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N-[4-(4-Fluorophenyl)-5-hydroxymethyl-6-isopropylpyrimidin-2-yl]-*N*-methylmethanesulfonamide

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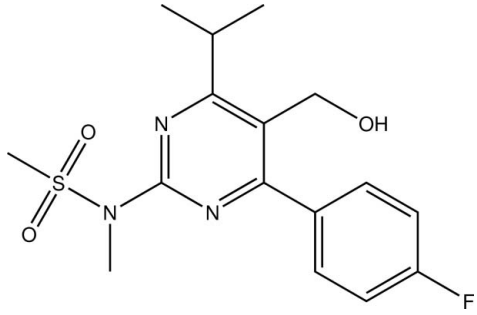
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.057; wR factor = 0.168; data-to-parameter ratio = 14.9.

In the title compound, $C_{16}H_{20}FN_3O_3S$, the pyrimidine and benzene rings are oriented at a dihedral angle of 38.8 (3)°. An intramolecular C—H···O hydrogen bond occurs. The crystal structure is stabilized by O—H···N hydrogen bonds. In addition, C—H···O interactions are also present.

Related literature

For a related structure, see: He *et al.* (2008).



Experimental

Crystal data

$C_{16}H_{20}FN_3O_3S$ M_r = 353.41
Monoclinic, $P2_1/c$
a = 5.8080 (12) Å
b = 11.803 (2) Å
c = 25.867 (5) Å
β = 93.10 (3)°
V = 1770.6 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.21 mm^{-1}
T = 293 K
0.30 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.939, T_{max} = 0.979
3576 measured reflections
3234 independent reflections
2200 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.168
S = 1.00
3234 reflections
217 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···N2^{i}	0.82	2.10	2.896 (3)	165
C5—H5A···O1	0.93	2.53	3.309 (4)	141
C15—H15C···O3^{ii}	0.96	2.39	3.350 (4)	176
C16—H16A···O3^{iii}	0.96	2.55	3.402 (4)	148

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

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); 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: PV2454).

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supporting information

Acta Cryst. (2011). E**67**, o3432 [https://doi.org/10.1107/S1600536811049051]

N-[4-(4-Fluorophenyl)-5-hydroxymethyl-6-isopropylpyrimidin-2-yl]-*N*-methylmethanesulfonamide

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S1. Comment

The derivatives of pyrimidine are important chemical compound. We report here the crystal structure of the title compound.

In the title molecule (Fig. 1), the pyrimidine (N1/N2/C7—C10) and benzene (C1—C6) rings are inclined at a dihedral angle of 38.7 (3)°. The structure is stabilized by intermolecular hydrogen bonding of the type O—H···N. In addition, intramolecular C—H···N and C—H···O and intermolecular C—H···O hydrogen bonding interactions are also present in the crystal structure (Table 1). The crystal structure of a related compound has been reported recently (He *et al.*, 2008).

S2. Experimental

For the preparation of the title compound, sodium salt of *N*-methyl methane sulphonamide (106 g, 631.00 mmol) and 4-(4-fluorophenyl)-6-isopropyl- 2-methyl sulfonylpyrimidine-5-methanol (92 g, 284.0 mmol) were added to dimethylformamide (1000 ml) in a round bottom flask, and then stirred for 1 h at 303 K. After completion of the reaction, demineralized water (1000 ml) was added and stirred for 1 h. The mixture was filtered, washed with water, and then dried (yield; 90%). Crystals of the title compound suitable for X-ray analysis were obtained by slow evaporation of a methanol solution.

S3. Refinement

H atoms were positioned geometrically with O—H = 0.82 Å and C—H = 0.93, 0.96, 0.97 and 0.98 Å for aryl, methyl, methylene and methine H atoms, respectively, and constrained to ride on their parent atoms with U_{iso}(H) = 1.5 times U_{eq}(C-methyl), and 1.2 times U_{eq}(all other C/O atoms).

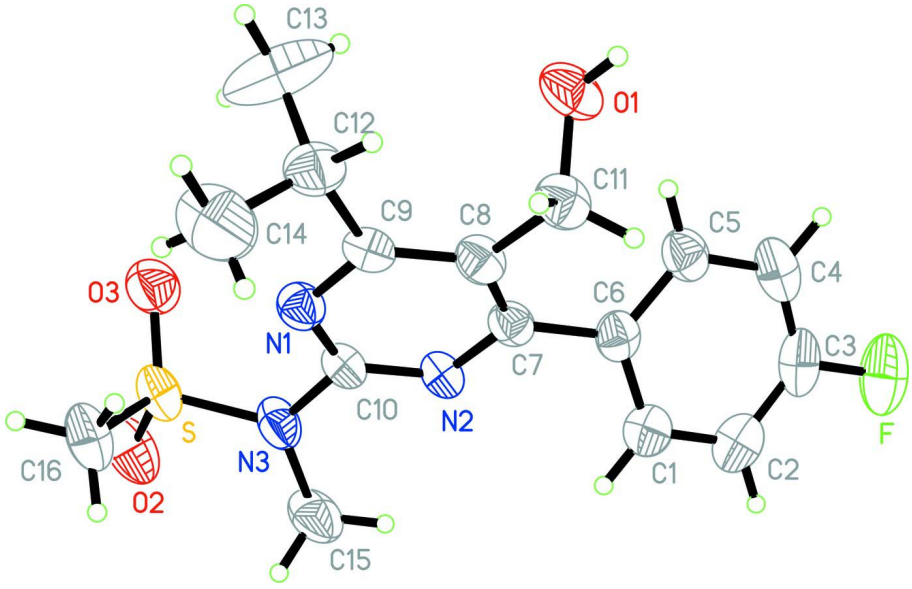

Figure 1

The molecular structure of the title compound showing displacement ellipsoids at the 50% probability level.

_N_-[4-(4-Fluorophenyl)-5-hydroxymethyl-6-isopropylpyrimidin-2-yl]- _N_-methylmethanesulfonamide

Crystal data

$C_{16}H_{20}FN_3O_3S$
$M_r = 353.41$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 5.8080$ (12) Å
$b = 11.803$ (2) Å
$c = 25.867$ (5) Å
$\beta = 93.10$ (3)°
$V = 1770.6$ (6) Å^3
$Z = 4$
$F(000) = 744$
$D_x = 1.326$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9$–13°
$\mu = 0.21$ mm^{-1}
$T = 293$ K
Block, colorless
0.30 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.939$, $T_{max} = 0.979$
3576 measured reflections
3234 independent reflections
2200 reflections with $I > 2\sigma(I)$
$R_{int} = 0.034$
$\theta_{max} = 25.4°$, $\theta_{min} = 1.6°$
$h = 0 \rightarrow 7$
$k = 0 \rightarrow 14$
$l = -31 \rightarrow 31$
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.057$
$wR(F^2) = 0.168$
$S = 1.00$
3234 reflections
217 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

$w = 1/[\sigma^2(F_o^2) + (0.098P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.35$ e Å^{-3}

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S	0.19836 (15)	0.47414 (7)	0.06975 (3)	0.0468 (3)
F	0.3816 (4)	0.31374 (19)	0.44364 (7)	0.0810 (7)
O1	−0.2240 (4)	0.06223 (19)	0.24443 (9)	0.0574 (7)
H1A	−0.2602	0.0043	0.2595	0.086*
N1	0.1043 (4)	0.2659 (2)	0.12672 (9)	0.0390 (6)
C1	0.4508 (6)	0.3093 (3)	0.30694 (13)	0.0491 (8)
H1C	0.5589	0.3309	0.2836	0.059*
O2	0.2945 (5)	0.58313 (19)	0.06090 (9)	0.0665 (7)
N2	0.2636 (4)	0.34440 (19)	0.20528 (9)	0.0358 (6)
C2	0.4998 (7)	0.3252 (3)	0.35873 (14)	0.0572 (9)
H2C	0.6402	0.3558	0.3708	0.069*
N3	0.3087 (5)	0.4355 (2)	0.12759 (9)	0.0454 (7)
O3	−0.0463 (4)	0.4631 (2)	0.06957 (9)	0.0610 (7)
C3	0.3365 (7)	0.2948 (3)	0.39237 (12)	0.0548 (9)
C4	0.1316 (7)	0.2479 (3)	0.37660 (13)	0.0550 (9)
H4A	0.0251	0.2278	0.4006	0.066*
C5	0.0833 (6)	0.2305 (3)	0.32381 (12)	0.0453 (8)
H5A	−0.0562	0.1979	0.3124	0.054*
C6	0.2421 (5)	0.2614 (2)	0.28818 (11)	0.0384 (7)
C7	0.1927 (5)	0.2532 (2)	0.23138 (11)	0.0362 (7)
C8	0.0778 (5)	0.1639 (2)	0.20606 (11)	0.0383 (7)
C9	0.0296 (5)	0.1763 (2)	0.15288 (11)	0.0374 (7)
C10	0.2204 (5)	0.3436 (2)	0.15435 (11)	0.0358 (7)
C11	0.0129 (6)	0.0571 (3)	0.23370 (13)	0.0461 (8)
H11A	0.0408	−0.0083	0.2122	0.055*
H11B	0.1062	0.0496	0.2658	0.055*
C12	−0.1153 (6)	0.0935 (3)	0.12057 (13)	0.0510 (9)
H12A	−0.1274	0.0234	0.1405	0.061*
C13	−0.3530 (8)	0.1421 (5)	0.1122 (2)	0.117 (2)
H13A	−0.4160	0.1570	0.1450	0.176*

H13B	−0.4498	0.0889	0.0932	0.176*
H13C	−0.3455	0.2114	0.0929	0.176*
C14	−0.0079 (9)	0.0648 (4)	0.07052 (16)	0.0964 (17)
H14A	0.1431	0.0337	0.0778	0.145*
H14B	0.0039	0.1322	0.0500	0.145*
H14C	−0.1023	0.0102	0.0518	0.145*
C15	0.5194 (6)	0.4925 (3)	0.14861 (13)	0.0599 (10)
H15A	0.5627	0.4619	0.1821	0.090*
H15B	0.4908	0.5722	0.1516	0.090*
H15C	0.6419	0.4803	0.1258	0.090*
C16	0.3091 (7)	0.3781 (3)	0.02622 (12)	0.0582 (10)
H16A	0.2511	0.3961	−0.0083	0.087*
H16B	0.2629	0.3027	0.0349	0.087*
H16C	0.4744	0.3828	0.0281	0.087*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S	0.0611 (6)	0.0414 (5)	0.0392 (5)	0.0019 (4)	0.0142 (4)	0.0066 (3)
F	0.124 (2)	0.0785 (16)	0.0398 (12)	0.0170 (14)	−0.0057 (12)	−0.0016 (10)
O1	0.0590 (16)	0.0441 (13)	0.0707 (16)	−0.0090 (11)	0.0171 (12)	0.0151 (11)
N1	0.0419 (14)	0.0378 (14)	0.0376 (14)	−0.0066 (12)	0.0051 (11)	−0.0017 (11)
C1	0.048 (2)	0.052 (2)	0.0471 (19)	0.0018 (16)	0.0007 (15)	0.0042 (15)
O2	0.099 (2)	0.0409 (14)	0.0616 (16)	−0.0055 (13)	0.0266 (14)	0.0134 (11)
N2	0.0414 (14)	0.0336 (13)	0.0332 (13)	−0.0017 (11)	0.0096 (10)	0.0011 (10)
C2	0.061 (2)	0.056 (2)	0.054 (2)	0.0067 (18)	−0.0092 (18)	0.0006 (17)
N3	0.0580 (17)	0.0442 (15)	0.0345 (14)	−0.0159 (13)	0.0089 (12)	0.0010 (11)
O3	0.0540 (15)	0.0709 (17)	0.0590 (15)	0.0089 (12)	0.0095 (11)	0.0162 (12)
C3	0.084 (3)	0.0451 (19)	0.0352 (18)	0.0163 (19)	−0.0012 (18)	0.0034 (15)
C4	0.077 (3)	0.047 (2)	0.0422 (19)	0.0133 (19)	0.0180 (18)	0.0092 (15)
C5	0.0510 (19)	0.0401 (17)	0.0455 (18)	0.0035 (15)	0.0077 (15)	0.0080 (14)
C6	0.0438 (17)	0.0320 (15)	0.0394 (17)	0.0104 (14)	0.0034 (13)	0.0044 (12)
C7	0.0348 (16)	0.0353 (16)	0.0392 (16)	0.0049 (13)	0.0079 (12)	0.0038 (13)
C8	0.0417 (17)	0.0310 (15)	0.0428 (17)	0.0009 (13)	0.0079 (13)	0.0027 (13)
C9	0.0343 (16)	0.0345 (16)	0.0442 (17)	−0.0026 (13)	0.0089 (13)	−0.0017 (13)
C10	0.0387 (17)	0.0346 (16)	0.0349 (16)	−0.0016 (13)	0.0104 (12)	0.0001 (12)
C11	0.050 (2)	0.0355 (17)	0.054 (2)	0.0001 (14)	0.0067 (15)	0.0064 (14)
C12	0.051 (2)	0.0440 (19)	0.058 (2)	−0.0140 (16)	0.0015 (16)	−0.0028 (15)
C13	0.055 (3)	0.106 (4)	0.186 (6)	0.000 (3)	−0.040 (3)	−0.042 (4)
C14	0.109 (4)	0.110 (4)	0.073 (3)	−0.052 (3)	0.026 (3)	−0.047 (3)
C15	0.065 (2)	0.062 (2)	0.054 (2)	−0.0297 (19)	0.0150 (17)	−0.0032 (17)
C16	0.080 (3)	0.058 (2)	0.0375 (18)	0.008 (2)	0.0176 (17)	0.0002 (16)

Geometric parameters (Å, º)

S—O2	1.426 (2)	C6—C7	1.485 (4)
S—O3	1.427 (3)	C7—C8	1.391 (4)
S—N3	1.659 (3)	C8—C9	1.396 (4)

S—C16	1.745 (3)	C8—C11	1.507 (4)
F—C3	1.356 (4)	C9—C12	1.511 (4)
O1—C11	1.419 (4)	C11—H11A	0.9700
O1—H1A	0.8200	C11—H11B	0.9700
N1—C10	1.325 (4)	C12—C13	1.500 (6)
N1—C9	1.340 (4)	C12—C14	1.506 (5)
C1—C2	1.367 (4)	C12—H12A	0.9800
C1—C6	1.400 (4)	C13—H13A	0.9600
C1—H1C	0.9300	C13—H13B	0.9600
N2—C10	1.328 (3)	C13—H13C	0.9600
N2—C7	1.347 (4)	C14—H14A	0.9600
C2—C3	1.369 (5)	C14—H14B	0.9600
C2—H2C	0.9300	C14—H14C	0.9600
N3—C10	1.399 (4)	C15—H15A	0.9600
N3—C15	1.474 (4)	C15—H15B	0.9600
C3—C4	1.355 (5)	C15—H15C	0.9600
C4—C5	1.395 (4)	C16—H16A	0.9600
C4—H4A	0.9300	C16—H16B	0.9600
C5—C6	1.388 (4)	C16—H16C	0.9600
C5—H5A	0.9300		

O2—S—O3	118.72 (16)	N1—C10—N2	127.1 (3)
O2—S—N3	104.90 (14)	N1—C10—N3	117.4 (3)
O3—S—N3	108.28 (14)	N2—C10—N3	115.5 (2)
O2—S—C16	108.74 (16)	O1—C11—C8	109.1 (2)
O3—S—C16	109.90 (18)	O1—C11—H11A	109.9
N3—S—C16	105.41 (16)	C8—C11—H11A	109.9
C11—O1—H1A	109.5	O1—C11—H11B	109.9
C10—N1—C9	116.3 (2)	C8—C11—H11B	109.9
C2—C1—C6	121.7 (3)	H11A—C11—H11B	108.3
C2—C1—H1C	119.2	C13—C12—C14	112.5 (4)
C6—C1—H1C	119.2	C13—C12—C9	108.3 (3)
C10—N2—C7	116.5 (2)	C14—C12—C9	112.1 (3)
C1—C2—C3	118.2 (3)	C13—C12—H12A	107.9
C1—C2—H2C	120.9	C14—C12—H12A	107.9
C3—C2—H2C	120.9	C9—C12—H12A	107.9
C10—N3—C15	119.3 (3)	C12—C13—H13A	109.5
C10—N3—S	121.7 (2)	C12—C13—H13B	109.5
C15—N3—S	118.7 (2)	H13A—C13—H13B	109.5
C4—C3—F	118.8 (3)	C12—C13—H13C	109.5
C4—C3—C2	122.9 (3)	H13A—C13—H13C	109.5
F—C3—C2	118.3 (4)	H13B—C13—H13C	109.5
C3—C4—C5	118.8 (3)	C12—C14—H14A	109.5
C3—C4—H4A	120.6	C12—C14—H14B	109.5
C5—C4—H4A	120.6	H14A—C14—H14B	109.5
C6—C5—C4	120.4 (3)	C12—C14—H14C	109.5
C6—C5—H5A	119.8	H14A—C14—H14C	109.5
C4—C5—H5A	119.8	H14B—C14—H14C	109.5

C5—C6—C1	118.1 (3)	N3—C15—H15A	109.5
C5—C6—C7	122.7 (3)	N3—C15—H15B	109.5
C1—C6—C7	119.1 (3)	H15A—C15—H15B	109.5
N2—C7—C8	121.4 (3)	N3—C15—H15C	109.5
N2—C7—C6	113.3 (3)	H15A—C15—H15C	109.5
C8—C7—C6	125.3 (3)	H15B—C15—H15C	109.5
C7—C8—C9	116.8 (3)	S—C16—H16A	109.5
C7—C8—C11	122.4 (3)	S—C16—H16B	109.5
C9—C8—C11	120.8 (3)	H16A—C16—H16B	109.5
N1—C9—C8	121.8 (3)	S—C16—H16C	109.5
N1—C9—C12	114.6 (3)	H16A—C16—H16C	109.5
C8—C9—C12	123.6 (3)	H16B—C16—H16C	109.5

C6—C1—C2—C3	−1.2 (5)	C6—C7—C8—C9	174.4 (3)
O2—S—N3—C10	167.0 (2)	N2—C7—C8—C11	175.2 (3)
O3—S—N3—C10	39.2 (3)	C6—C7—C8—C11	−7.2 (4)
C16—S—N3—C10	−78.3 (3)	C10—N1—C9—C8	−1.9 (4)
O2—S—N3—C15	−19.4 (3)	C10—N1—C9—C12	176.2 (3)
O3—S—N3—C15	−147.1 (3)	C7—C8—C9—N1	4.6 (4)
C16—S—N3—C15	95.3 (3)	C11—C8—C9—N1	−173.8 (3)
C1—C2—C3—C4	1.3 (5)	C7—C8—C9—C12	−173.3 (3)
C1—C2—C3—F	−178.0 (3)	C11—C8—C9—C12	8.3 (4)
F—C3—C4—C5	178.8 (3)	C9—N1—C10—N2	−2.6 (4)
C2—C3—C4—C5	−0.5 (5)	C9—N1—C10—N3	177.3 (2)
C3—C4—C5—C6	−0.4 (5)	C7—N2—C10—N1	4.0 (4)
C4—C5—C6—C1	0.5 (4)	C7—N2—C10—N3	−175.9 (2)
C4—C5—C6—C7	−175.1 (3)	C15—N3—C10—N1	−151.1 (3)
C2—C1—C6—C5	0.3 (5)	S—N3—C10—N1	22.6 (4)
C2—C1—C6—C7	176.1 (3)	C15—N3—C10—N2	28.9 (4)
C10—N2—C7—C8	−0.8 (4)	S—N3—C10—N2	−157.5 (2)
C10—N2—C7—C6	−178.7 (2)	C7—C8—C11—O1	100.5 (3)
C5—C6—C7—N2	136.6 (3)	C9—C8—C11—O1	−81.2 (3)
C1—C6—C7—N2	−39.0 (4)	N1—C9—C12—C13	−77.6 (4)
C5—C6—C7—C8	−41.2 (4)	C8—C9—C12—C13	100.4 (4)
C1—C6—C7—C8	143.3 (3)	N1—C9—C12—C14	47.0 (4)
N2—C7—C8—C9	−3.1 (4)	C8—C9—C12—C14	−134.9 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*A*···N2^{i}	0.82	2.10	2.896 (3)	165
C5—H5*A*···O1	0.93	2.53	3.309 (4)	141
C15—H15*A*···N2	0.96	2.33	2.765 (4)	107
C15—H15*C*···O3^{ii}	0.96	2.39	3.350 (4)	176
C16—H16*A*···O3^{iii}	0.96	2.55	3.402 (4)	148

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