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Methyl 3,4-bis(cyclopropylmethoxy)-benzoate

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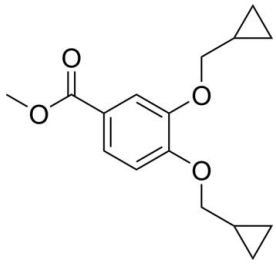
Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.002 Å; R factor = 0.041; wR factor = 0.114; data-to-parameter ratio = 21.2.

The title compound, $C_{16}H_{20}O_4$, was obtained unintentionally as the byproduct of an attempted synthesis of methyl 3-(cyclopropylmethoxy)-4-hydroxybenzoate. In the crystal, the molecules are linked by intermolecular C—H···O interactions.

Related literature

For the preparation, see: Bose *et al.* (2005). For a similar structure, see: Hou *et al.* (2010).



Experimental

Crystal data

$C_{16}H_{20}O_4$ M_r = 276.33
Orthorhombic, $P2_12_12_1$
a = 4.9018 (8) Å
b = 15.543 (2) Å
c = 18.846 (2) Å
V = 1435.9 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 113 K
0.22 × 0.20 × 0.18 mm

Data collection

Rigaku Saturn724 CCD diffractometer
Absorption correction: multi-scan (*REQAB*; Jacobson, 1998)
T_{min} = 0.891, T_{max} = 0.984
20068 measured reflections
3852 independent reflections
3300 reflections with $F^2 > 2.0\sigma(F^2)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.114
S = 1.06
3852 reflections
182 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.57 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C12—H12B···O3^{i}	0.99	2.55	3.4073 (18)	145

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

Data collection: *CrystalClear-SM Expert* (Rigaku, 2009); cell refinement: *CrystalClear-SM Expert*; data reduction: *CrystalClear-SM Expert*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *CrystalStructure* (Rigaku, 2009); software used to prepare material for publication: *CrystalStructure*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JH2279).

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supplementary materials

Acta Cryst. (2011). E**67**, o1130 [**doi:10.1107/S1600536811013158**]

Methyl 3,4-bis(cyclopropylmethoxy)benzoate

X.-C. Cheng, J.-J. Hou, C.-Z. Xie, R.-L. Wang and W.-R. Xu

Comment

Roflumilast is an effective phosphodiesterase-4 inhibitor (PDE4 inhibitor), which can be used in the treatment of asthma, inflammation, bronchitis, allergy and other disorders related to immune system, heart and kidney. During the development of our own PDE4 inhibitors, roflumilast was synthesized as the positive control in the bioactivity screening, and the title compound,methyl 3,4-bis(cyclopropylmethoxy)benzoate, was a byproduct during preparation of the intermediate methyl 3-(cyclopropylmethoxy)-4-hydroxybenzoate.The crystallographic analysis of the title compound is done to confirm the chemical structure of the title compound. In the title compound, all bond lengths and angles are normal and in a good agreement with those reported previously (Hou, *et al.*, 2010). In the crystal structure, the hydroxy groups are involved in the formation of intermolecular C—H···O hydrogen bonds(Tab 1), which link the molecules related by translation along axis b into one-dimensional chains.

Experimental

A mixture of 3,4-dihydroxy methyl benzoate (1.68 g, 10 mmol) and potassium carbonate (2.76 g, 20 mmol) in acetone (50 ml) was added with a solution of cyclopropyl methyl bromide (1.35 g, 10 mmol) in acetone (50 ml). The reaction mixture was stirred at 40 °C for 18 h, and then was filtered. The filtrate was evaporated on a rotary evaporator to get the dried solid, which was then purified by flash column chromatography to obtain methyl 3-(cyclopropylmethoxy)-4-hydroxybenzoate, methyl 4-(cyclopropylmethoxy)-3-hydroxybenzoate, and the title compound methyl 3,4-bis(cyclopropylmethoxy)benzoate(Bose,*et al.*,2005).

Crystals suitable for X-ray diffraction were obtained through slow evaporation of a solution of the pure title compound in ethyl acetate/n-hexane (1/10 by volume).

Refinement

H atoms were positioned geometrically (C—H = 0.95–1.00 Å) and refined as riding, with U_{iso}(H) = 1.2 U_{eq} of the parent atom.

Figures

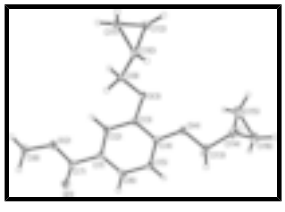

Fig. 1. The molecular structure of the title compound, with atom labels and 30% probability displacement ellipsoids, and H atoms are shown as small spheres of arbitrary radius.

Fig. 2. The packing of the title compound, showing the one-dimensional structure,with intermolecular hydrogen bonds (dashed lines); for clarity H atoms have been omitted.

Methyl 3,4-bis(cyclopropylmethoxy)benzoate

Crystal data

$C_{16}H_{20}O_4$	$F(000) = 592.00$
$M_r = 276.33$	$D_x = 1.278$ Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation, $\lambda = 0.71075$ Å
Hall symbol: P 2ac 2ab	Cell parameters from 6064 reflections
$a = 4.9018$ (8) Å	$\theta = 1.3$–$31.4°$
$b = 15.543$ (2) Å	$\mu = 0.09$ mm^{-1}
$c = 18.846$ (2) Å	$T = 113$ K
$V = 1435.9$ (3) Å^3	Prism, colorless
$Z = 4$	0.22 × 0.20 × 0.18 mm

Data collection

Rigaku Saturn724 CCD diffractometer	3300 reflections with $F^2 > 2.0\sigma(F^2)$
Detector resolution: 14.222 pixels mm^{-1}	$R_{int} = 0.034$
ω scans	$\theta_{max} = 29.1°$
Absorption correction: multi-scan (REQAB; Jacobson, 1998)	$h = -6\rightarrow6$
$T_{min} = 0.891$, $T_{max} = 0.984$	$k = -21\rightarrow21$
20068 measured reflections	$l = -25\rightarrow25$
3852 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.041$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.114$	H-atom parameters constrained
$S = 1.06$	$w = 1/[\sigma^2(F_o^2) + (0.0778P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
3852 reflections	$(\Delta/\sigma)_{max} = 0.001$
182 parameters	$\Delta\rho_{max} = 0.57$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.19$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	

Special details

Geometry. ENTER SPECIAL DETAILS OF THE MOLECULAR GEOMETRY

Refinement. Refinement was performed using all reflections. The weighted *R*-factor (*wR*) and goodness of fit (*S*) are based on F^2. *R*-factor (gt) are based on *F*. The threshold expression of $F^2 > 2.0\ \sigma(F^2)$ is used only for calculating *R*-factor (gt).

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O(1)	0.3872 (2)	0.69719 (7)	0.66936 (5)	0.0321
O(2)	0.6968 (2)	0.59026 (7)	0.66908 (5)	0.0306
O(3)	0.7825 (2)	0.48335 (5)	0.42141 (5)	0.0209
O(4)	0.4320 (2)	0.56811 (6)	0.34625 (4)	0.0250
C(1)	0.4862 (3)	0.62225 (8)	0.56094 (7)	0.0192
C(2)	0.6558 (3)	0.56094 (8)	0.52884 (7)	0.0184
C(3)	0.6319 (3)	0.54351 (8)	0.45719 (6)	0.0174
C(4)	0.4378 (3)	0.58918 (8)	0.41612 (7)	0.0193
C(5)	0.2695 (3)	0.64967 (8)	0.44863 (7)	0.0213
C(6)	0.2930 (3)	0.66574 (8)	0.52102 (7)	0.0211
C(7)	0.5139 (3)	0.64194 (8)	0.63788 (7)	0.0216
C(8)	0.7487 (4)	0.60502 (11)	0.74361 (7)	0.0406
C(9)	0.9395 (3)	0.42507 (8)	0.46452 (6)	0.0199
C(10)	1.0118 (3)	0.34763 (8)	0.42075 (7)	0.0205
C(11)	1.2662 (3)	0.29938 (9)	0.44212 (8)	0.0262
C(12)	1.2614 (3)	0.35005 (9)	0.37423 (7)	0.0246
C(13)	0.2283 (3)	0.61039 (9)	0.30217 (7)	0.0302
C(14)	0.2718 (4)	0.57919 (10)	0.22787 (7)	0.0330
C(15)	0.5052 (4)	0.61632 (12)	0.18752 (10)	0.0442
C(16)	0.2225 (4)	0.64137 (13)	0.16830 (8)	0.0428
H(2)	0.7878	0.5312	0.5565	0.022*
H(5)	0.1381	0.6800	0.4213	0.026*
H(6)	0.1765	0.7066	0.5431	0.025*
H(8A)	0.5750	0.6078	0.7693	0.049*
H(8B)	0.8469	0.6595	0.7495	0.049*
H(8C)	0.8593	0.5578	0.7626	0.049*
H(9A)	0.8319	0.4073	0.5065	0.024*
H(9B)	1.1078	0.4538	0.4813	0.024*
H(10)	0.8558	0.3120	0.4033	0.025*
H(11A)	1.3747	0.3225	0.4821	0.031*
H(11B)	1.2633	0.2358	0.4386	0.031*
H(12A)	1.2558	0.3176	0.3291	0.030*
H(12B)	1.3671	0.4043	0.3725	0.030*
H(13A)	0.2504	0.6736	0.3045	0.036*
H(13B)	0.0423	0.5955	0.3186	0.036*
H(14)	0.2271	0.5175	0.2183	0.040*
H(15A)	0.6201	0.6597	0.2118	0.053*
H(15B)	0.6047	0.5779	0.1546	0.053*

H(16A)	0.1462	0.6184	0.1235	0.051*
H(16B)	0.1615	0.7002	0.1807	0.051*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O(1)	0.0398	0.0311	0.0253	0.0080	0.0001	−0.0084
O(2)	0.0391	0.0348	0.0179	0.0103	−0.0046	−0.0070
O(3)	0.0251	0.0207	0.0170	0.0073	−0.0011	0.0001
O(4)	0.0346	0.0232	0.0170	0.0084	−0.0041	0.0008
C(1)	0.0205	0.0176	0.0194	−0.0020	0.0025	−0.0008
C(2)	0.0171	0.0187	0.0194	−0.0004	−0.0002	−0.0003
C(3)	0.0192	0.0152	0.0178	0.0006	0.0017	−0.0000
C(4)	0.0228	0.0182	0.0170	−0.0013	−0.0017	0.0020
C(5)	0.0222	0.0188	0.0229	0.0010	−0.0014	0.0024
C(6)	0.0213	0.0185	0.0235	0.0012	0.0042	−0.0012
C(7)	0.0243	0.0197	0.0208	−0.0031	0.0009	−0.0025
C(8)	0.0575	0.0441	0.0202	0.0155	−0.0122	−0.0113
C(9)	0.0213	0.0205	0.0179	0.0038	−0.0004	0.0030
C(10)	0.0189	0.0203	0.0222	0.0011	0.0009	0.0013
C(11)	0.0251	0.0244	0.0291	0.0071	0.0026	0.0046
C(12)	0.0247	0.0255	0.0236	0.0017	0.0060	0.0002
C(13)	0.0337	0.0335	0.0235	0.0069	−0.0052	0.0038
C(14)	0.0389	0.0350	0.0251	0.0013	−0.0049	0.0053
C(15)	0.0457	0.0498	0.0371	0.0028	0.0038	0.0098
C(16)	0.0510	0.0542	0.0230	0.0148	−0.0043	0.0114

Geometric parameters (Å, °)

O(1)—C(7)	1.2143 (17)	C(15)—C(16)	1.484 (3)
O(2)—C(7)	1.3397 (11)	C(2)—H(2)	0.950
O(2)—C(8)	1.4458 (19)	C(5)—H(5)	0.950
O(3)—C(3)	1.3692 (14)	C(6)—H(6)	0.950
O(3)—C(9)	1.4397 (10)	C(8)—H(8A)	0.980
O(4)—C(4)	1.3572 (17)	C(8)—H(8B)	0.980
O(4)—C(13)	1.4558 (13)	C(8)—H(8C)	0.980
C(1)—C(2)	1.4017 (18)	C(9)—H(9A)	0.990
C(1)—C(6)	1.3856 (15)	C(9)—H(9B)	0.990
C(1)—C(7)	1.488 (3)	C(10)—H(10)	1.000
C(2)—C(3)	1.382 (3)	C(11)—H(11A)	0.990
C(3)—C(4)	1.4169 (14)	C(11)—H(11B)	0.990
C(4)—C(5)	1.3930 (18)	C(12)—H(12A)	0.990
C(5)—C(6)	1.392 (3)	C(12)—H(12B)	0.990
C(9)—C(10)	1.502 (2)	C(13)—H(13A)	0.990
C(10)—C(11)	1.5098 (14)	C(13)—H(13B)	0.990
C(10)—C(12)	1.506 (2)	C(14)—H(14)	1.000
C(11)—C(12)	1.5026 (17)	C(15)—H(15A)	0.990
C(13)—C(14)	1.497 (3)	C(15)—H(15B)	0.990
C(14)—C(15)	1.490 (3)	C(16)—H(16A)	0.990

C(14)—C(16)	1.5009 (15)	C(16)—H(16B)	0.990
O(1)···C(6)	2.875 (2)	H(6)···C(1)^{viii}	3.4340
O(1)···C(8)	2.6738 (14)	H(6)···C(2)^{iv}	3.4221
O(2)···C(2)	2.6895 (19)	H(6)···C(4)^{viii}	3.4694
O(3)···O(4)	2.5873 (12)	H(6)···C(5)^{viii}	2.9995
O(3)···C(12)	3.2546 (14)	H(6)···C(6)^{viii}	2.9886
O(4)···C(15)	3.1046 (16)	H(6)···H(2)^{iv}	3.3350
C(1)···C(4)	2.787 (3)	H(6)···H(5)^{viii}	3.2433
C(2)···C(5)	2.7880 (15)	H(6)···H(5)^{i}	2.9456
C(2)···C(9)	2.804 (2)	H(6)···H(6)^{viii}	3.2354
C(3)···C(6)	2.7955 (18)	H(6)···H(6)^{i}	3.2354
C(5)···C(13)	2.834 (3)	H(8A)···O(3)^{ii}	3.2735
O(1)···C(13)^{i}	3.468 (4)	H(8A)···C(10)^{ii}	2.9685
O(2)···C(15)^{ii}	3.545 (2)	H(8A)···C(12)^{ii}	2.6574
O(3)···C(5)^{iii}	3.556 (2)	H(8A)···C(14)^{ix}	3.4558
O(3)···C(12)^{iv}	3.407 (2)	H(8A)···H(8C)^{iv}	3.5954
O(4)···C(12)^{iv}	3.5306 (19)	H(8A)···H(10)^{ii}	2.8381
C(2)···C(6)^{iii}	3.526 (3)	H(8A)···H(12A)^{ii}	2.2901
C(3)···C(5)^{iii}	3.538 (3)	H(8A)···H(12B)^{ii}	2.9188
C(5)···O(3)^{iv}	3.556 (3)	H(8A)···H(12B)^{v}	3.3617
C(5)···C(3)^{iv}	3.538 (3)	H(8A)···H(13B)^{ix}	3.3436
C(6)···C(2)^{iv}	3.526 (3)	H(8A)···H(14)^{ix}	2.6278
C(8)···C(12)^{ii}	3.578 (3)	H(8A)···H(16B)^{i}	3.1585
C(8)···C(12)^{v}	3.509 (7)	H(8B)···O(1)^{iii}	3.1047
C(12)···O(3)^{iii}	3.407 (4)	H(8B)···C(12)^{v}	3.0389
C(12)···O(4)^{iii}	3.531 (3)	H(8B)···C(16)^{i}	3.5151
C(12)···C(8)^{vi}	3.578 (5)	H(8B)···H(10)^{ii}	3.0967
C(12)···C(8)^{vii}	3.509 (5)	H(8B)···H(12A)^{ii}	3.3323
C(13)···O(1)^{viii}	3.468 (3)	H(8B)···H(12A)^{v}	2.4836
C(15)···O(2)^{vi}	3.545 (3)	H(8B)···H(12B)^{v}	2.8849
C(15)···C(16)^{iii}	3.556 (4)	H(8B)···H(13A)^{i}	2.8266
C(16)···C(15)^{iv}	3.556 (3)	H(8B)···H(14)^{ii}	3.5027
O(1)···H(6)	2.5982	H(8B)···H(15A)^{i}	3.1979
O(1)···H(8A)	2.5145	H(8B)···H(16B)^{i}	2.7043
O(1)···H(8B)	2.7752	H(8C)···O(3)^{ii}	3.1385
O(2)···H(2)	2.3550	H(8C)···O(4)^{ii}	2.7133
O(3)···H(2)	2.6517	H(8C)···C(12)^{v}	3.1519
O(3)···H(10)	2.7092	H(8C)···C(13)^{ii}	3.3878
O(3)···H(12B)	3.2512	H(8C)···C(14)^{ii}	2.8694
O(4)···H(5)	2.6652	H(8C)···C(15)^{ii}	3.1254
O(4)···H(14)	2.7276	H(8C)···H(8A)^{iii}	3.5954

O(4)···H(15A)	3.0496	H(8C)···H(10)^{ii}	3.4987
C(1)···H(5)	3.2624	H(8C)···H(12A)^{v}	2.9798
C(2)···H(6)	3.2736	H(8C)···H(12B)^{v}	2.5373
C(2)···H(9A)	2.5735	H(8C)···H(13B)^{ix}	3.2663
C(2)···H(9B)	2.9122	H(8C)···H(14)^{ix}	3.2138
C(3)···H(5)	3.2890	H(8C)···H(14)^{ii}	2.4852
C(3)···H(9A)	2.5109	H(8C)···H(15A)^{ii}	3.5154
C(3)···H(9B)	2.7551	H(8C)···H(15B)^{ii}	2.9371
C(4)···H(2)	3.2784	H(9A)···C(11)^{iv}	3.4606
C(4)···H(6)	3.2707	H(9A)···C(11)^{x}	3.3711
C(4)···H(13A)	2.6440	H(9A)···C(15)^{ii}	3.5231
C(4)···H(13B)	2.6731	H(9A)···H(11A)^{iv}	2.6402
C(5)···H(13A)	2.7429	H(9A)···H(11A)^{x}	3.5855
C(5)···H(13B)	2.8201	H(9A)···H(11B)^{x}	2.4769
C(6)···H(2)	3.2707	H(9A)···H(12B)^{iv}	3.4008
C(7)···H(2)	2.6681	H(9A)···H(15B)^{ii}	2.8173
C(7)···H(6)	2.6334	H(9A)···H(16A)^{ix}	3.2426
C(7)···H(8A)	2.5499	H(9A)···H(16A)^{ii}	3.4014
C(7)···H(8B)	2.6766	H(9B)···O(3)^{iii}	3.5250
C(7)···H(8C)	3.1783	H(9B)···O(4)^{iii}	3.4870
C(9)···H(2)	2.5055	H(9B)···C(1)^{iii}	3.5419
C(9)···H(11A)	2.6830	H(9B)···C(2)^{iii}	3.2847
C(9)···H(11B)	3.3775	H(9B)···C(3)^{iii}	2.9578
C(9)···H(12A)	3.4221	H(9B)···C(4)^{iii}	2.9243
C(9)···H(12B)	2.7392	H(9B)···C(5)^{iii}	3.2049
C(11)···H(9A)	2.9697	H(9B)···C(6)^{iii}	3.4973
C(11)···H(9B)	2.6293	H(9B)···H(15B)^{ii}	3.4627
C(12)···H(9A)	3.3820	H(9B)···H(16A)^{ii}	3.1460
C(12)···H(9B)	2.6912	H(10)···C(8)^{vi}	3.3147
C(13)···H(5)	2.5315	H(10)···C(11)^{iv}	2.9874
C(13)···H(15A)	2.6793	H(10)···C(11)^{x}	3.4165
C(13)···H(15B)	3.3759	H(10)···C(12)^{iv}	3.0231
C(13)···H(16A)	3.3933	H(10)···C(16)^{xi}	3.0002
C(13)···H(16B)	2.7007	H(10)···H(8A)^{vi}	2.8381
C(15)···H(13A)	2.6862	H(10)···H(8B)^{vi}	3.0967
C(15)···H(13B)	3.3697	H(10)···H(8C)^{vi}	3.4987
C(16)···H(13A)	2.6193	H(10)···H(11A)^{iv}	2.7913
C(16)···H(13B)	3.0514	H(10)···H(11A)^{x}	3.0070
H(2)···H(9A)	2.1545	H(10)···H(11B)^{iv}	3.2057
H(2)···H(9B)	2.4315	H(10)···H(11B)^{x}	3.1032
H(5)···H(6)	2.3396	H(10)···H(12A)^{iv}	3.2582

H(5)···H(13A)	2.2709	H(10)···H(12B)^{iv}	2.8516
H(5)···H(13B)	2.3861	H(10)···H(16A)^{xi}	3.0514
H(9A)···H(10)	2.4474	H(10)···H(16B)^{xi}	2.3527
H(9A)···H(11A)	3.0045	H(11A)···O(3)^{iii}	3.3990
H(9B)···H(10)	2.9237	H(11A)···C(9)^{iii}	3.2119
H(9B)···H(11A)	2.4243	H(11A)···C(10)^{iii}	3.3530
H(9B)···H(11B)	3.5652	H(11A)···C(10)^{xii}	3.2863
H(9B)···H(12B)	2.5324	H(11A)···C(11)^{xii}	3.0519
H(10)···H(11A)	2.9497	H(11A)···C(16)^{ii}	3.5854
H(10)···H(11B)	2.4153	H(11A)···H(9A)^{iii}	2.6402
H(10)···H(12A)	2.4108	H(11A)···H(9A)^{xii}	3.5855
H(10)···H(12B)	2.9459	H(11A)···H(10)^{iii}	2.7913
H(11A)···H(12A)	2.9435	H(11A)···H(10)^{xii}	3.0070
H(11A)···H(12B)	2.4248	H(11A)···H(11A)^{x}	3.3981
H(11B)···H(12A)	2.4247	H(11A)···H(11A)^{xii}	3.3981
H(11B)···H(12B)	2.9436	H(11A)···H(11B)^{x}	3.4694
H(13A)···H(14)	2.9227	H(11A)···H(11B)^{xii}	2.5855
H(13A)···H(15A)	2.5272	H(11A)···H(16A)^{ii}	2.8206
H(13A)···H(16A)	3.5547	H(11B)···C(9)^{xii}	3.2146
H(13A)···H(16B)	2.4089	H(11B)···C(10)^{xii}	3.1923
H(13B)···H(14)	2.4211	H(11B)···C(11)^{x}	3.3599
H(13B)···H(16B)	3.1209	H(11B)···C(11)^{xii}	3.3805
H(14)···H(15A)	2.9345	H(11B)···C(15)^{xiii}	3.2233
H(14)···H(15B)	2.3983	H(11B)···C(16)^{xi}	3.4476
H(14)···H(16A)	2.4103	H(11B)···C(16)^{xiii}	3.5454
H(14)···H(16B)	2.9445	H(11B)···H(9A)^{xii}	2.4769
H(15A)···H(16A)	2.9283	H(11B)···H(10)^{iii}	3.2057
H(15A)···H(16B)	2.4063	H(11B)···H(10)^{xii}	3.1032
H(15B)···H(16A)	2.4063	H(11B)···H(11A)^{x}	2.5855
H(15B)···H(16B)	2.9283	H(11B)···H(11A)^{xii}	3.4694
O(1)···H(5)^{i}	2.8414	H(11B)···H(11B)^{x}	3.3990
O(1)···H(8B)^{iv}	3.1047	H(11B)···H(11B)^{xii}	3.3990
O(1)···H(12A)^{ii}	3.0988	H(11B)···H(15A)^{xiii}	3.1240
O(1)···H(13A)^{i}	2.7280	H(11B)···H(15B)^{xiii}	3.0867
O(1)···H(13B)^{i}	3.3183	H(11B)···H(16A)^{xi}	2.9551
O(1)···H(14)^{ix}	3.5071	H(11B)···H(16B)^{xi}	3.1149
O(1)···H(15A)^{viii}	3.4180	H(12A)···O(1)^{vi}	3.0988
O(1)···H(16B)^{i}	3.5118	H(12A)···C(8)^{vi}	3.1867
O(2)···H(14)^{ix}	2.8256	H(12A)···C(8)^{vii}	3.1528
O(2)···H(14)^{ii}	3.4119	H(12A)···C(15)^{xiii}	3.3551
O(2)···H(15B)^{ii}	2.8026	H(12A)···H(8A)^{vi}	2.2901

O(3)···H(5)^{iii}	3.5191	H(12A)···H(8B)^{vi}	3.3323
O(3)···H(8A)^{vi}	3.2735	H(12A)···H(8B)^{vii}	2.4836
O(3)···H(8C)^{vi}	3.1385	H(12A)···H(8C)^{vii}	2.9798
O(3)···H(9B)^{iv}	3.5250	H(12A)···H(10)^{iii}	3.2582
O(3)···H(11A)^{iv}	3.3990	H(12A)···H(15A)^{xiii}	2.6428
O(3)···H(12B)^{iv}	2.5506	H(12A)···H(16B)^{xi}	2.7473
O(3)···H(13B)^{iii}	2.9009	H(12A)···H(16B)^{xiii}	3.3943
O(4)···H(8C)^{vi}	2.7133	H(12B)···O(3)^{iii}	2.5506
O(4)···H(9B)^{iv}	3.4870	H(12B)···O(4)^{iii}	2.6136
O(4)···H(12B)^{iv}	2.6136	H(12B)···C(3)^{iii}	2.9854
O(4)···H(13B)^{iii}	3.0664	H(12B)···C(4)^{iii}	3.0090
C(1)···H(5)^{i}	3.1796	H(12B)···C(8)^{vii}	3.0774
C(1)···H(6)^{i}	3.4340	H(12B)···C(9)^{iii}	3.3140
C(1)···H(9B)^{iv}	3.5419	H(12B)···C(10)^{iii}	3.4040
C(2)···H(6)^{iii}	3.4221	H(12B)···C(13)^{iii}	3.5333
C(2)···H(9B)^{iv}	3.2847	H(12B)···H(8A)^{vi}	2.9188
C(2)···H(15B)^{ii}	3.4133	H(12B)···H(8A)^{vii}	3.3617
C(3)···H(5)^{iii}	3.3346	H(12B)···H(8B)^{vii}	2.8849
C(3)···H(9B)^{iv}	2.9578	H(12B)···H(8C)^{vii}	2.5373
C(3)···H(12B)^{iv}	2.9854	H(12B)···H(9A)^{iii}	3.4008
C(3)···H(13B)^{iii}	3.3947	H(12B)···H(10)^{iii}	2.8516
C(4)···H(6)^{i}	3.4694	H(12B)···H(13B)^{iii}	3.5216
C(4)···H(9B)^{iv}	2.9243	H(12B)···H(14)^{iii}	3.4659
C(4)···H(12B)^{iv}	3.0090	H(13A)···O(1)^{viii}	2.7280
C(4)···H(13B)^{iii}	3.4883	H(13A)···C(7)^{viii}	3.2771
C(5)···H(6)^{i}	2.9995	H(13A)···C(8)^{viii}	3.5579
C(5)···H(9B)^{iv}	3.2049	H(13A)···H(8B)^{viii}	2.8266
C(6)···H(2)^{iv}	3.3092	H(13A)···H(15A)^{iv}	3.5566
C(6)···H(5)^{i}	3.1287	H(13B)···O(1)^{viii}	3.3183
C(6)···H(6)^{i}	2.9886	H(13B)···O(3)^{iv}	2.9009
C(6)···H(9B)^{iv}	3.4973	H(13B)···O(4)^{iv}	3.0664
C(7)···H(5)^{i}	3.0449	H(13B)···C(3)^{iv}	3.3947
C(7)···H(13A)^{i}	3.2771	H(13B)···C(4)^{iv}	3.4883
C(7)···H(14)^{ix}	3.1360	H(13B)···H(8A)^{xiv}	3.3436
C(8)···H(10)^{ii}	3.3147	H(13B)···H(8C)^{xiv}	3.2663
C(8)···H(12A)^{ii}	3.1867	H(13B)···H(12B)^{iv}	3.5216
C(8)···H(12A)^{v}	3.1528	H(13B)···H(15A)^{iv}	3.0552
C(8)···H(12B)^{v}	3.0774	H(14)···O(1)^{xiv}	3.5071
C(8)···H(13A)^{i}	3.5579	H(14)···O(2)^{xiv}	2.8256
C(8)···H(14)^{ix}	3.0483	H(14)···O(2)^{vi}	3.4119
C(8)···H(14)^{ii}	3.2335	H(14)···C(7)^{xiv}	3.1360

C(8)···H(15B)^{ii}	3.3790	H(14)···C(8)^{xiv}	3.0483
C(8)···H(16B)^{i}	3.3736	H(14)···C(8)^{vi}	3.2335
C(9)···H(11A)^{iv}	3.2119	H(14)···H(8A)^{xiv}	2.6278
C(9)···H(11B)^{x}	3.2146	H(14)···H(8B)^{vi}	3.5027
C(9)···H(12B)^{iv}	3.3140	H(14)···H(8C)^{xiv}	3.2138
C(9)···H(15B)^{ii}	3.5885	H(14)···H(8C)^{vi}	2.4852
C(10)···H(8A)^{vi}	2.9685	H(14)···H(12B)^{iv}	3.4659
C(10)···H(11A)^{iv}	3.3530	H(14)···H(15B)^{iv}	3.4109
C(10)···H(11A)^{x}	3.2863	H(15A)···O(1)^{i}	3.4180
C(10)···H(11B)^{x}	3.1923	H(15A)···C(12)^{xv}	3.4231
C(10)···H(12B)^{iv}	3.4040	H(15A)···C(13)^{iii}	3.5184
C(10)···H(16B)^{xi}	3.1033	H(15A)···C(14)^{iii}	3.4443
C(11)···H(9A)^{iii}	3.4606	H(15A)···C(16)^{iii}	3.0775
C(11)···H(9A)^{xii}	3.3711	H(15A)···H(8B)^{viii}	3.1979
C(11)···H(10)^{iii}	2.9874	H(15A)···H(8C)^{vi}	3.5154
C(11)···H(10)^{xii}	3.4165	H(15A)···H(11B)^{xv}	3.1240
C(11)···H(11A)^{x}	3.0519	H(15A)···H(12A)^{xv}	2.6428
C(11)···H(11B)^{x}	3.3805	H(15A)···H(13A)^{iii}	3.5566
C(11)···H(11B)^{xii}	3.3599	H(15A)···H(13B)^{iii}	3.0552
C(11)···H(16B)^{xi}	3.4832	H(15A)···H(16A)^{iii}	3.1351
C(12)···H(8A)^{vi}	2.6574	H(15A)···H(16B)^{iii}	2.7897
C(12)···H(8B)^{vii}	3.0389	H(15B)···O(2)^{vi}	2.8026
C(12)···H(8C)^{vii}	3.1519	H(15B)···C(2)^{vi}	3.4133
C(12)···H(10)^{iii}	3.0231	H(15B)···C(8)^{vi}	3.3790
C(12)···H(15A)^{xiii}	3.4231	H(15B)···C(9)^{vi}	3.5885
C(12)···H(16B)^{xi}	3.2856	H(15B)···C(14)^{iii}	3.5496
C(13)···H(8C)^{vi}	3.3878	H(15B)···C(16)^{iii}	3.1951
C(13)···H(12B)^{iv}	3.5333	H(15B)···H(2)^{vi}	2.5642
C(13)···H(15A)^{iv}	3.5184	H(15B)···H(8C)^{vi}	2.9371
C(14)···H(8A)^{xiv}	3.4558	H(15B)···H(9A)^{vi}	2.8173
C(14)···H(8C)^{vi}	2.8694	H(15B)···H(9B)^{vi}	3.4627
C(14)···H(15A)^{iv}	3.4443	H(15B)···H(11B)^{xv}	3.0867
C(14)···H(15B)^{iv}	3.5496	H(15B)···H(14)^{iii}	3.4109
C(15)···H(2)^{vi}	3.5201	H(15B)···H(16A)^{iii}	2.7897
C(15)···H(8C)^{vi}	3.1254	H(15B)···H(16B)^{iii}	3.3623
C(15)···H(9A)^{vi}	3.5231	H(16A)···C(15)^{iv}	3.3656
C(15)···H(11B)^{xv}	3.2233	H(16A)···H(2)^{xiv}	3.3954
C(15)···H(12A)^{xv}	3.3551	H(16A)···H(9A)^{xiv}	3.2426
C(15)···H(16A)^{iii}	3.3656	H(16A)···H(9A)^{vi}	3.4014
C(15)···H(16B)^{iii}	3.4736	H(16A)···H(9B)^{vi}	3.1460
C(16)···H(8B)^{viii}	3.5151	H(16A)···H(10)^{xvi}	3.0514

C(16)···H(10)^{xvi}	3.0002	H(16A)···H(11A)^{vi}	2.8206
C(16)···H(11A)^{vi}	3.5854	H(16A)···H(11B)^{xvi}	2.9551
C(16)···H(11B)^{xvi}	3.4476	H(16A)···H(15A)^{iv}	3.1351
C(16)···H(11B)^{xv}	3.5454	H(16A)···H(15B)^{iv}	2.7897
C(16)···H(15A)^{iv}	3.0775	H(16B)···O(1)^{viii}	3.5118
C(16)···H(15B)^{iv}	3.1951	H(16B)···C(8)^{viii}	3.3736
H(2)···C(6)^{iii}	3.3092	H(16B)···C(10)^{xvi}	3.1033
H(2)···C(15)^{ii}	3.5201	H(16B)···C(11)^{xvi}	3.4832
H(2)···H(6)^{iii}	3.3350	H(16B)···C(12)^{xvi}	3.2856
H(2)···H(15B)^{ii}	2.5642	H(16B)···C(15)^{iv}	3.4736
H(2)···H(16A)^{ix}	3.3954	H(16B)···H(8A)^{viii}	3.1585
H(5)···O(1)^{viii}	2.8414	H(16B)···H(8B)^{viii}	2.7043
H(5)···O(3)^{iv}	3.5191	H(16B)···H(10)^{xvi}	2.3527
H(5)···C(1)^{viii}	3.1796	H(16B)···H(11B)^{xvi}	3.1149
H(5)···C(3)^{iv}	3.3346	H(16B)···H(12A)^{xvi}	2.7473
H(5)···C(6)^{viii}	3.1287	H(16B)···H(12A)^{xv}	3.3943
H(5)···C(7)^{viii}	3.0449	H(16B)···H(15A)^{iv}	2.7897
H(5)···H(6)^{viii}	2.9456	H(16B)···H(15B)^{iv}	3.3623
H(5)···H(6)^{i}	3.2433		
C(7)—O(2)—C(8)	116.68 (12)	H(8A)—C(8)—H(8B)	109.475
C(3)—O(3)—C(9)	116.11 (9)	H(8A)—C(8)—H(8C)	109.468
C(4)—O(4)—C(13)	117.33 (10)	H(8B)—C(8)—H(8C)	109.472
C(2)—C(1)—C(6)	120.17 (12)	O(3)—C(9)—H(9A)	109.948
C(2)—C(1)—C(7)	120.42 (11)	O(3)—C(9)—H(9B)	109.953
C(6)—C(1)—C(7)	119.40 (11)	C(10)—C(9)—H(9A)	109.961
C(1)—C(2)—C(3)	120.30 (12)	C(10)—C(9)—H(9B)	109.956
O(3)—C(3)—C(2)	124.69 (11)	H(9A)—C(9)—H(9B)	108.328
O(3)—C(3)—C(4)	115.80 (10)	C(9)—C(10)—H(10)	116.366
C(2)—C(3)—C(4)	119.50 (11)	C(11)—C(10)—H(10)	116.367
O(4)—C(4)—C(3)	115.03 (11)	C(12)—C(10)—H(10)	116.375
O(4)—C(4)—C(5)	125.26 (12)	C(10)—C(11)—H(11A)	117.777
C(3)—C(4)—C(5)	119.71 (12)	C(10)—C(11)—H(11B)	117.773
C(4)—C(5)—C(6)	120.21 (12)	C(12)—C(11)—H(11A)	117.769
C(1)—C(6)—C(5)	120.09 (12)	C(12)—C(11)—H(11B)	117.772
O(1)—C(7)—O(2)	123.48 (12)	H(11A)—C(11)—H(11B)	114.909
O(1)—C(7)—C(1)	125.10 (12)	C(10)—C(12)—H(12A)	117.744
O(2)—C(7)—C(1)	111.42 (11)	C(10)—C(12)—H(12B)	117.746
O(3)—C(9)—C(10)	108.69 (9)	C(11)—C(12)—H(12A)	117.745
C(9)—C(10)—C(11)	116.52 (11)	C(11)—C(12)—H(12B)	117.747
C(9)—C(10)—C(12)	119.44 (11)	H(12A)—C(12)—H(12B)	114.871
C(11)—C(10)—C(12)	59.78 (9)	O(4)—C(13)—H(13A)	110.344
C(10)—C(11)—C(12)	59.97 (9)	O(4)—C(13)—H(13B)	110.344
C(10)—C(12)—C(11)	60.25 (9)	C(14)—C(13)—H(13A)	110.360
O(4)—C(13)—C(14)	106.85 (12)	C(14)—C(13)—H(13B)	110.358
C(13)—C(14)—C(15)	117.46 (14)	H(13A)—C(13)—H(13B)	108.585

C(13)—C(14)—C(16)	117.91 (14)	C(13)—C(14)—H(14)	116.594
C(15)—C(14)—C(16)	59.51 (12)	C(15)—C(14)—H(14)	116.598
C(14)—C(15)—C(16)	60.61 (12)	C(16)—C(14)—H(14)	116.603
C(14)—C(16)—C(15)	59.89 (12)	C(14)—C(15)—H(15A)	117.706
C(1)—C(2)—H(2)	119.856	C(14)—C(15)—H(15B)	117.711
C(3)—C(2)—H(2)	119.846	C(16)—C(15)—H(15A)	117.702
C(4)—C(5)—H(5)	119.900	C(16)—C(15)—H(15B)	117.720
C(6)—C(5)—H(5)	119.892	H(15A)—C(15)—H(15B)	114.826
C(1)—C(6)—H(6)	119.947	C(14)—C(16)—H(16A)	117.785
C(5)—C(6)—H(6)	119.960	C(14)—C(16)—H(16B)	117.794
O(2)—C(8)—H(8A)	109.469	C(15)—C(16)—H(16A)	117.781
O(2)—C(8)—H(8B)	109.474	C(15)—C(16)—H(16B)	117.788
O(2)—C(8)—H(8C)	109.470	H(16A)—C(16)—H(16B)	114.905
C(8)—O(2)—C(7)—O(1)	−2.4 (2)	C(2)—C(3)—C(4)—O(4)	179.31 (11)
C(8)—O(2)—C(7)—C(1)	177.99 (12)	C(2)—C(3)—C(4)—C(5)	−1.41 (19)
C(3)—O(3)—C(9)—C(10)	160.99 (10)	O(4)—C(4)—C(5)—C(6)	179.71 (12)
C(9)—O(3)—C(3)—C(2)	11.35 (17)	C(3)—C(4)—C(5)—C(6)	0.52 (20)
C(9)—O(3)—C(3)—C(4)	−167.98 (11)	C(4)—C(5)—C(6)—C(1)	0.69 (20)
C(4)—O(4)—C(13)—C(14)	177.72 (12)	O(3)—C(9)—C(10)—C(11)	153.94 (11)
C(13)—O(4)—C(4)—C(3)	177.39 (11)	O(3)—C(9)—C(10)—C(12)	85.31 (14)
C(13)—O(4)—C(4)—C(5)	−1.83 (19)	C(9)—C(10)—C(11)—C(12)	−110.19 (13)
C(2)—C(1)—C(6)—C(5)	−1.01 (19)	C(9)—C(10)—C(12)—C(11)	105.34 (13)
C(6)—C(1)—C(2)—C(3)	0.09 (19)	C(11)—C(10)—C(12)—C(11)	0.0
C(2)—C(1)—C(7)—O(1)	175.66 (13)	C(12)—C(10)—C(11)—C(12)	0.0
C(2)—C(1)—C(7)—O(2)	−4.76 (17)	C(10)—C(11)—C(12)—C(10)	0.0
C(7)—C(1)—C(2)—C(3)	−179.24 (12)	O(4)—C(13)—C(14)—C(15)	−76.77 (16)
C(6)—C(1)—C(7)—O(1)	−3.7 (2)	O(4)—C(13)—C(14)—C(16)	−144.94 (14)
C(6)—C(1)—C(7)—O(2)	175.90 (12)	C(13)—C(14)—C(15)—C(16)	−107.83 (16)
C(7)—C(1)—C(6)—C(5)	178.33 (12)	C(13)—C(14)—C(16)—C(15)	107.08 (17)
C(1)—C(2)—C(3)—O(3)	−178.20 (12)	C(15)—C(14)—C(16)—C(15)	0.0
C(1)—C(2)—C(3)—C(4)	1.11 (19)	C(16)—C(14)—C(15)—C(16)	0.0
O(3)—C(3)—C(4)—O(4)	−1.31 (17)	C(14)—C(15)—C(16)—C(14)	0.0
O(3)—C(3)—C(4)—C(5)	177.96 (11)		

Symmetry codes: (i) *x*+1/2, −*y*+3/2, −*z*+1; (ii) −*x*+3/2, −*y*+1, *z*+1/2; (iii) *x*+1, *y*, *z*; (iv) *x*−1, *y*, *z*; (v) −*x*+5/2, −*y*+1, *z*+1/2; (vi) −*x*+3/2, −*y*+1, *z*−1/2; (vii) −*x*+5/2, −*y*+1, *z*−1/2; (viii) *x*−1/2, −*y*+3/2, −*z*+1; (ix) −*x*+1/2, −*y*+1, *z*+1/2; (x) *x*−1/2, −*y*+1/2, −*z*+1; (xi) −*x*+1, *y*−1/2, −*z*+1/2; (xii) *x*+1/2, −*y*+1/2, −*z*+1; (xiii) −*x*+2, *y*−1/2, −*z*+1/2; (xiv) −*x*+1/2, −*y*+1, *z*−1/2; (xv) −*x*+2, *y*+1/2, −*z*+1/2; (xvi) −*x*+1, *y*+1/2, −*z*+1/2.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12B···O3^{iii}	0.99	2.55	3.4073 (18)	145

Symmetry codes: (iii) *x*+1, *y*, *z*.

Fig. 1

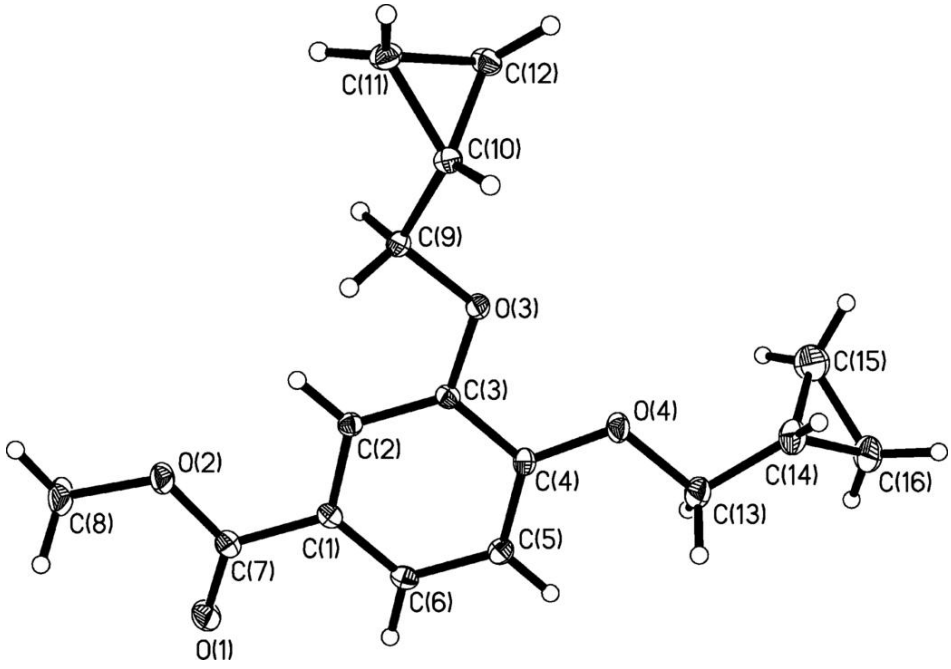

Fig. 2