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## Structure Reports

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## 2,3-Bis(2-chlorobenzyl)naphthalene-1,4-dione

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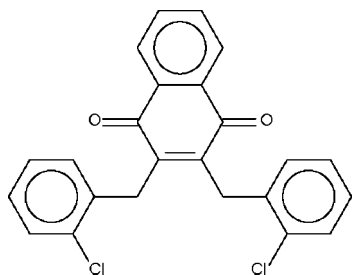
Received 21 November 2007; accepted 21 December 2007

Key indicators: single-crystal X-ray study;  $T = 295$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.045;  $wR$  factor = 0.131; data-to-parameter ratio = 17.3.

The title disubstituted naphthalene-1,4-dione,  $\text{C}_{24}\text{H}_{16}\text{Cl}_2\text{O}_2$ , has the two chlorobenzyl substituents related by a non-crystallographic twofold rotation axis, generating a chiral conformation; both enantiomers are present. The two chlorobenzene rings are nearly perpendicular to the fused ring system, making angles of  $88.8$  (1) and  $77.5$  (1)° with it.

### Related literature

2,3-Bis(2-chlorobenzyl)naphthalene-1,4-dione was the unexpected product in the attempted synthesis of 2-(2-chlorobenzyl)naphthalene-1,4-dione by a free-radical alkylation (see Boehm *et al.*, 1981; Chen *et al.*, 2005; Tsai *et al.*, 2001). Although the title dichloro compound has not been reported, 2,3-dibenzyl-naphthalene-1,4-dione has been known for a long time (Baxter & Sanders, 1974; Chen *et al.*, 2005; Oettmeier *et al.*, 1986; Sharma & Torrsell, 1978; Yamago *et al.*, 2002). This class of compounds exhibits antitumour activity (Driscoll, 1974; Driscoll *et al.*, 1974).



### Experimental

#### Crystal data

$\text{C}_{24}\text{H}_{16}\text{Cl}_2\text{O}_2$   
 $M_r = 407.27$   
 Monoclinic,  $P2_1/n$   
 $a = 9.998$  (1) Å  
 $b = 10.272$  (1) Å  
 $c = 18.804$  (2) Å  
 $\beta = 99.071$  (1)°  
 $V = 1907.1$  (3) Å<sup>3</sup>  
 $Z = 4$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.36$  mm<sup>-1</sup>  
 $T = 295$  (2) K  
 $0.5 \times 0.5 \times 0.5$  mm

#### Data collection

Bruker SMART area-detector diffractometer  
 Absorption correction: none  
 12440 measured reflections  
 4366 independent reflections  
 2921 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.035$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$   
 $wR(F^2) = 0.131$   
 $S = 1.02$   
 4366 reflections  
 253 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.18$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.42$  e Å<sup>-3</sup>

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: OB2105).

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**supplementary materials**

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## 2,3-Bis(2-chlorobenzyl)naphthalene-1,4-dione

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### Experimental

The title compound (I) was synthesized by using a modification of the procedure by Boehm *et al.* (1981). 1,4-Naphthoquinone (4 g, 25.3 mmol), 2-(2-chlorophenyl)acetic acid (8.60 g, 50.6 mmol), and silver nitrate (2.13 g, 12.6 mmol) in acetonitrile (60 ml) were heated to 353 K. A solution of ammonium peroxydisulfate in 30 ml water was added dropwise within 30 min. The mixture was then refluxed for another 2 h. The solution was concentrated to give a brown solid. The crude product was purified by column chromatography (silica gel, 1:2 dichloromethane:petroleum ether) to give yellow solid in 40% yield. Single crystals of (I) were obtained by slow evaporation of a 1:2 dichloromethane:petroleum ether (50 ml) solution.

### Figures

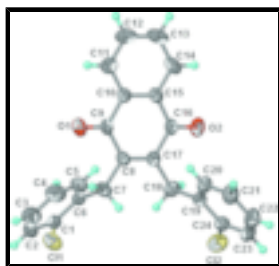


Fig. 1. The molecular structure of (I) with 50% probability ellipsoids for the non-H atoms.

## 2,3-Bis(2-chlorobenzyl)naphthalene-1,4-dione

### Crystal data

$C_{24}H_{16}Cl_2O_2$

$M_r = 407.27$

Monoclinic,  $P2_1/n$

Hall symbol:  $-P\ 2_1/n$

$a = 9.998$  (1) Å

$b = 10.272$  (1) Å

$c = 18.804$  (2) Å

$\beta = 99.071$  (1)°

$V = 1907.1$  (3) Å<sup>3</sup>

$Z = 4$

$F_{000} = 840$

$D_x = 1.418$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation

$\lambda = 0.71073$  Å

Cell parameters from 6278 reflections

$\theta = 2.2$ – $27.5$ °

$\mu = 0.36$  mm<sup>-1</sup>

$T = 295$  (2) K

Block, yellow

$0.5 \times 0.5 \times 0.5$  mm

### Data collection

Bruker SMART area-detector

2921 reflections with  $I > 2\sigma(I)$

# supplementary materials

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diffractometer

Radiation source: medium-focus sealed tube

Monochromator: graphite

$T = 295(2)$  K

$\varphi$  and  $\omega$  scans

Absorption correction: none

12440 measured reflections

4366 independent reflections

$$R_{\text{int}} = 0.035$$

$$\theta_{\text{max}} = 27.5^\circ$$

$$\theta_{\text{min}} = 2.2^\circ$$

$$h = -12 \rightarrow 12$$

$$k = -13 \rightarrow 13$$

$$l = -24 \rightarrow 11$$

## Refinement

Refinement on  $F^2$

Least-squares matrix: full

$$R[F^2 > 2\sigma(F^2)] = 0.044$$

$$wR(F^2) = 0.131$$

$$S = 1.02$$

4366 reflections

253 parameters

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

$$w = 1/[\sigma^2(F_o^2) + (0.0628P)^2 + 0.3578P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\text{max}} = 0.001$$

$$\Delta\rho_{\text{max}} = 0.18 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\text{min}} = -0.42 \text{ e } \text{\AA}^{-3}$$

Extinction correction: none

## Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	0.53007 (6)	0.55702 (6)	0.66580 (4)	0.0779 (2)
C12	-0.07641 (7)	0.55387 (6)	0.39047 (4)	0.0856 (2)
O1	0.48173 (15)	1.00324 (15)	0.60292 (8)	0.0693 (4)
O2	0.03492 (16)	1.00716 (16)	0.40921 (9)	0.0763 (5)
C1	0.39509 (19)	0.64277 (17)	0.69086 (10)	0.0508 (5)
C2	0.3610 (3)	0.6185 (2)	0.75798 (11)	0.0707 (7)
H2	0.4108	0.5589	0.7887	0.085*
C3	0.2548 (3)	0.6819 (2)	0.77899 (13)	0.0821 (8)
H3	0.2317	0.6658	0.8242	0.099*
C4	0.1817 (3)	0.7694 (2)	0.73391 (13)	0.0743 (7)
H4	0.1081	0.8118	0.7481	0.089*
C5	0.2169 (2)	0.79478 (19)	0.66737 (10)	0.0550 (5)
H5	0.1667	0.8550	0.6373	0.066*
C6	0.32481 (18)	0.73306 (16)	0.64433 (9)	0.0438 (4)
C7	0.3679 (2)	0.76083 (18)	0.57207 (10)	0.0524 (5)
H7A	0.4658	0.7688	0.5789	0.063*
H7B	0.3431	0.6871	0.5405	0.063*
C8	0.30674 (17)	0.88202 (17)	0.53547 (9)	0.0426 (4)
C9	0.37930 (17)	1.00544 (18)	0.55815 (9)	0.0438 (4)
C10	0.32835 (17)	1.12940 (16)	0.52474 (9)	0.0422 (4)
C11	0.39225 (19)	1.24608 (19)	0.54606 (11)	0.0555 (5)
H11	0.4650	1.2469	0.5836	0.067*

C12	0.3487 (2)	1.3603 (2)	0.51213 (14)	0.0698 (6)
H12	0.3920	1.4382	0.5266	0.084*
C13	0.2408 (2)	1.3596 (2)	0.45657 (15)	0.0745 (7)
H13	0.2131	1.4368	0.4330	0.089*
C14	0.1739 (2)	1.2458 (2)	0.43582 (12)	0.0618 (5)
H14	0.1002	1.2465	0.3988	0.074*
C15	0.21608 (18)	1.12966 (17)	0.47002 (9)	0.0447 (4)
C16	0.14175 (18)	1.00724 (18)	0.45086 (9)	0.0476 (4)
C17	0.19512 (18)	0.88289 (17)	0.48466 (9)	0.0443 (4)
C18	0.1139 (2)	0.76407 (19)	0.45892 (11)	0.0566 (5)
H18A	0.0185	0.7835	0.4574	0.068*
H18B	0.1372	0.6946	0.4936	0.068*
C19	0.13550 (17)	0.71606 (17)	0.38532 (10)	0.0466 (4)
C20	0.23605 (19)	0.76496 (19)	0.35008 (10)	0.0542 (5)
H20	0.2938	0.8290	0.3724	0.065*
C21	0.2529 (2)	0.7209 (2)	0.28236 (12)	0.0675 (6)
H21	0.3211	0.7555	0.2597	0.081*
C22	0.1689 (3)	0.6263 (3)	0.24877 (13)	0.0762 (7)
H22	0.1804	0.5965	0.2034	0.091*
C23	0.0682 (2)	0.5759 (2)	0.28197 (13)	0.0694 (6)
H23	0.0108	0.5121	0.2592	0.083*
C24	0.05248 (19)	0.62039 (18)	0.34967 (11)	0.0553 (5)

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C11	0.0746 (4)	0.0631 (4)	0.0901 (5)	0.0227 (3)	-0.0056 (3)	0.0124 (3)
C12	0.0785 (4)	0.0736 (4)	0.1006 (5)	-0.0274 (3)	0.0012 (3)	0.0022 (3)
O1	0.0628 (9)	0.0722 (10)	0.0629 (9)	0.0011 (7)	-0.0211 (7)	0.0104 (7)
O2	0.0696 (9)	0.0745 (10)	0.0715 (10)	0.0136 (8)	-0.0305 (8)	-0.0104 (8)
C1	0.0583 (11)	0.0404 (9)	0.0490 (10)	-0.0050 (8)	-0.0064 (9)	0.0029 (8)
C2	0.1079 (19)	0.0510 (12)	0.0466 (12)	-0.0113 (12)	-0.0080 (12)	0.0110 (10)
C3	0.134 (2)	0.0653 (15)	0.0519 (13)	-0.0148 (16)	0.0309 (14)	0.0074 (11)
C4	0.0997 (18)	0.0636 (14)	0.0682 (14)	-0.0003 (13)	0.0402 (13)	0.0038 (11)
C5	0.0640 (12)	0.0510 (11)	0.0520 (11)	0.0054 (9)	0.0157 (9)	0.0062 (9)
C6	0.0519 (10)	0.0376 (8)	0.0399 (9)	-0.0032 (7)	0.0012 (7)	0.0009 (7)
C7	0.0638 (11)	0.0465 (10)	0.0479 (10)	0.0142 (9)	0.0118 (9)	0.0056 (8)
C8	0.0507 (10)	0.0447 (10)	0.0333 (8)	0.0093 (8)	0.0100 (7)	0.0031 (7)
C9	0.0442 (9)	0.0532 (10)	0.0332 (8)	0.0052 (8)	0.0037 (7)	0.0011 (7)
C10	0.0453 (9)	0.0439 (9)	0.0385 (9)	0.0045 (7)	0.0102 (7)	-0.0013 (7)
C11	0.0516 (10)	0.0549 (11)	0.0603 (12)	-0.0011 (9)	0.0097 (9)	-0.0046 (9)
C12	0.0709 (14)	0.0443 (11)	0.0967 (18)	-0.0021 (10)	0.0214 (13)	-0.0018 (11)
C13	0.0813 (15)	0.0472 (12)	0.0973 (18)	0.0131 (11)	0.0211 (14)	0.0212 (12)
C14	0.0621 (12)	0.0606 (13)	0.0610 (12)	0.0173 (10)	0.0050 (10)	0.0161 (10)
C15	0.0478 (9)	0.0467 (10)	0.0397 (9)	0.0097 (8)	0.0073 (7)	0.0034 (7)
C16	0.0505 (10)	0.0539 (11)	0.0359 (9)	0.0089 (8)	-0.0009 (8)	-0.0028 (8)
C17	0.0509 (10)	0.0465 (10)	0.0365 (9)	0.0017 (8)	0.0097 (7)	-0.0027 (7)
C18	0.0641 (12)	0.0530 (11)	0.0531 (11)	-0.0076 (9)	0.0101 (9)	-0.0038 (9)

## supplementary materials

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C19	0.0468 (9)	0.0418 (9)	0.0476 (10)	0.0059 (8)	-0.0036 (8)	-0.0018 (8)
C20	0.0497 (10)	0.0586 (11)	0.0514 (11)	0.0030 (9)	-0.0007 (9)	-0.0095 (9)
C21	0.0657 (13)	0.0817 (16)	0.0549 (12)	0.0107 (12)	0.0092 (10)	-0.0092 (11)
C22	0.0836 (16)	0.0850 (17)	0.0555 (13)	0.0203 (14)	-0.0027 (12)	-0.0212 (12)
C23	0.0766 (15)	0.0565 (12)	0.0658 (14)	0.0061 (11)	-0.0176 (12)	-0.0163 (11)
C24	0.0538 (11)	0.0436 (10)	0.0626 (12)	0.0045 (8)	-0.0089 (9)	-0.0003 (9)

### *Geometric parameters (Å, °)*

C11—C1	1.738 (2)	C11—H11	0.9300
C12—C24	1.740 (2)	C12—C13	1.379 (3)
O1—C9	1.219 (2)	C12—H12	0.9300
O2—C16	1.221 (2)	C13—C14	1.372 (3)
C1—C2	1.381 (3)	C13—H13	0.9300
C1—C6	1.388 (2)	C14—C15	1.389 (3)
C2—C3	1.357 (3)	C14—H14	0.9300
C2—H2	0.9300	C15—C16	1.476 (3)
C3—C4	1.365 (3)	C16—C17	1.488 (2)
C3—H3	0.9300	C17—C18	1.503 (3)
C4—C5	1.377 (3)	C18—C19	1.516 (3)
C4—H4	0.9300	C18—H18A	0.9700
C5—C6	1.379 (3)	C18—H18B	0.9700
C5—H5	0.9300	C19—C20	1.383 (3)
C6—C7	1.516 (2)	C19—C24	1.389 (2)
C7—C8	1.505 (2)	C20—C21	1.386 (3)
C7—H7A	0.9700	C20—H20	0.9300
C7—H7B	0.9700	C21—C22	1.371 (3)
C8—C17	1.350 (2)	C21—H21	0.9300
C8—C9	1.489 (3)	C22—C23	1.367 (3)
C9—C10	1.475 (2)	C22—H22	0.9300
C10—C11	1.387 (3)	C23—C24	1.385 (3)
C10—C15	1.398 (2)	C23—H23	0.9300
C11—C12	1.373 (3)		
C2—C1—C6	121.6 (2)	C14—C13—C12	120.6 (2)
C2—C1—C11	118.05 (16)	C14—C13—H13	119.7
C6—C1—C11	120.36 (15)	C12—C13—H13	119.7
C3—C2—C1	119.8 (2)	C13—C14—C15	120.1 (2)
C3—C2—H2	120.1	C13—C14—H14	120.0
C1—C2—H2	120.1	C15—C14—H14	120.0
C2—C3—C4	120.1 (2)	C14—C15—C10	119.49 (18)
C2—C3—H3	119.9	C14—C15—C16	120.95 (17)
C4—C3—H3	119.9	C10—C15—C16	119.53 (15)
C3—C4—C5	120.0 (2)	O2—C16—C15	120.86 (17)
C3—C4—H4	120.0	O2—C16—C17	119.48 (18)
C5—C4—H4	120.0	C15—C16—C17	119.62 (15)
C4—C5—C6	121.55 (19)	C8—C17—C16	120.59 (16)
C4—C5—H5	119.2	C8—C17—C18	124.53 (16)
C6—C5—H5	119.2	C16—C17—C18	114.86 (16)
C5—C6—C1	116.92 (17)	C17—C18—C19	114.23 (16)

C5—C6—C7	122.83 (16)	C17—C18—H18A	108.7
C1—C6—C7	120.25 (16)	C19—C18—H18A	108.7
C8—C7—C6	114.41 (15)	C17—C18—H18B	108.7
C8—C7—H7A	108.7	C19—C18—H18B	108.7
C6—C7—H7A	108.7	H18A—C18—H18B	107.6
C8—C7—H7B	108.7	C20—C19—C24	116.75 (18)
C6—C7—H7B	108.7	C20—C19—C18	122.56 (16)
H7A—C7—H7B	107.6	C24—C19—C18	120.68 (18)
C17—C8—C9	120.54 (15)	C19—C20—C21	121.61 (19)
C17—C8—C7	124.07 (17)	C19—C20—H20	119.2
C9—C8—C7	115.39 (15)	C21—C20—H20	119.2
O1—C9—C10	120.42 (17)	C22—C21—C20	120.0 (2)
O1—C9—C8	120.02 (17)	C22—C21—H21	120.0
C10—C9—C8	119.54 (15)	C20—C21—H21	120.0
C11—C10—C15	119.39 (16)	C23—C22—C21	120.0 (2)
C11—C10—C9	120.69 (16)	C23—C22—H22	120.0
C15—C10—C9	119.90 (15)	C21—C22—H22	120.0
C12—C11—C10	120.40 (19)	C22—C23—C24	119.5 (2)
C12—C11—H11	119.8	C22—C23—H23	120.2
C10—C11—H11	119.8	C24—C23—H23	120.2
C11—C12—C13	120.0 (2)	C19—C24—C23	122.1 (2)
C11—C12—H12	120.0	C19—C24—C12	119.53 (16)
C13—C12—H12	120.0	C23—C24—C12	118.37 (16)
C6—C1—C2—C3	1.4 (3)	C9—C10—C15—C14	-176.07 (17)
C11—C1—C2—C3	-178.88 (18)	C11—C10—C15—C16	-175.53 (16)
C1—C2—C3—C4	0.0 (4)	C9—C10—C15—C16	5.7 (2)
C2—C3—C4—C5	-0.9 (4)	C14—C15—C16—O2	-6.5 (3)
C3—C4—C5—C6	0.4 (4)	C10—C15—C16—O2	171.68 (18)
C4—C5—C6—C1	0.9 (3)	C14—C15—C16—C17	175.61 (17)
C4—C5—C6—C7	-178.7 (2)	C10—C15—C16—C17	-6.2 (3)
C2—C1—C6—C5	-1.8 (3)	C9—C8—C17—C16	-0.6 (2)
C11—C1—C6—C5	178.44 (14)	C7—C8—C17—C16	-179.65 (16)
C2—C1—C6—C7	177.85 (18)	C9—C8—C17—C18	-179.10 (16)
C11—C1—C6—C7	-1.9 (2)	C7—C8—C17—C18	1.8 (3)
C5—C6—C7—C8	14.0 (3)	O2—C16—C17—C8	-174.32 (18)
C1—C6—C7—C8	-165.61 (16)	C15—C16—C17—C8	3.6 (3)
C6—C7—C8—C17	-96.4 (2)	O2—C16—C17—C18	4.4 (3)
C6—C7—C8—C9	84.5 (2)	C15—C16—C17—C18	-177.76 (16)
C17—C8—C9—O1	-178.37 (17)	C8—C17—C18—C19	-104.7 (2)
C7—C8—C9—O1	0.8 (2)	C16—C17—C18—C19	76.7 (2)
C17—C8—C9—C10	0.1 (2)	C17—C18—C19—C20	9.4 (3)
C7—C8—C9—C10	179.22 (15)	C17—C18—C19—C24	-169.87 (16)
O1—C9—C10—C11	-3.0 (3)	C24—C19—C20—C21	0.2 (3)
C8—C9—C10—C11	178.54 (16)	C18—C19—C20—C21	-179.10 (18)
O1—C9—C10—C15	175.71 (17)	C19—C20—C21—C22	-0.2 (3)
C8—C9—C10—C15	-2.7 (2)	C20—C21—C22—C23	0.3 (3)
C15—C10—C11—C12	-2.2 (3)	C21—C22—C23—C24	-0.4 (3)
C9—C10—C11—C12	176.55 (18)	C20—C19—C24—C23	-0.3 (3)
C10—C11—C12—C13	0.1 (3)	C18—C19—C24—C23	179.01 (18)

## supplementary materials

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C11—C12—C13—C14	1.6 (4)	C20—C19—C24—C12	179.97 (14)
C12—C13—C14—C15	-1.1 (3)	C18—C19—C24—C12	-0.7 (2)
C13—C14—C15—C10	-1.1 (3)	C22—C23—C24—C19	0.4 (3)
C13—C14—C15—C16	177.1 (2)	C22—C23—C24—C12	-179.87 (17)
C11—C10—C15—C14	2.7 (3)		

Fig. 1

