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6,6′-Oxydichroman

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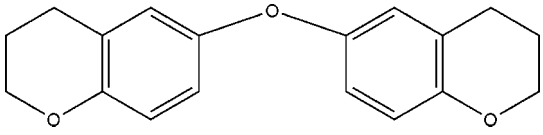
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.006 Å; R factor = 0.072; wR factor = 0.216; data-to-parameter ratio = 8.9.

The title compound, $C_{18}H_{18}O_3$, was synthesized from dichroman in concentrated sulfuric acid. The molecule has a twofold axis passing through the central O atom. The dihedral angle between the two symmetry-related benzene rings is 63.6 (3)°. Weak C—H$\cdots\pi$ interactions are present in the structure.

Related literature

For related literature, see: Allen *et al.* (1987); Li *et al.* (2006, 2007); Xiao, Shi *et al.* (2007); Xiao, Xue *et al.* (2007); Huang *et al.* (2007); Zhang *et al.* (2007); Shi *et al.* (2007); Cao *et al.* (2007); Ruan *et al.* (2006).



Experimental

Crystal data

$C_{18}H_{18}O_3$
M_r = 282.32
Orthorhombic, *Fdd*2
a = 17.515 (4) Å
b = 29.660 (6) Å
c = 5.7680 (12) Å
V = 2996.4 (10) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 298 (2) K
0.23 × 0.20 × 0.20 mm

Data collection

Bruker APEX area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.981, T_{max} = 0.983
4727 measured reflections
855 independent reflections
810 reflections with $I > 2\sigma(I)$
R_{int} = 0.073

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.073
$wR(F^2)$ = 0.216
S = 1.13
855 reflections
96 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.55 e Å^{-3}

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

*Cg*1 is the centroid of the ring C1–C6.

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C8—H8*B*$\cdots$*Cg*1^{i}	0.97	2.91	3.856 (6)	167

Symmetry code: (i) $x+\frac{1}{4}, -y+\frac{3}{4}, z-\frac{1}{4}$.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1996); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BQ2049).

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supporting information

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6,6′-Oxydichroman

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S1. Comment

Synthesized organic compounds with a pyran cycle are found being good biological activities (Li *et al.*, 2007; Xiao, Shi *et al.*, 2007; Xiao, Xue *et al.*, 2007; Huang *et al.*, 2007; Zhang *et al.*, 2007; Shi *et al.*, 2007; Cao *et al.*, 2007; Ruan *et al.*, 2006; Li *et al.*, 2006). So we prepared a series of derivatives with pyran cycles. Here we report the crystal structure of the title compound.

The title compound consists of an oxygen atom bridged two chroman (Fig. 1). The molecule has a twofold axis symmetry position at the central O1 atom. The dihedal angle between the two symmetry-related benzene rings is 63.6 (3)°. In each chroman, all the atoms, except C8 atom, are nearly coplanar, with mean deviation from plane by 0.021 (4) Å. C8 atom is located 0.577 (4)Å above the plane defined by other non-hydrogen atoms. All the bond values are within normal ranges (Allen *et al.*, 1987). There exists weak C8–H8B···*Cg*1 interaction (*Cg*1:C1—C6) (Table 1, Fig. 2).

S2. Experimental

Dichroman was disolved in toluene solution and a few drops of concentrated sulfuric acid was added. The above solution was refluxed for two hours. After the solution was cooled to room temperature colorless microcrystals were precipitated. They were filtered, washed with toluene for three times. Yield: 32%.

S3. Refinement

C-bound H atoms were included in the riding model approximation with C—H = 0.93 Å, and with U_{iso}(H) = 1.2U_{eq}(C). 631 Friedel pairs were averaged before the final refinement as the absolute configuration could not be determined unambiguously.

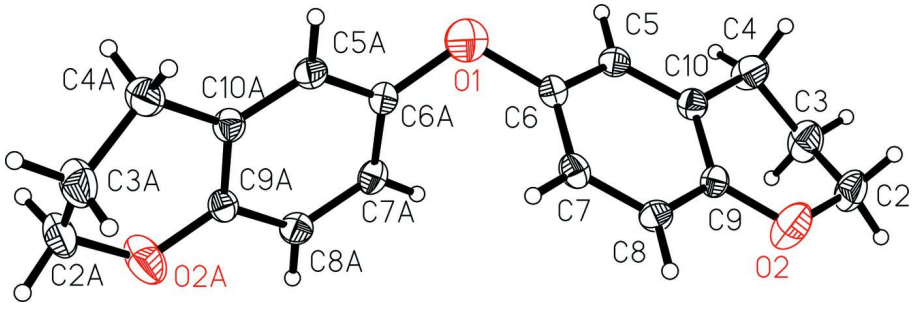

Figure 1

The molecular structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme. Symmetry code (i): -*x*, 1 - *y*, *z*.

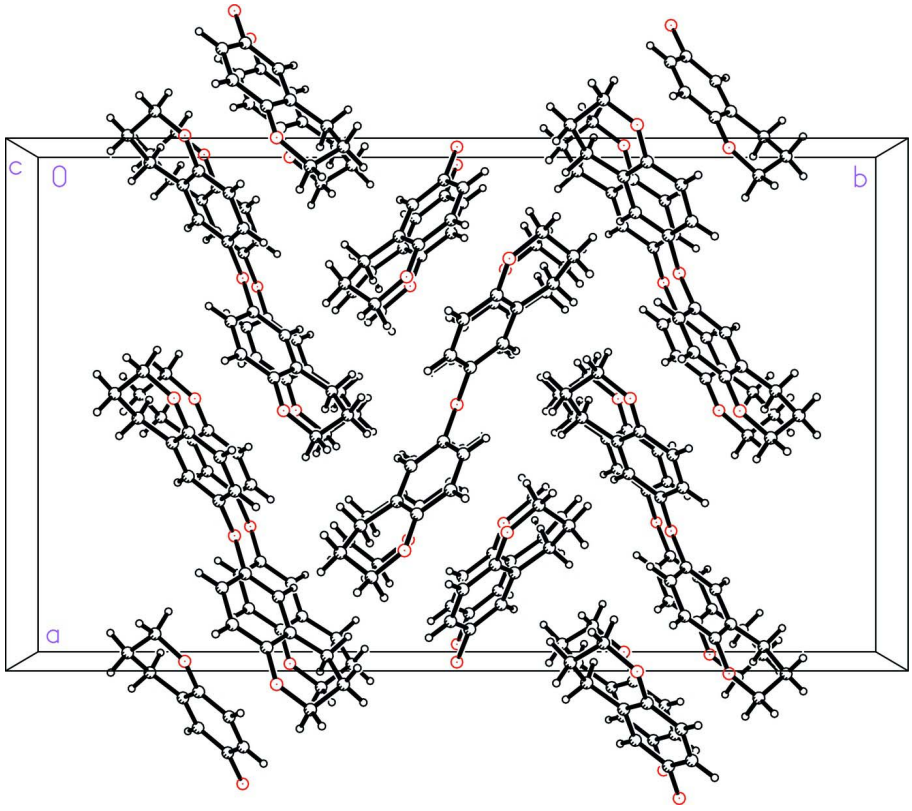

Figure 2

Molecular packing of (I) viewed down the *c* axis. The weak C–H···*Cg* interactions are shown as dashed lines.

6,6′-Oxydichroman

Crystal data

$C_{18}H_{18}O_3$
$M_r = 282.32$
Orthorhombic, *Fdd*2
Hall symbol: F 2 -2d
$a = 17.515\ (4)$ Å
$b = 29.660\ (6)$ Å
$c = 5.7680\ (12)$ Å
$V = 2996.4\ (10)$ Å^3
$Z = 8$
$F(000) = 1200$
$D_x = 1.252$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 872 reflections
$\theta = 2.5$–$24.3°$
$\mu = 0.08$ mm^{-1}
$T = 298$ K
Prism, colorless
0.23 × 0.20 × 0.20 mm

Data collection

Bruker APEX area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.981$, $T_{max} = 0.983$
4727 measured reflections
855 independent reflections
810 reflections with $I > 2\sigma(I)$
$R_{int} = 0.073$
$\theta_{max} = 26.5°$, $\theta_{min} = 2.7°$
$h = -21 \rightarrow 21$
$k = -36 \rightarrow 32$
$l = -7 \rightarrow 6$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.073$
$wR(F^2) = 0.216$
$S = 1.13$
855 reflections
96 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
$w = 1/[\sigma^2(F_o^2) + (0.1367P)^2 + 4.7011P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.32$ e Å^{-3}
$\Delta\rho_{min} = -0.55$ e Å^{-3}
Absolute structure: Flack (1983), 631 Friedel pairs

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.5000	0.5000	0.0623 (13)	0.090 (2)
O2	0.7616 (2)	0.44456 (13)	0.5838 (9)	0.0779 (14)
C1	0.6782 (2)	0.43486 (12)	0.2525 (7)	0.0378 (9)
C2	0.6140 (2)	0.44952 (12)	0.1313 (7)	0.0387 (9)
H2	0.6008	0.4345	−0.0045	0.046*
C3	0.5687 (2)	0.48541 (13)	0.2029 (8)	0.0386 (9)
C4	0.5900 (2)	0.50701 (12)	0.4073 (8)	0.0412 (9)
H4	0.5609	0.5310	0.4615	0.049*
C5	0.6539 (2)	0.49345 (13)	0.5317 (8)	0.0413 (9)
H5	0.6670	0.5087	0.6669	0.050*
C6	0.69851 (19)	0.45734 (12)	0.4570 (7)	0.0360 (8)
C7	0.7245 (3)	0.39470 (15)	0.1747 (9)	0.0561 (12)
H7A	0.7590	0.4039	0.0521	0.067*
H7B	0.6904	0.3719	0.1122	0.067*
C8	0.7703 (3)	0.37452 (17)	0.3725 (12)	0.0640 (14)
H8A	0.7361	0.3583	0.4754	0.077*
H8B	0.8067	0.3531	0.3104	0.077*
C9	0.8113 (3)	0.40910 (16)	0.5053 (11)	0.0614 (15)
H9A	0.8355	0.3951	0.6384	0.074*
H9B	0.8511	0.4220	0.4091	0.074*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.081 (4)	0.128 (5)	0.060 (4)	0.032 (3)	0.000	0.000
O2	0.074 (2)	0.075 (2)	0.085 (3)	0.0149 (17)	−0.036 (2)	−0.021 (2)
C1	0.0383 (17)	0.0360 (17)	0.039 (2)	0.0011 (13)	−0.0009 (17)	−0.0039 (17)
C2	0.0395 (18)	0.0405 (17)	0.0360 (19)	0.0017 (14)	−0.0002 (17)	−0.0066 (16)
C3	0.0315 (16)	0.0445 (17)	0.040 (2)	0.0029 (13)	0.0022 (16)	0.0016 (17)
C4	0.0379 (18)	0.0384 (17)	0.047 (2)	0.0051 (14)	0.0054 (17)	−0.0053 (18)
C5	0.0408 (19)	0.0434 (19)	0.040 (2)	−0.0047 (15)	−0.0021 (17)	−0.0125 (17)
C6	0.0313 (15)	0.0370 (16)	0.040 (2)	−0.0023 (13)	−0.0014 (16)	0.0013 (16)
C7	0.065 (3)	0.053 (2)	0.051 (3)	0.019 (2)	−0.011 (2)	−0.010 (2)
C8	0.059 (3)	0.057 (2)	0.076 (4)	0.020 (2)	−0.010 (3)	−0.003 (3)
C9	0.048 (2)	0.064 (3)	0.072 (4)	0.0163 (19)	−0.018 (3)	−0.001 (3)

Geometric parameters (Å, º)

O1—C3^{i}	1.514 (5)	C4—H4	0.9300
O1—C3	1.514 (6)	C5—C6	1.394 (5)
O2—C6	1.379 (5)	C5—H5	0.9300
O2—C9	1.438 (6)	C7—C8	1.518 (7)
C1—C2	1.393 (5)	C7—H7A	0.9700
C1—C6	1.401 (6)	C7—H7B	0.9700
C1—C7	1.509 (5)	C8—C9	1.468 (7)
C2—C3	1.390 (5)	C8—H8A	0.9700
C2—H2	0.9300	C8—H8B	0.9700
C3—C4	1.393 (6)	C9—H9A	0.9700
C4—C5	1.388 (6)	C9—H9B	0.9700
C3^{i}—O1—C3	115.2 (6)	C5—C6—C1	118.9 (3)
C6—O2—C9	121.3 (4)	C1—C7—C8	111.8 (4)
C2—C1—C6	118.6 (3)	C1—C7—H7A	109.3
C2—C1—C7	122.0 (4)	C8—C7—H7A	109.3
C6—C1—C7	119.3 (3)	C1—C7—H7B	109.3
C3—C2—C1	123.4 (4)	C8—C7—H7B	109.3
C3—C2—H2	118.3	H7A—C7—H7B	107.9
C1—C2—H2	118.3	C9—C8—C7	112.1 (4)
C2—C3—C4	116.8 (3)	C9—C8—H8A	109.2
C2—C3—O1	120.9 (4)	C7—C8—H8A	109.2
C4—C3—O1	122.4 (4)	C9—C8—H8B	109.2
C5—C4—C3	121.4 (3)	C7—C8—H8B	109.2
C5—C4—H4	119.3	H8A—C8—H8B	107.9
C3—C4—H4	119.3	O2—C9—C8	112.3 (4)
C4—C5—C6	121.0 (4)	O2—C9—H9A	109.1
C4—C5—H5	119.5	C8—C9—H9A	109.1
C6—C5—H5	119.5	O2—C9—H9B	109.1
O2—C6—C5	119.8 (4)	C8—C9—H9B	109.1
O2—C6—C1	121.3 (3)	H9A—C9—H9B	107.9

C6—C1—C2—C3	−0.1 (6)	C4—C5—C6—O2	−180.0 (4)
C7—C1—C2—C3	177.7 (4)	C4—C5—C6—C1	0.3 (6)
C1—C2—C3—C4	−0.2 (6)	C2—C1—C6—O2	−179.7 (4)
C1—C2—C3—O1	179.4 (3)	C7—C1—C6—O2	2.4 (6)
C3^{i}—O1—C3—C2	142.1 (4)	C2—C1—C6—C5	0.1 (5)
C3^{i}—O1—C3—C4	−38.3 (3)	C7—C1—C6—C5	−177.8 (4)
C2—C3—C4—C5	0.5 (6)	C2—C1—C7—C8	−158.0 (4)
O1—C3—C4—C5	−179.1 (4)	C6—C1—C7—C8	19.9 (6)
C3—C4—C5—C6	−0.6 (6)	C1—C7—C8—C9	−47.3 (6)
C9—O2—C6—C5	−176.6 (4)	C6—O2—C9—C8	−31.5 (7)
C9—O2—C6—C1	3.2 (7)	C7—C8—C9—O2	53.2 (7)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8b···*Cg*1^{ii}	0.97	2.91	3.856 (6)	167

Symmetry code: (ii) *x*+1/4, −*y*+3/4, *z*−1/4.