.0075 (15)	0.0231 (15)
C1	0.0533 (18)	0.0547 (16)	0.0487 (17)	0.0054 (12)	0.0020 (14)	0.0132 (14)
C2	0.078 (2)	0.0415 (14)	0.0605 (19)	-0.0031 (14)	-0.0059 (17)	0.0043 (14)
C3	0.0585 (17)	0.0529 (15)	0.0627 (19)	-0.0129 (13)	-0.0115 (15)	0.0035 (14)
C4	0.0457 (14)	0.0534 (14)	0.0430 (15)	-0.0017 (11)	0.0040 (13)	0.0085 (12)
C5	0.0580 (17)	0.0520 (15)	0.0543 (17)	-0.0027 (13)	-0.0022 (15)	0.0005 (13)
C6	0.0557 (18)	0.0606 (17)	0.0534 (18)	-0.0076 (14)	-0.0060 (15)	0.0069 (14)
C7	0.0431 (13)	0.0520 (13)	0.0453 (17)	0.0104 (10)	0.0012 (13)	0.0046 (13)
C8	0.0524 (16)	0.0467 (13)	0.0604 (17)	0.0088 (12)	0.0042 (15)	-0.0067 (13)
C9	0.0453 (15)	0.0563 (16)	0.0597 (18)	0.0090 (12)	0.0078 (14)	-0.0005 (14)
C10	0.0506 (16)	0.0555 (16)	0.0497 (17)	0.0051 (12)	-0.0056 (13)	0.0021 (12)
C11	0.0692 (18)	0.0491 (13)	0.0568 (18)	0.0042 (12)	-0.0026 (18)	-0.0081 (14)
C12	0.0601 (17)	0.0582 (15)	0.0471 (17)	0.0176 (12)	0.0093 (14)	-0.0015 (13)
C13	0.069 (2)	0.0714 (19)	0.085 (2)	-0.0119 (15)	0.001 (2)	-0.0069 (18)

Geometric parameters (Å, °)

S1—O3	1.619 (2)	C5—C6	1.374 (4)
S1—O4	1.419 (2)	C6—H6	0.9300
S1—O5	1.4152 (19)	C7—C8	1.390 (4)
S1—C7	1.741 (3)	C7—C12	1.386 (3)
F1—C2	1.332 (3)	C8—H8	0.9300
O1—N1	1.216 (4)	C8—C9	1.375 (3)
O2—N1	1.206 (3)	C9—H9	0.9300
O3—C4	1.401 (3)	C9—C10	1.373 (4)

N1—C1	1.475 (4)	C10—C11	1.382 (4)
C1—C2	1.374 (4)	C10—C13	1.509 (4)
C1—C6	1.380 (4)	C11—H11	0.9300
C2—C3	1.372 (4)	C11—C12	1.374 (4)
C3—H3	0.9300	C12—H12	0.9300
C3—C4	1.371 (4)	C13—H13A	0.9600
C4—C5	1.369 (4)	C13—H13B	0.9600
C5—H5	0.9300	C13—H13C	0.9600
F1—C2—C1	121.8 (3)	C6—C1—N1	117.8 (3)
F1—C2—C3	117.2 (3)	C6—C5—H5	120.4
O1—N1—C1	117.7 (3)	C7—C8—H8	120.5
O2—N1—O1	124.3 (3)	C7—C12—H12	120.3
O2—N1—C1	118.0 (3)	C8—C7—S1	119.45 (19)
O3—S1—C7	103.48 (11)	C8—C9—H9	119.1
O4—S1—O3	107.89 (11)	C9—C8—C7	119.0 (2)
O4—S1—C7	109.59 (13)	C9—C8—H8	120.5
O5—S1—O3	103.14 (14)	C9—C10—C11	118.5 (2)
O5—S1—O4	120.23 (14)	C9—C10—C13	120.2 (3)
O5—S1—C7	110.97 (11)	C10—C9—C8	121.7 (2)
C1—C6—H6	119.9	C10—C9—H9	119.1
C2—C1—N1	122.7 (3)	C10—C11—H11	119.4
C2—C1—C6	119.5 (3)	C10—C13—H13A	109.5
C2—C3—H3	120.7	C10—C13—H13B	109.5
C3—C2—C1	121.0 (3)	C10—C13—H13C	109.5
C3—C4—O3	120.3 (2)	C11—C10—C13	121.3 (3)
C4—O3—S1	117.93 (16)	C11—C12—C7	119.3 (2)
C4—C3—C2	118.6 (3)	C11—C12—H12	120.3
C4—C3—H3	120.7	C12—C7—S1	120.46 (19)
C4—C5—H5	120.4	C12—C7—C8	120.1 (2)
C4—C5—C6	119.1 (3)	C12—C11—C10	121.3 (2)
C5—C4—O3	118.0 (2)	C12—C11—H11	119.4
C5—C4—C3	121.7 (2)	H13A—C13—H13B	109.5
C5—C6—C1	120.2 (3)	H13A—C13—H13C	109.5
C5—C6—H6	119.9	H13B—C13—H13C	109.5

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C6—H6 \cdots O2 ⁱ	0.93	2.55	3.224 (4)	129

Symmetry code: (i) $-x+1, -y, z+1/2$.