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2,10-Dibromo-6-isobutyl-6-methyldibenzo[*d*,*f*][1,3]dioxepine

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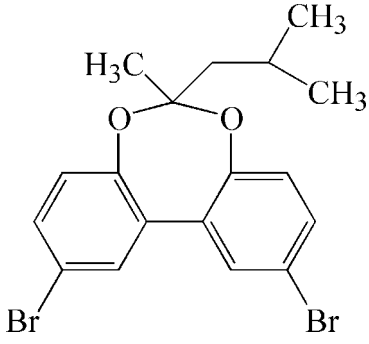
Key indicators: single-crystal X-ray study; T = 193 K; mean σ(C–C) = 0.003 Å; R factor = 0.029; wR factor = 0.052; data-to-parameter ratio = 19.4.

In the crystal structure of the title compound, $C_{18}H_{18}Br_2O_2$, the two benzene rings of the bridged biphenyl unit are twisted by 38.0 (1)°.

Related literature

For the synthesis of the title compound, see: Zhang *et al.* (2003).



Experimental

Crystal data

$C_{18}H_{18}Br_2O_2$
M_r = 426.14
Monoclinic, $P2_1/c$
a = 8.3372 (9) Å
b = 19.362 (2) Å
c = 10.8066 (16) Å
β = 100.803 (5)°
V = 1713.5 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 4.74 mm^{-1}
T = 193 (2) K
0.13 × 0.12 × 0.12 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.587, T_{max} = 0.607
(expected range = 0.547–0.567)
7425 measured reflections
3924 independent reflections
2495 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.052
S = 0.85
3924 reflections
202 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.51 e Å^{-3}

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC and Rigaku, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* .

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NC2099).

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2,10-Dibromo-6-isobutyl-6-methyldibenzo[*d*,*f*][1,3]dioxepine

Hai-Quan Zhang, Bao-Li, Guang-Di Yang and Yu-Guang Ma

S1. Comment

Recently we have reported on the synthesis of the title compound 2,10-Dibromo -6-isobutyl-6-methyl-Dibenzo [d,f][1,3] Dioxepine (Zhang *et al.* 2003). Herein we present the crysal structure of this compound. In the crystal structure of the title compound the two benzene rings of the bridged biphenyl unit are twisted by 38.0 (1)° and the 7-membered ring is in a boat conformation.

S2. Experimental

The title compound was synthesized as described previously (Zhang *et al.* 2003). Single crystals were obtained by slow evaporation of the solvent from a methanol solution at room temperature.

S3. Refinement

All H atoms were positioned with idealized geometry and refined isotropic with U_{iso}(H) = 1.5U_{eq}(C) for methyl and U_{iso}(H) = 1.2U_{eq}(C)for all other H atoms using a riding model with C—H = 0.97 Å for methyl and C—H = 0.93 Å for all other H atoms.

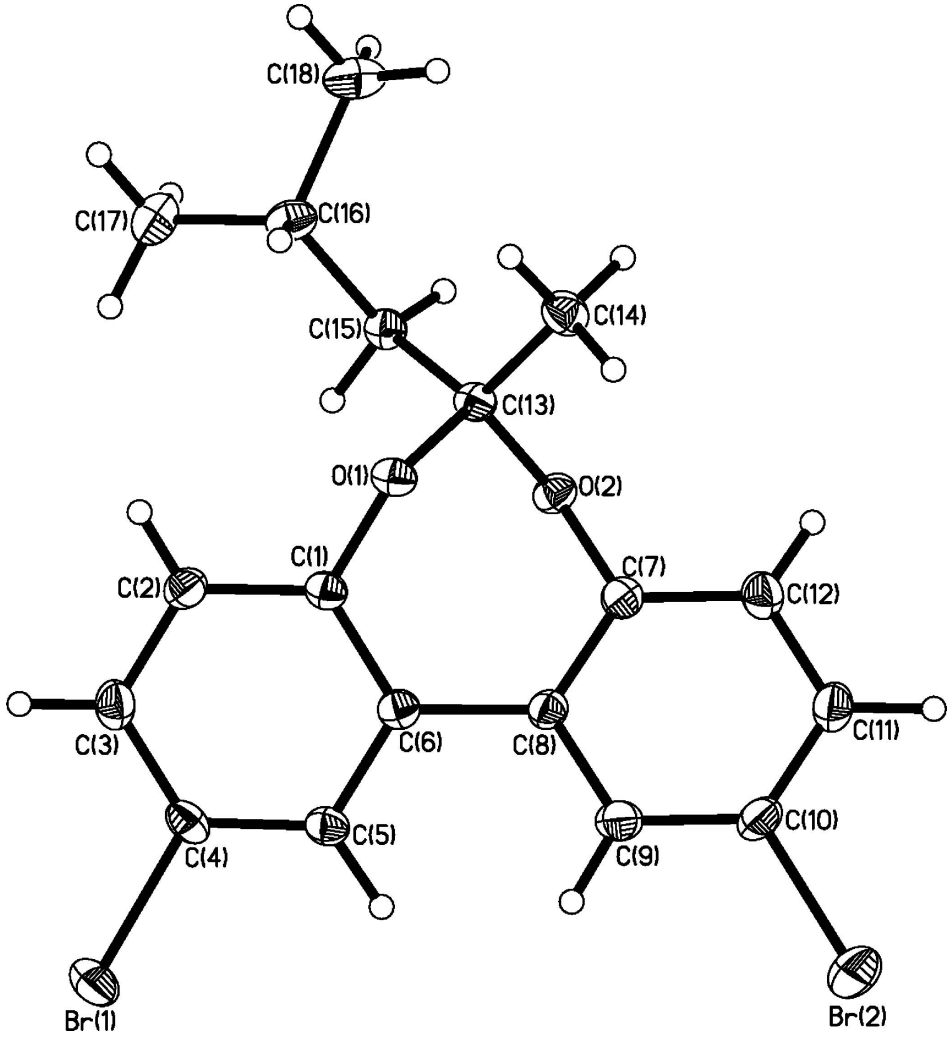

Figure 1

Crystal structure of the title compound with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii.

2,10-Dibromo-6-isobutyl-6-methyldibenzo[d,f][1,3]dioxepine

Crystal data

$C_{18}H_{18}Br_2O_2$
$M_r = 426.14$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 8.3372\ (9)$ Å
$b = 19.362\ (2)$ Å
$c = 10.8066\ (16)$ Å
$\beta = 100.803\ (5)°$
$V = 1713.5\ (4)$ Å^3
$Z = 4$
$F(000) = 848$
$D_x = 1.652$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 12167 reflections
$\theta = 2.1–54.9°$
$\mu = 4.74$ mm^{-1}
$T = 193$ K
Block, colorless
0.13 × 0.12 × 0.12 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.587, T_{max} = 0.607
7425 measured reflections
3924 independent reflections
2495 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 27.5°, θ_{min} = 2.1°
h = −10→10
k = −25→25
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.052
S = 0.85
3924 reflections
202 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
$w = 1/[\sigma^2(F_o^2) + (0.0206P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.51 e Å^{-3}

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	1.32353 (3)	0.596022 (13)	0.68875 (3)	0.03946 (9)
Br2	1.32322 (4)	0.994048 (14)	0.80855 (4)	0.06258 (12)
C1	1.0296 (3)	0.70180 (11)	0.9608 (2)	0.0219 (5)
C2	1.0540 (3)	0.63147 (11)	0.9678 (2)	0.0273 (6)
H2	1.0100	0.6051	1.0276	0.033*
C3	1.1424 (3)	0.59896 (11)	0.8880 (2)	0.0294 (6)
H3	1.1596	0.5504	0.8921	0.035*
C4	1.2052 (3)	0.63899 (12)	0.8022 (2)	0.0264 (6)
C5	1.1833 (3)	0.70927 (11)	0.7948 (2)	0.0238 (5)
H5	1.2283	0.7354	0.7353	0.029*
C6	1.0946 (3)	0.74206 (11)	0.8752 (2)	0.0220 (5)
C7	0.9182 (3)	0.84461 (11)	0.8840 (2)	0.0240 (5)
C8	1.0681 (3)	0.81720 (11)	0.8698 (2)	0.0226 (5)
C9	1.1879 (3)	0.86301 (12)	0.8475 (2)	0.0305 (6)
H9	1.2914	0.8460	0.8370	0.037*
C10	1.1573 (3)	0.93303 (12)	0.8403 (2)	0.0329 (7)

C11	1.0099 (3)	0.95998 (11)	0.8559 (2)	0.0333 (6)
H11	0.9914	1.0084	0.8517	0.040*
C12	0.8886 (3)	0.91495 (11)	0.8779 (2)	0.0297 (6)
H12	0.7856	0.9324	0.8887	0.036*
C13	0.7930 (3)	0.76216 (11)	1.0048 (2)	0.0231 (5)
C14	0.7645 (3)	0.81026 (11)	1.1085 (2)	0.0304 (6)
H14A	0.7726	0.7843	1.1873	0.046*
H14B	0.6554	0.8308	1.0860	0.046*
H14C	0.8469	0.8470	1.1195	0.046*
C15	0.6684 (3)	0.70529 (11)	0.9684 (2)	0.0239 (5)
H15A	0.7027	0.6767	0.9020	0.029*
H15B	0.5622	0.7267	0.9316	0.029*
C16	0.6434 (3)	0.65766 (12)	1.0774 (2)	0.0271 (6)
H16	0.7452	0.6592	1.1431	0.032*
C17	0.6163 (3)	0.58315 (12)	1.0329 (2)	0.0404 (7)
H17A	0.6030	0.5538	1.1041	0.061*
H17B	0.7106	0.5673	0.9984	0.061*
H17C	0.5177	0.5804	0.9674	0.061*
C18	0.5010 (3)	0.68121 (13)	1.1386 (2)	0.0414 (7)
H18A	0.3989	0.6784	1.0767	0.062*
H18B	0.5193	0.7290	1.1677	0.062*
H18C	0.4938	0.6512	1.2104	0.062*
O1	0.95321 (18)	0.73412 (7)	1.04861 (13)	0.0232 (4)
O2	0.78958 (17)	0.79979 (7)	0.88965 (14)	0.0243 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.03994 (16)	0.03630 (15)	0.04601 (17)	0.00641 (13)	0.01797 (13)	−0.00815 (14)
Br2	0.0675 (2)	0.03245 (16)	0.1003 (3)	−0.01841 (15)	0.0480 (2)	−0.00512 (17)
C1	0.0173 (13)	0.0274 (13)	0.0206 (13)	0.0013 (10)	0.0024 (11)	0.0006 (10)
C2	0.0242 (13)	0.0273 (13)	0.0309 (15)	0.0009 (11)	0.0066 (12)	0.0070 (11)
C3	0.0267 (13)	0.0213 (12)	0.0391 (16)	0.0033 (11)	0.0029 (12)	0.0019 (12)
C4	0.0170 (13)	0.0304 (13)	0.0316 (15)	0.0042 (10)	0.0039 (12)	−0.0069 (11)
C5	0.0198 (12)	0.0276 (13)	0.0243 (14)	0.0006 (10)	0.0050 (11)	0.0011 (11)
C6	0.0178 (13)	0.0247 (12)	0.0225 (13)	−0.0005 (10)	0.0016 (11)	−0.0007 (10)
C7	0.0244 (14)	0.0260 (13)	0.0212 (14)	−0.0015 (10)	0.0030 (11)	−0.0009 (10)
C8	0.0283 (13)	0.0207 (11)	0.0205 (13)	−0.0008 (10)	0.0083 (11)	−0.0004 (10)
C9	0.0306 (15)	0.0298 (14)	0.0338 (17)	−0.0028 (11)	0.0131 (14)	−0.0036 (11)
C10	0.0423 (16)	0.0252 (13)	0.0362 (17)	−0.0089 (12)	0.0203 (14)	−0.0007 (11)
C11	0.0458 (18)	0.0205 (12)	0.0354 (16)	0.0023 (12)	0.0122 (14)	0.0013 (11)
C12	0.0313 (14)	0.0279 (14)	0.0303 (15)	0.0064 (11)	0.0067 (12)	0.0034 (11)
C13	0.0245 (13)	0.0279 (12)	0.0167 (13)	0.0044 (11)	0.0032 (11)	0.0033 (10)
C14	0.0294 (14)	0.0320 (14)	0.0304 (16)	0.0018 (12)	0.0073 (12)	−0.0052 (12)
C15	0.0207 (13)	0.0280 (12)	0.0222 (13)	0.0017 (10)	0.0020 (11)	−0.0004 (11)
C16	0.0215 (13)	0.0352 (14)	0.0237 (14)	−0.0039 (11)	0.0025 (11)	0.0023 (11)
C17	0.0423 (17)	0.0333 (15)	0.0468 (18)	−0.0035 (13)	0.0116 (14)	0.0074 (13)
C18	0.0331 (16)	0.0539 (17)	0.0408 (18)	−0.0094 (13)	0.0163 (14)	−0.0011 (14)

O1	0.0193 (9)	0.0305 (9)	0.0197 (9)	0.0023 (7)	0.0033 (7)	0.0001 (7)
O2	0.0226 (9)	0.0266 (9)	0.0230 (9)	−0.0009 (7)	0.0028 (8)	0.0043 (7)

Geometric parameters (Å, º)

Br1—C4	1.903 (2)	C11—H11	0.9500
Br2—C10	1.898 (2)	C12—H12	0.9500
C1—C2	1.377 (3)	C13—O1	1.437 (3)
C1—O1	1.387 (2)	C13—O2	1.438 (2)
C1—C6	1.395 (3)	C13—C14	1.510 (3)
C2—C3	1.386 (3)	C13—C15	1.515 (3)
C2—H2	0.9500	C14—H14A	0.9800
C3—C4	1.384 (3)	C14—H14B	0.9800
C3—H3	0.9500	C14—H14C	0.9800
C4—C5	1.373 (3)	C15—C16	1.541 (3)
C5—C6	1.394 (3)	C15—H15A	0.9900
C5—H5	0.9500	C15—H15B	0.9900
C6—C8	1.471 (3)	C16—C17	1.524 (3)
C7—C12	1.384 (3)	C16—C18	1.532 (3)
C7—O2	1.390 (2)	C16—H16	1.0000
C7—C8	1.393 (3)	C17—H17A	0.9800
C8—C9	1.390 (3)	C17—H17B	0.9800
C9—C10	1.379 (3)	C17—H17C	0.9800
C9—H9	0.9500	C18—H18A	0.9800
C10—C11	1.375 (3)	C18—H18B	0.9800
C11—C12	1.390 (3)	C18—H18C	0.9800
C2—C1—O1	119.39 (18)	O1—C13—C14	104.76 (18)
C2—C1—C6	121.15 (19)	O2—C13—C14	110.46 (17)
O1—C1—C6	119.15 (19)	O1—C13—C15	111.15 (17)
C1—C2—C3	120.3 (2)	O2—C13—C15	103.98 (18)
C1—C2—H2	119.8	C14—C13—C15	116.49 (17)
C3—C2—H2	119.8	C13—C14—H14A	109.5
C4—C3—C2	118.3 (2)	C13—C14—H14B	109.5
C4—C3—H3	120.8	H14A—C14—H14B	109.5
C2—C3—H3	120.8	C13—C14—H14C	109.5
C5—C4—C3	122.06 (19)	H14A—C14—H14C	109.5
C5—C4—Br1	118.37 (16)	H14B—C14—H14C	109.5
C3—C4—Br1	119.57 (16)	C13—C15—C16	114.87 (19)
C4—C5—C6	119.70 (19)	C13—C15—H15A	108.6
C4—C5—H5	120.2	C16—C15—H15A	108.6
C6—C5—H5	120.2	C13—C15—H15B	108.6
C5—C6—C1	118.4 (2)	C16—C15—H15B	108.6
C5—C6—C8	121.27 (18)	H15A—C15—H15B	107.5
C1—C6—C8	120.31 (18)	C17—C16—C18	109.68 (19)
C12—C7—O2	119.0 (2)	C17—C16—C15	111.01 (19)
C12—C7—C8	121.7 (2)	C18—C16—C15	112.25 (19)
O2—C7—C8	118.87 (18)	C17—C16—H16	107.9

C9—C8—C7	117.69 (19)	C18—C16—H16	107.9
C9—C8—C6	122.05 (19)	C15—C16—H16	107.9
C7—C8—C6	120.24 (18)	C16—C17—H17A	109.5
C10—C9—C8	120.4 (2)	C16—C17—H17B	109.5
C10—C9—H9	119.8	H17A—C17—H17B	109.5
C8—C9—H9	119.8	C16—C17—H17C	109.5
C11—C10—C9	121.8 (2)	H17A—C17—H17C	109.5
C11—C10—Br2	118.97 (17)	H17B—C17—H17C	109.5
C9—C10—Br2	119.27 (18)	C16—C18—H18A	109.5
C10—C11—C12	118.6 (2)	C16—C18—H18B	109.5
C10—C11—H11	120.7	H18A—C18—H18B	109.5
C12—C11—H11	120.7	C16—C18—H18C	109.5
C7—C12—C11	119.8 (2)	H18A—C18—H18C	109.5
C7—C12—H12	120.1	H18B—C18—H18C	109.5
C11—C12—H12	120.1	C1—O1—C13	117.59 (17)
O1—C13—O2	110.04 (15)	C7—O2—C13	117.64 (17)

O1—C1—C2—C3	−174.4 (2)	C8—C9—C10—C11	0.6 (4)
C6—C1—C2—C3	−0.9 (4)	C8—C9—C10—Br2	−179.60 (18)
C1—C2—C3—C4	0.1 (4)	C9—C10—C11—C12	−0.8 (4)
C2—C3—C4—C5	0.5 (4)	Br2—C10—C11—C12	179.40 (19)
C2—C3—C4—Br1	−178.75 (18)	O2—C7—C12—C11	−171.7 (2)
C3—C4—C5—C6	−0.4 (4)	C8—C7—C12—C11	0.6 (4)
Br1—C4—C5—C6	178.88 (17)	C10—C11—C12—C7	0.2 (4)
C4—C5—C6—C1	−0.4 (3)	O1—C13—C15—C16	63.4 (2)
C4—C5—C6—C8	−179.8 (2)	O2—C13—C15—C16	−178.25 (16)
C2—C1—C6—C5	1.0 (4)	C14—C13—C15—C16	−56.5 (3)
O1—C1—C6—C5	174.5 (2)	C13—C15—C16—C17	−142.7 (2)
C2—C1—C6—C8	−179.6 (2)	C13—C15—C16—C18	94.2 (2)
O1—C1—C6—C8	−6.0 (3)	C2—C1—O1—C13	−111.5 (2)
C12—C7—C8—C9	−0.8 (4)	C6—C1—O1—C13	74.8 (3)
O2—C7—C8—C9	171.5 (2)	O2—C13—O1—C1	−45.4 (2)
C12—C7—C8—C6	−179.2 (2)	C14—C13—O1—C1	−164.10 (17)
O2—C7—C8—C6	−6.8 (3)	C15—C13—O1—C1	69.3 (2)
C5—C6—C8—C9	−37.5 (4)	C12—C7—O2—C13	−112.0 (2)
C1—C6—C8—C9	143.1 (2)	C8—C7—O2—C13	75.5 (2)
C5—C6—C8—C7	140.8 (2)	O1—C13—O2—C7	−45.3 (2)
C1—C6—C8—C7	−38.6 (3)	C14—C13—O2—C7	69.9 (2)
C7—C8—C9—C10	0.2 (4)	C15—C13—O2—C7	−164.38 (16)
C6—C8—C9—C10	178.5 (2)		