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2,5-Bis(3,4-dimethoxyphenyl)-1,3,4-oxadiazole

Tetsuji Moriguchi,* Ryota Kamoto, Venkataprasad Jalli and Akihiko Tsuge

Department of Applied Chemistry, Graduate School of Engineering, Kyushu, Institute of Technology, 1-1 Sensui-cho, Tobata-ku, Kitakyushu 804-8550, Japan. *Correspondence e-mail: moriguch@che.kyutech.ac.jp

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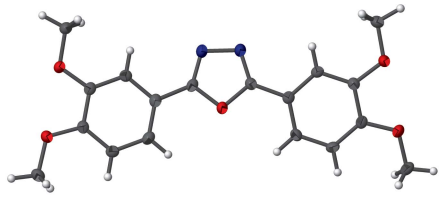
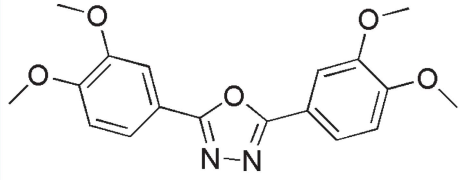
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{18}H_{18}N_2O_5$, which was synthesized by reacting *N*,*N*′-bis-(3,4-dimethoxybenzoyl)hydrazine with $POCl_3$, the dihedral angles between the central oxadiazole ring and pendant 3,4-dimethoxyphenyl rings are 11.37 (7) and 3.09 (7)°. In the crystal, weak C—H···O and π–π [shortest centroid–centroid separation = 3.7370 (11) Å] interactions are present, giving rise to a layered packing motif.

3D view



Chemical scheme



Structure description

1,3,4-Oxadiazole and its analog derivatives are important classes of heterocyclic compounds found in many biological and pharmacological active drug molecules. It was established as privileged class of compound due to its properties in medicinal chemistry (Zou *et al.*, 2002) such as anticonvulsive, antimitotic, anti-emetic, and muscle-relaxant properties. 1,3,4-Oxadiazoles are also used for platelet aggregation inhibition (Fray *et al.*, 1995). Highly functionalized 1,3,4-oxadiazole derivatives are found in drug molecules such as Nesapidil, Furamizole and Raltegravir. As part of our studies in this area, we herein report the crystal structure of the title compound, $C_{18}H_{18}N_2O_5$ (Fig. 1).

In the crystal, weak C—H···O (Table 1; Fig. 2) and π–π [shortest centroid–centroid separation = 3.7370 (11) Å] interactions are present, giving rise to a layered packing motif

Synthesis and crystallization

The title compound was synthesized (Fig. 3) as follows. *N*,*N*′-bis-(3,4-dimethoxybenzoyl)-hydrazine (1 mmol) was taken in a round-bottom flask. To this 10 ml of $POCl_3$ was added and the mixture was refluxed 4 h, quenched with ice, allowed to warm to room temperature, and extracted twice with EtOAc. The organic layers were washed once with 50 ml water, twice with 50 ml of brine solution, dried with Na_2SO_4, and concentrated.

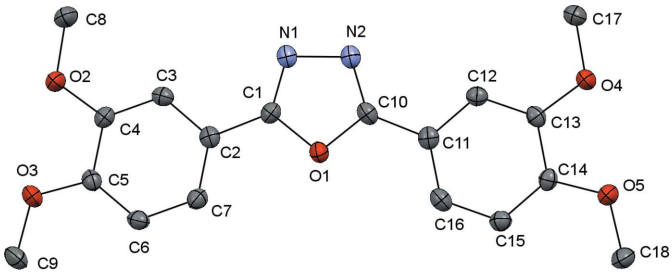


Figure 1
The molecular conformation for the title compound, with displacement ellipsoids drawn at the 50% probability level.

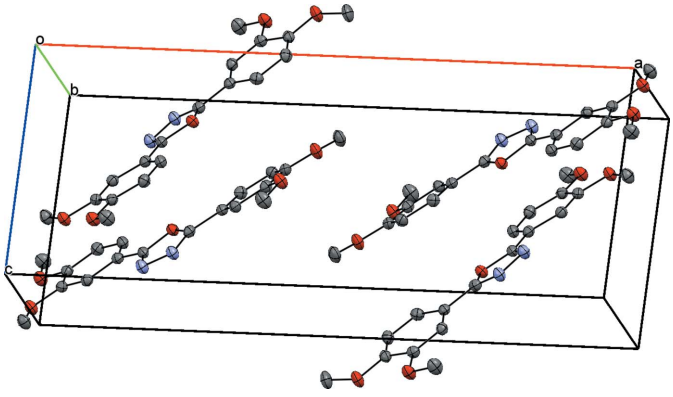


Figure 2
Crystal packing diagram of the title compound, viewed along the *b* axis, with H atoms omitted for clarity.

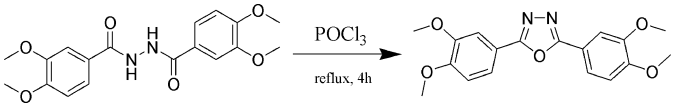


Figure 3
Reaction scheme for the synthesis of the title compound.

Colourless needles of the title compound were obtained by slow evaporation of a benzene solution of crude reaction mixture (m.p. 449–451 K) at room temperature. Analysis: δ_H ^{1}H NMR (DMSO-*d*6, 500 MHz) δ 7.72 (2*H*, *dd*, *J* = 8.3, 2.3 Hz, H-6, H-6′), 7.61 (2*H*, *d*, *J* = 2.3 Hz, H-2, H-2′), 7.18 (2*H*, *d*, *J* = 8.3 Hz, H-5, H-5′), 3.90 (6*H*, *s*, 2 OCH_3), 3.87 (6*H*, *s*, 2 OCH_3); LCMS: MH^+, 342.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C17—H17*B*···O5^{i}	0.96	2.48	3.4185 (18)	165
C18—H18*C*···O4^{ii}	0.96	2.52	3.4682 (18)	170

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{18}H_{20}N_2O_5$
M_r	342.34
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	120
a, *b*, *c* (Å)	22.135 (4), 8.4680 (16), 8.6198 (17)
β (°)	93.287 (2)
V (Å^3)	1613.0 (5)
Z	4
Radiation type	Mo *K*α
μ (mm^{-1})	0.10
Crystal size (mm)	0.50 × 0.20 × 0.20
Data collection	
Diffractometer	Bruker APEXII KY CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2009)
T_{min}, T_{max}	0.883, 0.979
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	15058, 2852, 2525
R_{int}	0.023
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.031, 0.101, 1.11
No. of reflections	2852
No. of parameters	230
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.22, −0.20

Computer programs: *APEX2* (Bruker, 2009), *SAINT* (Bruker, 2009), *SHELXS97* (Sheldrick, 2008), *SHELXL97* (Sheldrick, 2008), *Mercury* (Macrae *et al.*, 2008).

Acknowledgements

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full crystallographic data

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2,5-Bis(3,4-dimethoxyphenyl)-1,3,4-oxadiazole

Crystal data

$C_{18}H_{20}N_2O_5$
M_r = 342.34
Monoclinic, $P2_1/c$
a = 22.135 (4) Å
b = 8.4680 (16) Å
c = 8.6198 (17) Å
β = 93.287 (2)°
V = 1613.0 (5) Å^3
Z = 4
$F(000)$ = 720
D_x = 1.410 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 6712 reflections
θ = 2.6–28.4°
μ = 0.10 mm^{-1}
T = 120 K
Needle, colorless
0.50 × 0.20 × 0.20 mm

Data collection

Bruker APEXII KY CCD diffractometer
Radiation source: fine focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2009)
T_{min} = 0.883, T_{max} = 0.979
15058 measured reflections
2852 independent reflections
2525 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 25.0°, θ_{min} = 1.8°
h = −26→26
k = −10→10
l = −10→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.101
S = 1.11
2852 reflections
230 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
$w = 1/[\sigma^2(F_o^2) + (0.0579P)^2 + 0.3685P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\text{sigma}(F^2)$ 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}
C1	0.27372 (6)	0.16288 (15)	0.21202 (14)	0.0199 (3)
C2	0.32161 (6)	0.13524 (16)	0.10688 (14)	0.0201 (3)
C3	0.33172 (6)	−0.01921 (16)	0.05734 (15)	0.0208 (3)
H3	0.308	−0.1012	0.092	0.025*
C4	0.37645 (6)	−0.05059 (15)	−0.04213 (15)	0.0206 (3)
C5	0.41349 (6)	0.07362 (16)	−0.09198 (15)	0.0214 (3)
C6	0.40284 (6)	0.22591 (16)	−0.04297 (15)	0.0232 (3)
H6	0.4267	0.3083	−0.0764	0.028*
C7	0.35692 (6)	0.25712 (16)	0.05578 (15)	0.0223 (3)
H7	0.3499	0.3601	0.0876	0.027*
C8	0.35611 (7)	−0.32582 (17)	−0.04182 (18)	0.0304 (3)
H8A	0.3632	−0.3325	0.0689	0.046*
H8B	0.3691	−0.4219	−0.0888	0.046*
H8C	0.3137	−0.3104	−0.0668	0.046*
C9	0.50117 (6)	0.14775 (18)	−0.22004 (18)	0.0311 (3)
H9A	0.4817	0.2318	−0.2786	0.047*
H9B	0.532	0.1013	−0.2795	0.047*
H9C	0.5192	0.189	−0.1245	0.047*
C10	0.21076 (6)	0.29230 (16)	0.33921 (14)	0.0208 (3)
C11	0.17893 (6)	0.42755 (16)	0.39717 (15)	0.0214 (3)
C12	0.13070 (6)	0.39841 (16)	0.49227 (14)	0.0207 (3)
H12	0.1203	0.2951	0.5158	0.025*
C13	0.09880 (6)	0.52202 (16)	0.55061 (15)	0.0207 (3)
C14	0.11468 (6)	0.67876 (16)	0.51522 (14)	0.0207 (3)
C15	0.16219 (6)	0.70723 (16)	0.42105 (15)	0.0236 (3)
H15	0.1728	0.8104	0.3973	0.028*
C16	0.19402 (6)	0.58128 (17)	0.36215 (15)	0.0233 (3)
H16	0.2258	0.6007	0.2986	0.028*
C17	0.03218 (6)	0.34858 (16)	0.67204 (16)	0.0260 (3)
H17A	0.0216	0.2969	0.5751	0.039*
H17B	−0.0024	0.3509	0.7342	0.039*
H17C	0.0645	0.2919	0.7263	0.039*
C18	0.09139 (7)	0.95081 (16)	0.53565 (17)	0.0289 (3)
H18A	0.1322	0.9784	0.5683	0.043*
H18B	0.0638	1.0192	0.5855	0.043*
H18C	0.0858	0.9623	0.425	0.043*
N1	0.24338 (5)	0.05939 (14)	0.28535 (13)	0.0241 (3)
N2	0.20193 (5)	0.14444 (14)	0.36880 (13)	0.0248 (3)
O1	0.25592 (4)	0.31369 (11)	0.24030 (10)	0.0211 (2)
O2	0.38929 (4)	−0.19607 (11)	−0.09947 (11)	0.0259 (2)

O3	0.45738 (4)	0.03030 (11)	−0.18649 (11)	0.0280 (2)
O4	0.05134 (4)	0.50644 (11)	0.64300 (10)	0.0239 (2)
O5	0.08007 (4)	0.79125 (11)	0.57747 (11)	0.0248 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0205 (6)	0.0193 (7)	0.0198 (6)	0.0012 (5)	−0.0002 (5)	−0.0013 (5)
C2	0.0194 (6)	0.0228 (7)	0.0181 (6)	0.0007 (5)	0.0000 (5)	0.0007 (5)
C3	0.0199 (6)	0.0211 (7)	0.0213 (7)	−0.0023 (5)	0.0014 (5)	0.0019 (5)
C4	0.0212 (6)	0.0192 (7)	0.0213 (7)	0.0012 (5)	0.0004 (5)	−0.0004 (5)
C5	0.0197 (6)	0.0241 (7)	0.0206 (7)	0.0020 (5)	0.0028 (5)	0.0017 (5)
C6	0.0228 (7)	0.0217 (7)	0.0252 (7)	−0.0036 (5)	0.0035 (5)	0.0041 (5)
C7	0.0255 (7)	0.0185 (7)	0.0231 (7)	0.0020 (5)	0.0027 (5)	−0.0008 (5)
C8	0.0346 (8)	0.0199 (7)	0.0377 (8)	−0.0033 (6)	0.0109 (6)	−0.0030 (6)
C9	0.0230 (7)	0.0332 (8)	0.0380 (8)	−0.0024 (6)	0.0110 (6)	0.0047 (6)
C10	0.0180 (6)	0.0274 (7)	0.0172 (6)	−0.0010 (5)	0.0018 (5)	−0.0006 (5)
C11	0.0193 (6)	0.0257 (7)	0.0190 (6)	0.0010 (5)	−0.0004 (5)	−0.0011 (5)
C12	0.0211 (6)	0.0203 (7)	0.0205 (7)	−0.0014 (5)	0.0002 (5)	−0.0005 (5)
C13	0.0189 (6)	0.0251 (7)	0.0182 (6)	−0.0005 (5)	0.0006 (5)	−0.0007 (5)
C14	0.0202 (6)	0.0221 (7)	0.0198 (6)	0.0011 (5)	−0.0002 (5)	−0.0021 (5)
C15	0.0235 (7)	0.0217 (7)	0.0255 (7)	−0.0022 (5)	0.0017 (5)	0.0011 (5)
C16	0.0199 (6)	0.0289 (8)	0.0214 (7)	−0.0019 (6)	0.0034 (5)	0.0011 (5)
C17	0.0263 (7)	0.0227 (7)	0.0300 (7)	−0.0018 (6)	0.0088 (6)	0.0010 (6)
C18	0.0350 (8)	0.0194 (7)	0.0327 (8)	0.0017 (6)	0.0071 (6)	0.0012 (6)
N1	0.0238 (6)	0.0229 (6)	0.0261 (6)	0.0015 (5)	0.0069 (5)	−0.0007 (5)
N2	0.0233 (6)	0.0244 (6)	0.0275 (6)	0.0014 (5)	0.0076 (5)	−0.0018 (5)
O1	0.0214 (5)	0.0209 (5)	0.0215 (5)	0.0016 (4)	0.0052 (4)	−0.0004 (4)
O2	0.0291 (5)	0.0186 (5)	0.0310 (5)	−0.0006 (4)	0.0116 (4)	−0.0028 (4)
O3	0.0256 (5)	0.0254 (5)	0.0344 (6)	−0.0002 (4)	0.0140 (4)	0.0010 (4)
O4	0.0249 (5)	0.0213 (5)	0.0266 (5)	−0.0006 (4)	0.0099 (4)	−0.0003 (4)
O5	0.0274 (5)	0.0193 (5)	0.0283 (5)	0.0010 (4)	0.0073 (4)	−0.0013 (4)

Geometric parameters (Å, º)

C1—N1	1.2911 (17)	C10—O1	1.3625 (15)
C1—O1	1.3625 (16)	C10—C11	1.4485 (19)
C1—C2	1.4528 (18)	C11—C16	1.382 (2)
C2—C7	1.3818 (18)	C11—C12	1.4048 (18)
C2—C3	1.3978 (19)	C12—C13	1.3740 (19)
C3—C4	1.3723 (18)	C12—H12	0.93
C3—H3	0.93	C13—O4	1.3606 (16)
C4—O2	1.3634 (16)	C13—C14	1.4108 (19)
C4—C5	1.4156 (18)	C14—O5	1.3525 (16)
C5—O3	1.3538 (16)	C14—C15	1.3861 (19)
C5—C6	1.3815 (19)	C15—C16	1.3902 (19)
C6—C7	1.3882 (19)	C15—H15	0.93
C6—H6	0.93	C16—H16	0.93

C7—H7	0.93	C17—O4	1.4289 (16)
C8—O2	1.4266 (17)	C17—H17A	0.96
C8—H8A	0.96	C17—H17B	0.96
C8—H8B	0.96	C17—H17C	0.96
C8—H8C	0.96	C18—O5	1.4243 (16)
C9—O3	1.4298 (17)	C18—H18A	0.96
C9—H9A	0.96	C18—H18B	0.96
C9—H9B	0.96	C18—H18C	0.96
C9—H9C	0.96	N1—N2	1.3978 (15)
C10—N2	1.2949 (18)		

N1—C1—O1	112.63 (11)	C16—C11—C10	122.77 (12)
N1—C1—C2	127.93 (12)	C12—C11—C10	117.60 (12)
O1—C1—C2	119.44 (11)	C13—C12—C11	120.23 (12)
C7—C2—C3	119.88 (12)	C13—C12—H12	119.9
C7—C2—C1	121.65 (12)	C11—C12—H12	119.9
C3—C2—C1	118.47 (12)	O4—C13—C12	124.79 (12)
C4—C3—C2	120.39 (12)	O4—C13—C14	115.34 (11)
C4—C3—H3	119.8	C12—C13—C14	119.86 (12)
C2—C3—H3	119.8	O5—C14—C15	125.16 (12)
O2—C4—C3	125.06 (12)	O5—C14—C13	115.02 (11)
O2—C4—C5	115.15 (11)	C15—C14—C13	119.81 (12)
C3—C4—C5	119.80 (12)	C14—C15—C16	119.86 (12)
O3—C5—C6	125.30 (12)	C14—C15—H15	120.1
O3—C5—C4	115.44 (12)	C16—C15—H15	120.1
C6—C5—C4	119.26 (12)	C11—C16—C15	120.61 (12)
C5—C6—C7	120.60 (12)	C11—C16—H16	119.7
C5—C6—H6	119.7	C15—C16—H16	119.7
C7—C6—H6	119.7	O4—C17—H17A	109.5
C2—C7—C6	120.04 (12)	O4—C17—H17B	109.5
C2—C7—H7	120.0	H17A—C17—H17B	109.5
C6—C7—H7	120.0	O4—C17—H17C	109.5
O2—C8—H8A	109.5	H17A—C17—H17C	109.5
O2—C8—H8B	109.5	H17B—C17—H17C	109.5
H8A—C8—H8B	109.5	O5—C18—H18A	109.5
O2—C8—H8C	109.5	O5—C18—H18B	109.5
H8A—C8—H8C	109.5	H18A—C18—H18B	109.5
H8B—C8—H8C	109.5	O5—C18—H18C	109.5
O3—C9—H9A	109.5	H18A—C18—H18C	109.5
O3—C9—H9B	109.5	H18B—C18—H18C	109.5
H9A—C9—H9B	109.5	C1—N1—N2	106.10 (11)
O3—C9—H9C	109.5	C10—N2—N1	106.61 (10)
H9A—C9—H9C	109.5	C1—O1—C10	102.56 (10)
H9B—C9—H9C	109.5	C4—O2—C8	116.58 (10)
N2—C10—O1	112.10 (11)	C5—O3—C9	116.83 (11)
N2—C10—C11	127.93 (12)	C13—O4—C17	116.11 (10)
O1—C10—C11	119.96 (11)	C14—O5—C18	117.12 (10)
C16—C11—C12	119.63 (12)		

N1—C1—C2—C7	−169.37 (13)	C12—C13—C14—O5	179.47 (11)
O1—C1—C2—C7	11.53 (19)	O4—C13—C14—C15	−179.58 (11)
N1—C1—C2—C3	10.6 (2)	C12—C13—C14—C15	0.35 (19)
O1—C1—C2—C3	−168.55 (11)	O5—C14—C15—C16	−179.14 (12)
C7—C2—C3—C4	−0.16 (19)	C13—C14—C15—C16	−0.11 (19)
C1—C2—C3—C4	179.92 (11)	C12—C11—C16—C15	0.5 (2)
C2—C3—C4—O2	−178.77 (12)	C10—C11—C16—C15	−179.81 (12)
C2—C3—C4—C5	1.49 (19)	C14—C15—C16—C11	−0.3 (2)
O2—C4—C5—O3	−1.52 (17)	O1—C1—N1—N2	0.27 (14)
C3—C4—C5—O3	178.25 (11)	C2—C1—N1—N2	−178.88 (12)
O2—C4—C5—C6	178.38 (11)	O1—C10—N2—N1	−0.03 (14)
C3—C4—C5—C6	−1.85 (19)	C11—C10—N2—N1	179.64 (12)
O3—C5—C6—C7	−179.23 (12)	C1—N1—N2—C10	−0.14 (14)
C4—C5—C6—C7	0.9 (2)	N1—C1—O1—C10	−0.28 (14)
C3—C2—C7—C6	−0.83 (19)	C2—C1—O1—C10	178.95 (11)
C1—C2—C7—C6	179.10 (12)	N2—C10—O1—C1	0.18 (14)
C5—C6—C7—C2	0.4 (2)	C11—C10—O1—C1	−179.52 (11)
N2—C10—C11—C16	177.36 (13)	C3—C4—O2—C8	−4.66 (19)
O1—C10—C11—C16	−3.00 (19)	C5—C4—O2—C8	175.09 (12)
N2—C10—C11—C12	−2.9 (2)	C6—C5—O3—C9	9.25 (19)
O1—C10—C11—C12	176.74 (11)	C4—C5—O3—C9	−170.85 (12)
C16—C11—C12—C13	−0.21 (19)	C12—C13—O4—C17	−3.73 (18)
C10—C11—C12—C13	−179.96 (11)	C14—C13—O4—C17	176.20 (11)
C11—C12—C13—O4	179.74 (11)	C15—C14—O5—C18	3.64 (19)
C11—C12—C13—C14	−0.19 (19)	C13—C14—O5—C18	−175.43 (11)
O4—C13—C14—O5	−0.46 (17)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C17—H17*B*···O5^{i}	0.96	2.48	3.4185 (18)	165
C18—H18*C*···O4^{ii}	0.96	2.52	3.4682 (18)	170

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