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2,3-Dibromo-3-(4-bromo-6-methoxy-2-naphthyl)-1-(4-methoxyphenyl)propan-1-one

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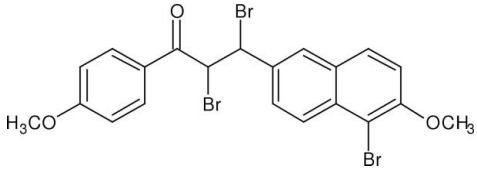
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.010 Å; R factor = 0.069; wR factor = 0.174; data-to-parameter ratio = 14.4.

In the title compound, $C_{21}H_{17}Br_3O_3$, the two aromatic residues are almost coplanar with one another [dihedral angle = 9.92 (6)°]. The two Br atoms at the Csp^3 atoms are in a *trans* conformation.

Related literature

For background, see: Yathirajan *et al.* (2007).



Experimental

Crystal data

$C_{21}H_{17}Br_3O_3$
M_r = 557.08
Monoclinic, $P2_1/c$
a = 7.4989 (5) Å
b = 20.3640 (16) Å
c = 13.6083 (8) Å
β = 104.844 (5)°
V = 2008.7 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 6.04 mm^{-1}
T = 173 (2) K
0.32 × 0.29 × 0.12 mm

Data collection

Stoe IPDS II two-circle diffractometer
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
T_{min} = 0.168, T_{max} = 0.471
23062 measured reflections
3531 independent reflections
3048 reflections with $I > 2\sigma(I)$
R_{int} = 0.064

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.069
$wR(F^2)$ = 0.174
S = 1.08
3531 reflections
246 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 2.68 e Å^{-3}
$\Delta\rho_{min}$ = −2.00 e Å^{-3}

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97*.

ANM thanks the University of Mysore for permission to carry out research work.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2503).

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supplementary materials

Acta Cryst. (2007). E**63**, o3755 [**doi:10.1107/S1600536807038883**]

2,3-Dibromo-3-(4-bromo-6-methoxy-2-naphthyl)-1-(4-methoxyphenyl)propan-1-one

B. K. Sarojini, B. Narayana, H. S. Yathirajan, A. N. Mayekar and M. Bolte

Comment

As part of our ongoing studies of chalcones (Yathirajan *et al.*, 2007), the title compound, (I), was serendipitiously prepapred by the bromination of ((2E)-3-(6-methoxy-2-naphthyl)-1-(4-methoxyphenyl)prop-2-en-1-one. As well the intended bromination as the central double bond, an excess of bromine also brominated of one of the naphthalene C atoms.

In the structure of (I) (Fig. 1) the two aromatic residues are almost coplanar to each other [dihedral angle 9.92 (6)°]. The two Br atoms at the sp^3 C atoms are *trans* with respect to each other [Br1—C1—C2—Br2 = 177.6 (3)°].

Experimental

(2E)-3-(6-Methoxy-2-naphthyl)-1-(4-methoxyphenyl)prop-2-en-1-one (3.18 g, 0.01 mol) was treated with bromine in acetic acid (30%) until the orange colour of the solution persisted. After stirring for half an hour, the contents were poured on to crushed ice. The resulting solid mass was collected by filtration. The compound was dried and recrystallized from ethanol. Colourless plates of (I) were obtained from acetone by slow evaporation (yield 78%; m.p.: 453–455 K). Analysis for $C_{21}H_{17}Br_3O_3$: found (calculated): C: 45.20 (45.28); H: 3.01% (3.08%).

Refinement

The H atoms were found in a difference map and were relocated to idealized positions (C—H = 0.95–1.00 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C). The methyl groups were allowed to rotate but not to tip. The highest difference peak is 0.96Å from Br2.

Figures

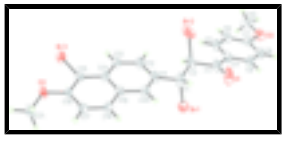


Fig. 1. View of the molecular structure of (I) with displacement ellipsoids drawn at the 50% probability level (arbitrary spheres for the H atoms).

2,3-Dibromo-3-(4-bromo-6-methoxy-2-naphthyl)-1-(4-methoxyphenyl)propan-1-one

Crystal data

$C_{21}H_{17}Br_3O_3$	F_{000} = 1088
M_r = 557.08	D_x = 1.842 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 24485 reflections

a = 7.4989 (5) Å	θ = 3.5–24.9º
b = 20.3640 (16) Å	μ = 6.04 mm^{-1}
c = 13.6083 (8) Å	T = 173 (2) K
β = 104.844 (5)º	Plate, colourless
V = 2008.7 (2) $Å^3$	0.32 × 0.29 × 0.12 mm
Z = 4	

Data collection

Stoe IPDS II two-circle diffractometer	3531 independent reflections
Radiation source: fine-focus sealed tube	3048 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.064
T = 173(2) K	θ_{max} = 25.0º
ω scans	θ_{min} = 3.6º
Absorption correction: multi-scan (MULABS; Spek, 2003; Blessing, 1995)	h = −8→8
T_{min} = 0.168, T_{max} = 0.471	k = −24→24
23062 measured reflections	l = −15→16

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.069	H-atom parameters constrained
$wR(F^2)$ = 0.174	$w = 1/[\sigma^2(F_o^2) + (0.0826P)^2 + 13.7141P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.08	$(\Delta/\sigma)_{max} < 0.001$
3531 reflections	$\Delta\rho_{max}$ = 2.68 e $Å^{-3}$
246 parameters	$\Delta\rho_{min}$ = −2.00 e $Å^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2$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}
Br1	0.04941 (12)	0.47310 (4)	0.87074 (7)	0.0472 (3)
Br2	0.58929 (13)	0.42001 (5)	0.78473 (10)	0.0648 (4)
Br3	0.09601 (11)	0.63997 (3)	0.36243 (6)	0.0349 (2)
O1	0.4516 (10)	0.4089 (3)	1.0061 (5)	0.0581 (19)
O2	0.3380 (9)	0.1035 (2)	0.9474 (5)	0.0475 (15)
O3	0.0774 (8)	0.7748 (2)	0.4366 (4)	0.0349 (12)
C1	0.2970 (13)	0.4863 (4)	0.8360 (6)	0.044 (2)
H1	0.3871	0.5068	0.8952	0.053*
C2	0.3487 (11)	0.4177 (4)	0.8256 (6)	0.0387 (19)
H2	0.2513	0.3947	0.7728	0.046*
C3	0.3941 (11)	0.3787 (3)	0.9265 (6)	0.0359 (18)
C4	0.3719 (10)	0.3062 (3)	0.9250 (6)	0.0322 (16)
C5	0.3182 (11)	0.2688 (4)	0.8372 (6)	0.0374 (18)
H5	0.2901	0.2902	0.7731	0.045*
C6	0.3046 (11)	0.2016 (4)	0.8407 (7)	0.0397 (19)
H6	0.2688	0.1770	0.7794	0.048*
C7	0.3434 (10)	0.1697 (3)	0.9338 (7)	0.0344 (17)
C8	0.3954 (11)	0.2069 (4)	1.0235 (7)	0.0382 (18)
H8	0.4207	0.1856	1.0877	0.046*
C9	0.4098 (11)	0.2734 (4)	1.0188 (6)	0.0352 (17)
H9	0.4461	0.2980	1.0800	0.042*
C10	0.2919 (15)	0.0637 (4)	0.8566 (8)	0.054 (3)
H10A	0.1671	0.0746	0.8166	0.081*
H10B	0.2969	0.0171	0.8755	0.081*
H10C	0.3802	0.0722	0.8161	0.081*
C11	0.2671 (11)	0.5312 (3)	0.7452 (6)	0.0336 (17)
C12	0.2265 (11)	0.5082 (3)	0.6424 (6)	0.0323 (16)
H12	0.2283	0.4624	0.6291	0.039*
C13	0.1856 (10)	0.5515 (3)	0.5637 (6)	0.0301 (15)
H13	0.1582	0.5351	0.4961	0.036*
C14	0.1824 (9)	0.6204 (3)	0.5791 (5)	0.0250 (14)
C15	0.1381 (9)	0.6679 (3)	0.4997 (5)	0.0248 (14)
C16	0.1246 (9)	0.7336 (3)	0.5192 (5)	0.0266 (14)
C17	0.1612 (10)	0.7555 (3)	0.6202 (6)	0.0307 (16)
H17	0.1525	0.8009	0.6339	0.037*
C18	0.2090 (11)	0.7121 (3)	0.6987 (6)	0.0319 (16)
H18	0.2342	0.7280	0.7666	0.038*
C19	0.2223 (10)	0.6436 (3)	0.6815 (5)	0.0266 (14)
C20	0.2677 (10)	0.5978 (3)	0.7623 (5)	0.0290 (15)
H20	0.2996	0.6135	0.8303	0.035*
C21	0.0410 (12)	0.8423 (3)	0.4539 (7)	0.0372 (18)
H21A	−0.0518	0.8449	0.4930	0.056*
H21B	−0.0051	0.8646	0.3886	0.056*
H21C	0.1551	0.8635	0.4921	0.056*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0581 (5)	0.0379 (5)	0.0542 (6)	0.0082 (4)	0.0299 (4)	0.0133 (4)
Br2	0.0492 (5)	0.0560 (6)	0.0967 (9)	0.0227 (4)	0.0325 (5)	0.0411 (6)
Br3	0.0562 (5)	0.0206 (4)	0.0267 (4)	−0.0027 (3)	0.0083 (3)	−0.0007 (3)
O1	0.096 (5)	0.022 (3)	0.038 (3)	−0.014 (3)	−0.017 (3)	0.006 (3)
O2	0.068 (4)	0.011 (2)	0.067 (4)	−0.001 (2)	0.024 (3)	0.002 (3)
O3	0.060 (3)	0.012 (2)	0.032 (3)	0.005 (2)	0.010 (2)	0.005 (2)
C1	0.076 (6)	0.017 (4)	0.033 (4)	−0.009 (4)	0.001 (4)	0.004 (3)
C2	0.048 (4)	0.019 (4)	0.039 (5)	−0.001 (3)	−0.006 (3)	0.007 (3)
C3	0.046 (4)	0.017 (3)	0.036 (4)	−0.003 (3)	−0.004 (3)	0.003 (3)
C4	0.034 (4)	0.019 (4)	0.040 (4)	−0.003 (3)	0.004 (3)	0.008 (3)
C5	0.049 (4)	0.019 (4)	0.040 (4)	−0.002 (3)	0.004 (3)	0.007 (3)
C6	0.049 (5)	0.018 (4)	0.049 (5)	−0.006 (3)	0.008 (4)	−0.004 (3)
C7	0.038 (4)	0.016 (3)	0.052 (5)	0.000 (3)	0.016 (3)	0.003 (3)
C8	0.051 (5)	0.021 (4)	0.042 (5)	0.002 (3)	0.011 (4)	0.009 (3)
C9	0.047 (4)	0.026 (4)	0.030 (4)	0.002 (3)	0.005 (3)	0.003 (3)
C10	0.088 (7)	0.013 (4)	0.074 (7)	−0.009 (4)	0.046 (6)	−0.009 (4)
C11	0.052 (4)	0.014 (3)	0.030 (4)	−0.004 (3)	0.002 (3)	0.005 (3)
C12	0.048 (4)	0.010 (3)	0.036 (4)	−0.003 (3)	0.004 (3)	0.000 (3)
C13	0.039 (4)	0.023 (4)	0.028 (4)	−0.005 (3)	0.006 (3)	−0.001 (3)
C14	0.029 (3)	0.016 (3)	0.029 (4)	−0.004 (2)	0.007 (3)	0.000 (3)
C15	0.034 (3)	0.014 (3)	0.027 (4)	−0.004 (3)	0.007 (3)	−0.001 (3)
C16	0.035 (3)	0.016 (3)	0.030 (4)	−0.001 (3)	0.009 (3)	0.007 (3)
C17	0.048 (4)	0.011 (3)	0.036 (4)	0.001 (3)	0.016 (3)	−0.002 (3)
C18	0.056 (5)	0.016 (3)	0.025 (4)	−0.001 (3)	0.012 (3)	−0.004 (3)
C19	0.036 (4)	0.015 (3)	0.029 (4)	−0.004 (3)	0.009 (3)	−0.001 (3)
C20	0.045 (4)	0.017 (3)	0.024 (4)	−0.004 (3)	0.008 (3)	−0.001 (3)
C21	0.055 (5)	0.014 (3)	0.041 (5)	0.005 (3)	0.010 (4)	0.007 (3)

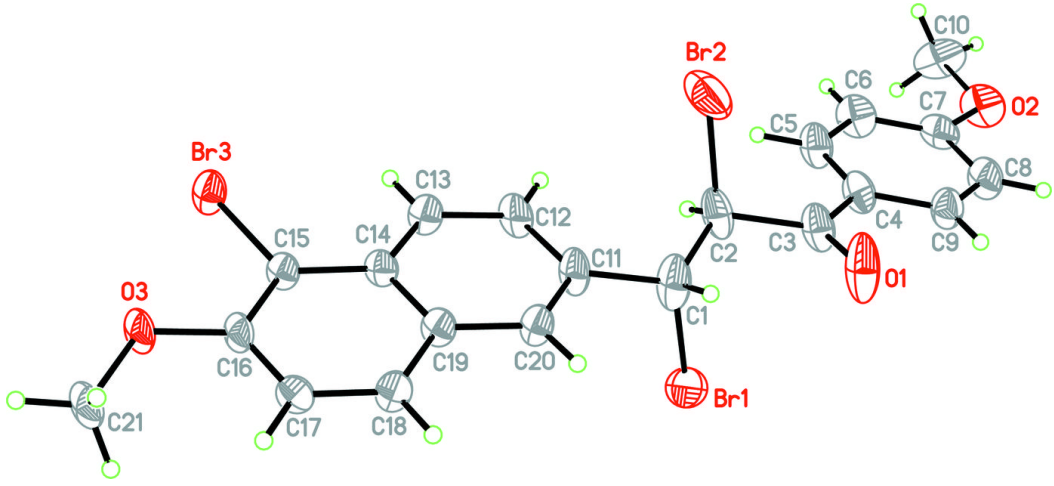
Geometric parameters (Å, °)

Br1—C1	2.048 (10)	C9—H9	0.9500
Br2—C2	2.020 (9)	C10—H10A	0.9800
Br3—C15	1.900 (7)	C10—H10B	0.9800
O1—C3	1.223 (10)	C10—H10C	0.9800
O2—C7	1.363 (8)	C11—C20	1.376 (9)
O2—C10	1.444 (11)	C11—C12	1.433 (11)
O3—C16	1.374 (8)	C12—C13	1.360 (10)
O3—C21	1.432 (8)	C12—H12	0.9500
C1—C2	1.466 (11)	C13—C14	1.420 (10)
C1—C11	1.507 (10)	C13—H13	0.9500
C1—H1	1.0000	C14—C15	1.424 (10)
C2—C3	1.547 (11)	C14—C19	1.430 (10)
C2—H2	1.0000	C15—C16	1.374 (9)
C3—C4	1.485 (10)	C16—C17	1.403 (10)
C4—C5	1.387 (11)	C17—C18	1.362 (10)

C4—C9	1.403 (11)	C17—H17	0.9500
C5—C6	1.374 (10)	C18—C19	1.422 (9)
C5—H5	0.9500	C18—H18	0.9500
C6—C7	1.386 (12)	C19—C20	1.416 (10)
C6—H6	0.9500	C20—H20	0.9500
C7—C8	1.405 (12)	C21—H21A	0.9800
C8—C9	1.360 (10)	C21—H21B	0.9800
C8—H8	0.9500	C21—H21C	0.9800
C7—O2—C10	116.6 (7)	H10A—C10—H10C	109.5
C16—O3—C21	118.1 (6)	H10B—C10—H10C	109.5
C2—C1—C11	119.1 (8)	C20—C11—C12	118.5 (6)
C2—C1—Br1	100.1 (6)	C20—C11—C1	117.8 (7)
C11—C1—Br1	108.4 (6)	C12—C11—C1	123.5 (6)
C2—C1—H1	109.6	C13—C12—C11	120.4 (6)
C11—C1—H1	109.6	C13—C12—H12	119.8
Br1—C1—H1	109.6	C11—C12—H12	119.8
C1—C2—C3	114.0 (7)	C12—C13—C14	122.2 (7)
C1—C2—Br2	106.3 (6)	C12—C13—H13	118.9
C3—C2—Br2	104.3 (5)	C14—C13—H13	118.9
C1—C2—H2	110.6	C13—C14—C15	124.6 (6)
C3—C2—H2	110.6	C13—C14—C19	117.6 (6)
Br2—C2—H2	110.6	C15—C14—C19	117.7 (6)
O1—C3—C4	121.6 (7)	C16—C15—C14	122.0 (6)
O1—C3—C2	118.4 (6)	C16—C15—Br3	118.7 (5)
C4—C3—C2	120.0 (7)	C14—C15—Br3	119.4 (5)
C5—C4—C9	117.9 (7)	C15—C16—O3	116.9 (6)
C5—C4—C3	124.4 (7)	C15—C16—C17	119.5 (6)
C9—C4—C3	117.7 (7)	O3—C16—C17	123.5 (6)
C6—C5—C4	121.7 (7)	C18—C17—C16	120.6 (6)
C6—C5—H5	119.1	C18—C17—H17	119.7
C4—C5—H5	119.1	C16—C17—H17	119.7
C5—C6—C7	119.8 (8)	C17—C18—C19	121.5 (7)
C5—C6—H6	120.1	C17—C18—H18	119.3
C7—C6—H6	120.1	C19—C18—H18	119.3
O2—C7—C6	125.5 (7)	C20—C19—C18	122.1 (6)
O2—C7—C8	115.3 (7)	C20—C19—C14	119.2 (6)
C6—C7—C8	119.2 (6)	C18—C19—C14	118.6 (6)
C9—C8—C7	120.2 (7)	C11—C20—C19	122.0 (7)
C9—C8—H8	119.9	C11—C20—H20	119.0
C7—C8—H8	119.9	C19—C20—H20	119.0
C8—C9—C4	121.1 (7)	O3—C21—H21A	109.5
C8—C9—H9	119.4	O3—C21—H21B	109.5
C4—C9—H9	119.4	H21A—C21—H21B	109.5
O2—C10—H10A	109.5	O3—C21—H21C	109.5
O2—C10—H10B	109.5	H21A—C21—H21C	109.5
H10A—C10—H10B	109.5	H21B—C21—H21C	109.5
O2—C10—H10C	109.5		
C11—C1—C2—C3	174.3 (7)	C20—C11—C12—C13	−1.5 (12)

Br1—C1—C2—C3	−68.0 (7)	C1—C11—C12—C13	174.4 (8)
C11—C1—C2—Br2	59.9 (9)	C11—C12—C13—C14	0.5 (12)
Br1—C1—C2—Br2	177.6 (3)	C12—C13—C14—C15	−179.1 (7)
C1—C2—C3—O1	−27.1 (12)	C12—C13—C14—C19	−0.6 (11)
Br2—C2—C3—O1	88.5 (9)	C13—C14—C15—C16	175.3 (7)
C1—C2—C3—C4	154.4 (8)	C19—C14—C15—C16	−3.2 (10)
Br2—C2—C3—C4	−90.0 (7)	C13—C14—C15—Br3	−4.6 (9)
O1—C3—C4—C5	−175.9 (9)	C19—C14—C15—Br3	176.9 (5)
C2—C3—C4—C5	2.5 (12)	C14—C15—C16—O3	−178.7 (6)
O1—C3—C4—C9	3.4 (12)	Br3—C15—C16—O3	1.2 (9)
C2—C3—C4—C9	−178.2 (7)	C14—C15—C16—C17	2.2 (10)
C9—C4—C5—C6	−1.0 (12)	Br3—C15—C16—C17	−177.9 (5)
C3—C4—C5—C6	178.3 (8)	C21—O3—C16—C15	172.8 (7)
C4—C5—C6—C7	0.6 (13)	C21—O3—C16—C17	−8.1 (10)
C10—O2—C7—C6	1.5 (11)	C15—C16—C17—C18	−0.3 (11)
C10—O2—C7—C8	−177.7 (7)	O3—C16—C17—C18	−179.4 (7)
C5—C6—C7—O2	−178.9 (8)	C16—C17—C18—C19	−0.5 (12)
C5—C6—C7—C8	0.3 (12)	C17—C18—C19—C20	−178.5 (7)
O2—C7—C8—C9	178.4 (7)	C17—C18—C19—C14	−0.6 (11)
C6—C7—C8—C9	−0.9 (12)	C13—C14—C19—C20	1.7 (10)
C7—C8—C9—C4	0.5 (13)	C15—C14—C19—C20	−179.7 (6)
C5—C4—C9—C8	0.4 (12)	C13—C14—C19—C18	−176.3 (7)
C3—C4—C9—C8	−179.0 (8)	C15—C14—C19—C18	2.3 (10)
C2—C1—C11—C20	−163.0 (8)	C12—C11—C20—C19	2.7 (12)
Br1—C1—C11—C20	83.7 (8)	C1—C11—C20—C19	−173.4 (7)
C2—C1—C11—C12	21.1 (13)	C18—C19—C20—C11	175.1 (7)
Br1—C1—C11—C12	−92.2 (9)	C14—C19—C20—C11	−2.8 (11)

Fig. 1


C10
Br2
C6
C7
O2
C5
C8
Br3
C13
C12
C15
C14
C4
C3
C2
C9
C11
O1
O3
C1
C16
C19
C20
Br1
C21
C17
C18