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1-(3,5-Dimethoxybenzyl)-1*H*-pyrrole

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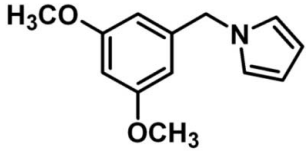
Key indicators: single-crystal X-ray study; T = 153 K; mean σ(C–C) = 0.002 Å; R factor = 0.041; wR factor = 0.140; data-to-parameter ratio = 15.4.

The title compound, $C_{13}H_{15}NO_2$, was synthesized from 3,5-dimethoxybenzaldehyde. The dihedral angle between the pyrrole and benzene rings is 89.91 (5)°. In the crystal, weak C—H···O and C—H···π interactions link the molecules into a three-dimensional network.

Related literature

For the anti-HIV-1 activity of *N*-(arylmethyl)-pyrrole, see: Liu *et al.* (2008); Teixeira *et al.* (2008). For a related structure, see: Wang *et al.* (2011). For the synthesis of 3,5-dimethoxybenzylamine, see: Yraola *et al.* (2006).



Experimental

Crystal data

$C_{13}H_{15}NO_2$
M_r = 217.26
Monoclinic, $P2_1/n$
a = 9.7569 (11) Å
b = 12.2303 (10) Å
c = 10.4181 (10) Å
β = 113.720 (7)°
V = 1138.2 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 153 K
0.21 × 0.21 × 0.16 mm

Data collection

Bruker APEXII CCD diffractometer
7643 measured reflections
2230 independent reflections
1717 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.140
S = 0.99
2230 reflections
145 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

Cg is the centroid of the C6–C11 ring.

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1A···Cg^{i}	0.93	2.79	3.694 (2)	165
C2—H2A···O2^{ii}	0.93	2.72	3.527 (2)	146
C5—H5A···O2^{iii}	0.97	2.68	3.609 (2)	161

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; 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: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RK2345).

References

Bruker (2001). *SAINT-Plus*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2005). *APEX2*. Bruker AXS Inc., Madison, Wisconsin, USA.
Liu, K., Lu, H., Hou, L., Qi, Z., Teixeira, C., Barbault, F., Fan, B. T., Liu, S., Jiang, S. & Xie, L. (2008). *J. Med. Chem.* **51**, 7843–7854.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Teixeira, C., Barbault, F., Rebehmed, J., Liu, K., Xie, L., Lu, H., Jiang, S., Fan, B. T. & Maurel, F. (2008). *Bioorg. Med. Chem.* **16**, 3039–3048.
Wang, C. J., Zeng, X. C. & Xu, S. H. (2011). *Acta Cryst.* E**67**, o2235.
Yraola, F., Garcia-Vicente, S., Fernandez-Recio, J., Albericio, F., Zorzano, A., Marti, L. & Royo, M. (2006). *J. Med. Chem.* **49**, 6197–6208.

supplementary materials

Acta Cryst. (2012). E**68**, o1372 [doi:10.1107/S1600536812015024]

1-(3,5-Dimethoxybenzyl)-1*H*-pyrrole

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Comment

A lot of *N*-(arylmethyl)-pyrrole derivatives show anti-HIV-1 activities, such as inhibitory activity on *gp*41 six-helix bundle formation in both molecule modeling study (Teixeira *et al.*, 2008) and activity assay (Liu *et al.*, 2008). The title compound may possess the same qualities. The title compound is prepared *via* two steps and the product of the first step can be added to the solution of the second step without purification.

In the title compound, as shown in Fig. 1, the pyrrole and benzene rings are on the different plane. The dihedral angle between the two plane is 89.91 (5)° and close to the dihedral angle in 1-benzyl-*N*-methyl-1*H*-pyrrole-2-carboxamide (Wang *et al.*, 2011). The N-C5-C6-C7 torsion angle is 26.37 (20)°. The structure is stabilized by the non-classical hydogen bonds (Table 1). The packing diagram is presented in Fig. 2.

Experimental

Starting material is 3,5-dimethoxy-benzaldehyde (20.9 g, 126 mmol) (Fig. 3). For the first step, 3,5-dimethoxy-benzyl-amine is prepared according to (Yraola *et al.*, 2006). 2,5-Dimethoxytetrahydrofuran (14.2 g, 108 mmol) and glacial acetic acid (150 ml) were added to the first step product. After stirring at 333 K for 6 h, solvent was removed under reduced pressure. The crude product was purified by flash column chromatography (petrol ether / *Et*O*Ac* (10 / 1), yielding the title compound (0.98 g, 52%) as a white solid. The product (16 mg) was dissolved in ethyl ether (1 ml) and methanol (0.05 ml). Single crystals suitable for X-ray diffraction experiment was obtained from the solution by cooling at 273 K for seven days. The molecule was characterized by NMR (Fig. 4).

^{1}H NMR (400 MHz, $CDCl_3$): δ 6.68(t, J = 2.1 Hz, 2H, H-2, H-5), 6.36(t, J = 2.2 Hz, 1H, H-4′), 6.25(d, J = 2.2 Hz, 2H, H-2′, H-6′), 6.18(t, J = 2.1 Hz, 2H, H-3, H-4), 4.99(s, 2H, CH_2), 3.73(s, 6H, OCH_3). ^{13}C NMR (100 MHz, $CDCl_3$): δ 161.2, 140.7, 121.3, 108.6, 105.07, 99.4, 55.3, 53.4. HRMS (ES^+): M/z $[M+Na]^+$ calcd. for $C_{13}H_{15}NO_2Na$: 240.1001; found: 240.1006.

Refinement

All H atoms attached to C atoms were treated as riding, withC–H = 0.96Å for methyl group, C–H = 0.97Å for methylene group, and C–H = 0.93Å for aromatic ring, with U_{iso}(H) = 1.2U_{eq}(C) of the carrier atoms to which they are attached and U_{iso}(H) = 1.5U_{eq}(C) for the methyl groups.

Computing details

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus* (Bruker, 2001); 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: *SHELXTL* (Sheldrick, 2008).

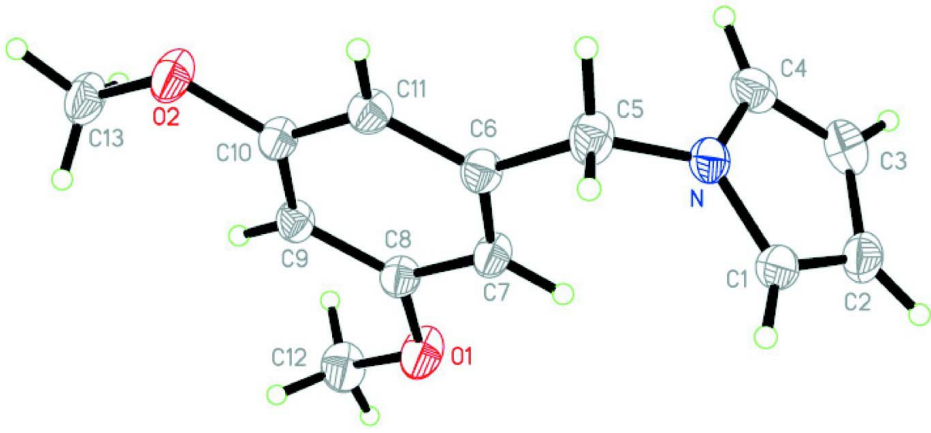

Figure 1

The molecular structure of title compound, showing the atom numbering scheme. Displacement ellipsoids are drawn at 30% probability level. H atoms are presented as a small spheres of arbitrary radius.

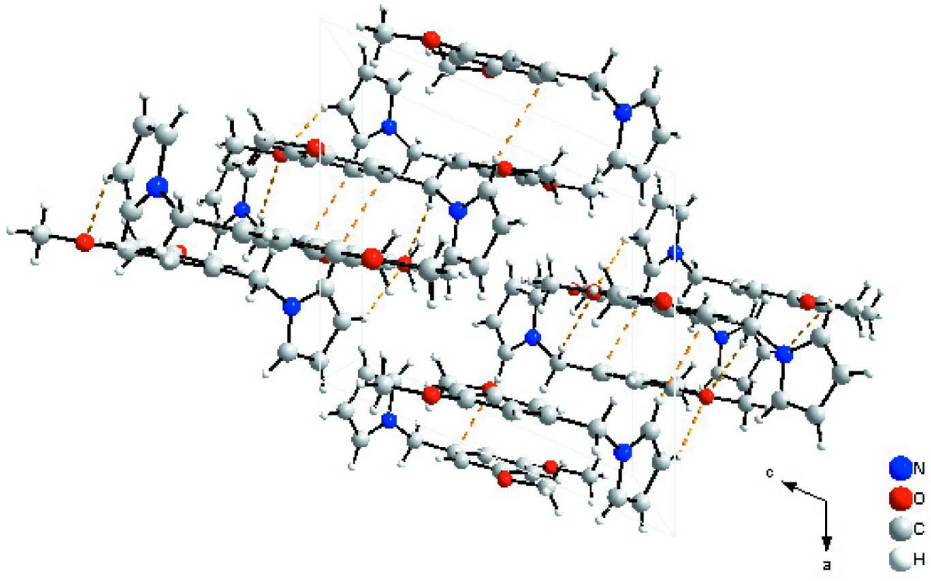

Figure 2

A view of the packing of the title compound along *b* axis.

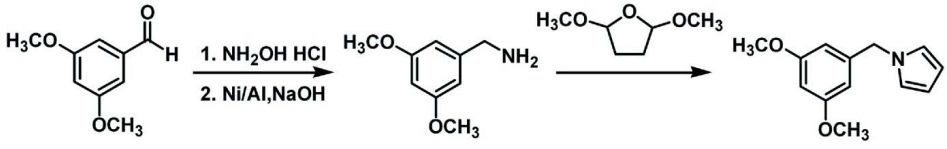

Figure 3

The synthetic route of the title compound.

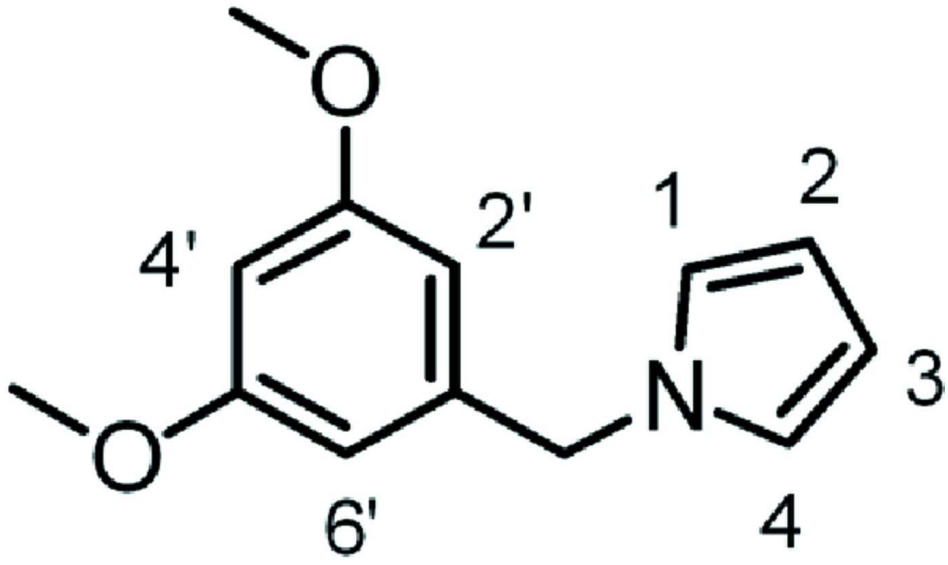

Figure 4

The structure of title compound, with atoms labeling corresponding to the characterization by NMR.

1-(3,5-Dimethoxybenzyl)-1*H*-pyrrole

Crystal data

$C_{13}H_{15}NO_2$
$M_r = 217.26$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 9.7569$ (11) Å
$b = 12.2303$ (10) Å
$c = 10.4181$ (10) Å
$\beta = 113.720$ (7)°
$V = 1138.2$ (2) Å^3
$Z = 4$
$F(000) = 464$
$D_x = 1.268$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2351 reflections
$\theta = 2.8$–26.0°
$\mu = 0.09$ mm^{-1}
$T = 153$ K
Needle, colorless
0.21 × 0.21 × 0.16 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
7643 measured reflections
2230 independent reflections
1717 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 26.0°$, $\theta_{min} = 2.7°$
$h = -12 \rightarrow 8$
$k = -15 \rightarrow 15$
$l = -11 \rightarrow 12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.140$
$S = 0.99$
2230 reflections
145 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

$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.014$

$\Delta\rho_{max} = 0.15$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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}
N	0.14877 (13)	0.21993 (9)	0.87889 (11)	0.0396 (3)
O1	0.13871 (14)	0.44438 (8)	0.47582 (11)	0.0572 (4)
O2	0.08702 (13)	0.07035 (8)	0.30839 (10)	0.0536 (3)
C11	0.13905 (15)	0.11206 (11)	0.54195 (14)	0.0397 (4)
H11A	0.1407	0.0373	0.5591	0.048*
C7	0.16360 (16)	0.29689 (11)	0.62375 (14)	0.0407 (4)
H7A	0.1814	0.3469	0.6959	0.049*
C9	0.10819 (16)	0.26083 (11)	0.37960 (14)	0.0384 (4)
H9A	0.0884	0.2858	0.2895	0.046*
C5	0.19169 (18)	0.14323 (12)	0.79460 (15)	0.0478 (4)
H5A	0.1358	0.0759	0.7850	0.057*
H5B	0.2971	0.1261	0.8437	0.057*
C8	0.13589 (16)	0.33349 (11)	0.48959 (14)	0.0394 (4)
C10	0.11093 (15)	0.14936 (10)	0.40823 (14)	0.0381 (4)
C1	0.24214 (17)	0.29419 (12)	0.96984 (15)	0.0455 (4)
H1A	0.3453	0.2980	0.9977	0.055*
C6	0.16472 (15)	0.18558 (11)	0.65030 (14)	0.0380 (4)
C12	0.0972 (2)	0.48874 (13)	0.33928 (16)	0.0573 (5)
H12A	0.1015	0.5671	0.3448	0.086*
H12B	−0.0028	0.4662	0.2808	0.086*
H12C	0.1650	0.4629	0.3000	0.086*
C4	0.00631 (16)	0.24161 (13)	0.86324 (15)	0.0475 (4)
H4A	−0.0788	0.2035	0.8059	0.057*
C2	0.15797 (19)	0.36182 (13)	1.01276 (16)	0.0514 (4)
H2A	0.1932	0.4197	1.0754	0.062*
C3	0.00937 (18)	0.32851 (14)	0.94572 (16)	0.0528 (4)
H3A	−0.0726	0.3599	0.9556	0.063*
C13	0.0520 (2)	0.10539 (13)	0.16790 (15)	0.0537 (4)
H13A	0.0360	0.0427	0.1082	0.081*
H13B	0.1336	0.1478	0.1651	0.081*
H13C	−0.0370	0.1493	0.1359	0.081*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N	0.0424 (7)	0.0461 (7)	0.0284 (6)	−0.0016 (5)	0.0122 (5)	0.0034 (5)
O1	0.0951 (9)	0.0343 (6)	0.0415 (6)	−0.0005 (5)	0.0266 (6)	0.0034 (4)
O2	0.0836 (8)	0.0395 (6)	0.0364 (6)	−0.0022 (5)	0.0230 (6)	−0.0046 (4)
C11	0.0453 (8)	0.0327 (7)	0.0388 (8)	0.0017 (6)	0.0146 (7)	0.0029 (6)
C7	0.0502 (9)	0.0380 (8)	0.0321 (8)	−0.0007 (6)	0.0146 (6)	−0.0032 (6)
C9	0.0433 (8)	0.0409 (8)	0.0304 (7)	0.0010 (6)	0.0142 (6)	0.0035 (6)
C5	0.0616 (10)	0.0434 (8)	0.0366 (8)	0.0042 (7)	0.0180 (7)	0.0038 (6)
C8	0.0473 (8)	0.0329 (7)	0.0387 (8)	0.0005 (6)	0.0180 (6)	0.0012 (6)
C10	0.0433 (8)	0.0362 (7)	0.0343 (7)	0.0005 (6)	0.0150 (6)	−0.0028 (6)
C1	0.0442 (8)	0.0564 (9)	0.0331 (8)	−0.0090 (7)	0.0126 (6)	0.0010 (7)
C6	0.0401 (8)	0.0393 (8)	0.0326 (7)	0.0021 (6)	0.0126 (6)	0.0025 (6)
C12	0.0857 (12)	0.0398 (8)	0.0516 (10)	0.0030 (8)	0.0332 (9)	0.0121 (7)
C4	0.0390 (8)	0.0630 (10)	0.0367 (8)	−0.0048 (7)	0.0114 (7)	0.0107 (7)
C2	0.0703 (11)	0.0494 (9)	0.0366 (8)	−0.0056 (8)	0.0237 (8)	−0.0016 (7)
C3	0.0556 (10)	0.0625 (10)	0.0472 (9)	0.0140 (8)	0.0278 (8)	0.0130 (8)
C13	0.0745 (11)	0.0525 (9)	0.0329 (8)	−0.0035 (8)	0.0203 (8)	−0.0068 (7)

Geometric parameters (Å, º)

N—C4	1.3589 (19)	C5—C6	1.5097 (19)
N—C1	1.3631 (18)	C5—H5A	0.9700
N—C5	1.4568 (18)	C5—H5B	0.9700
O1—C8	1.3652 (16)	C1—C2	1.362 (2)
O1—C12	1.4207 (17)	C1—H1A	0.9300
O2—C10	1.3693 (16)	C12—H12A	0.9600
O2—C13	1.4284 (17)	C12—H12B	0.9600
C11—C6	1.3849 (19)	C12—H12C	0.9600
C11—C10	1.3845 (19)	C4—C3	1.359 (2)
C11—H11A	0.9300	C4—H4A	0.9300
C7—C6	1.3883 (19)	C2—C3	1.393 (2)
C7—C8	1.3868 (19)	C2—H2A	0.9300
C7—H7A	0.9300	C3—H3A	0.9300
C9—C8	1.3880 (19)	C13—H13A	0.9600
C9—C10	1.3934 (19)	C13—H13B	0.9600
C9—H9A	0.9300	C13—H13C	0.9600
C4—N—C1	108.58 (13)	C2—C1—H1A	126.0
C4—N—C5	125.56 (13)	N—C1—H1A	126.0
C1—N—C5	125.05 (13)	C11—C6—C7	119.33 (13)
C8—O1—C12	118.35 (12)	C11—C6—C5	119.39 (12)
C10—O2—C13	117.65 (11)	C7—C6—C5	121.27 (13)
C6—C11—C10	120.26 (12)	O1—C12—H12A	109.5
C6—C11—H11A	119.9	O1—C12—H12B	109.5
C10—C11—H11A	119.9	H12A—C12—H12B	109.5
C6—C7—C8	120.00 (13)	O1—C12—H12C	109.5
C6—C7—H7A	120.0	H12A—C12—H12C	109.5
C8—C7—H7A	120.0	H12B—C12—H12C	109.5

C8—C9—C10	117.98 (12)	N—C4—C3	108.38 (13)
C8—C9—H9A	121.0	N—C4—H4A	125.8
C10—C9—H9A	121.0	C3—C4—H4A	125.8
N—C5—C6	113.71 (12)	C1—C2—C3	107.53 (15)
N—C5—H5A	108.8	C1—C2—H2A	126.2
C6—C5—H5A	108.8	C3—C2—H2A	126.2
N—C5—H5B	108.8	C4—C3—C2	107.42 (14)
C6—C5—H5B	108.8	C4—C3—H3A	126.3
H5A—C5—H5B	107.7	C2—C3—H3A	126.3
O1—C8—C7	115.00 (13)	O2—C13—H13A	109.5
O1—C8—C9	123.69 (13)	O2—C13—H13B	109.5
C7—C8—C9	121.31 (13)	H13A—C13—H13B	109.5
O2—C10—C11	115.85 (12)	O2—C13—H13C	109.5
O2—C10—C9	123.04 (12)	H13A—C13—H13C	109.5
C11—C10—C9	121.11 (13)	H13B—C13—H13C	109.5
C2—C1—N	108.07 (14)		

Hydrogen-bond geometry (Å, º)

Cg is the centroid of the C6–C11 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*A*···*Cg*^{i}	0.93	2.79	3.6935 (19)	165
C2—H2*A*···O2^{ii}	0.93	2.72	3.527 (2)	146
C5—H5*A*···O2^{iii}	0.97	2.68	3.609 (2)	161

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